CRANFIELD UNIVERSITY

CHRISTOPHER ROSE

DEVELOPING A NUTRIENT RECOVERY PROCESS FOR
RECOVERING NUTRIENTS IN ANAEROBIC DIGESTATE IN LOW
INCOME COUNTRIES

CRANFIELD WATER SCIENCE INSTITUTE
School of Energy, Environment and Agrifood

Doctor of Philosophy
2015

Supervisors:
Dr. Alison Parker and Prof. Elise Cartmell
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ABSTRACT

It is estimated that 2.7 billion people worldwide are served by on-site sanitation facilities that require faecal sludge management. Anaerobic digestion is a treatment mechanism that can provide faecal sludge management, methane production and an effluent digestate rich in nutrients. However, there is a paucity of information regarding the composition of the input faecal sludge which hinders the advancement of anaerobic digestion treatment and downstream nutrient recovery together with a lack of knowledge as to how best to recover these output nutrients in a simple process.

Following an initial review to collate composition data for fresh faeces and urine, practical studies examined the physical, biological and chemical composition and variation of four different types of faecal sludge from on-site sanitation facilities. Faecal sludge storage strongly influenced the biodegradability and methane production potential in subsequent anaerobic digestion. However, the high concentrations of ammonium observed in faecal sludge (520-1853 mg NH_4-N L^{-1}) were highlighted as a key goal for nutrient recovery and the ability of biochar and clinoptilolite as natural adsorbents for ammonium recovery in a drying bed application were investigated through batch and dynamic studies using synthetic and real digestate. Batch tests observed ammonium uptake of 5 and 12.2 mg NH_4-N/g for biochar and clinoptilolite respectively whilst under dynamic experimental conditions the most efficient operation for ammonium recovery was at the longest empty bed contact times (354 minutes), ensuring the maximum fertiliser value was obtained (60g NH_4-N/kg clinoptilolite). Nevertheless, clogging occurred rapidly at the surface of the media bed (0.04 – 0.5 kg TS/m^2), consequently a sacrificial sand layer (0.05 m) was included to increase the longevity of the nutrient recovery system (15 fold increase in TS application rates). It has been demonstrated that clinoptilolite can effectively be used as part of a sludge drying bed configuration to recover nutrients from digestate and the saturated media can be used directly as a fertiliser product or blended with the dried sludge to create a balanced nitrogen, phosphorus and potassium fertiliser product (5.9% NH_4-N/ 4.2% P/ ≥6.0% K^+).

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LIST OF ABBREVIATIONS

AD   Anaerobic digester / Anaerobic Digestion

BMP   Biochemical methane potential

BOD   Biological oxygen demand

BV   Bed volumes

COD_{sol} Soluble chemical oxygen demand

COD_{tot} Total chemical oxygen demand

DI   Deionized water

DNA   Deoxyribonucleic acid

EBCT   Empty bed contact time

ECOSAN   Ecological sanitation

EGSB   Expanded granular sludge bed

EPSRC   Engineering and Physical Sciences Research Council

FS   Faecal sludge

FSM   Faecal sludge management

HPLC   High performance liquid chromatography

HRT   Hydraulic retention time

IBC   Intermediate bulk container

LUB   Length of unused bed

MSW   Municipal solid waste

NGS   Next generation sequencing

NMDS   Non-metric multi-dimensional scaling

ORP   Oxygen reduction potential

OSS   On site sanitation facility

OTU   Operational taxonomic unit

PCR   Polymerase chain reaction

PE   Population equivalent

RE   Removal capacity

rRNA   Ribosomal ribonucleic acid

TN   Total nitrogen

TS   Total solids

TSS   Total suspended solids

UASB   Upflow anaerobic sludge blanket

UDDT	Urine diversion and dehydration toilet
USEPA	United States Environmental Protection Agency
VFA	Volatile fatty acids
VIP	Ventilated improved pit
VS	Volatile solids
WwTW	Waste water treatment works

NOTATIONS

C_0	Column influent concentration
C_e	Column effluent concentration
VS_{added}	Volatile solids added
D	Particle size of media
IC_{50}	Half maximal inhibitory concentration value
Q_e	Adsorption capacity at equilibrium
R_e	Reynolds number
t_b	Time to breakthrough
t_e	Time to exhaustion
V	Superficial velocity of flow
ρ	Density

All notations are introduced within the text and re-introduced for each individual chapter.

CHAPTER 1

INTRODUCTION

1 INTRODUCTION

1.1 PROJECT BACKGROUND

An estimated 2.6 billion people in the world lack access to improved sanitation, defined as the hygienic separation of human excreta from human contact (WHO/UNICEF, 2012). Diseases that are associated with inadequate sanitation are particularly associated with poverty and account for 10% of the total disease burden worldwide (Prüss-Üstün et al., 2008). Poor sanitation and faecal sludge management not only have negative impacts on human health but also affect the environment through the contamination of water bodies, soils and food sources (Peletz et al., 2011; Ziegelbauer et al., 2012). On-site sanitation (OSS) facilities are the predominant form of excreta disposal in urban populations of low income areas; for example, in urban areas of Ghana and Tanzania 85% of inhabitants are served by OSS facilities and in urban areas of the Philippines 98% rely on OSS facilities (Montangero and Strauss, 2004). However, when these facilities need emptying, there are often inadequate facilities or financial disincentives for the proper disposal of faecal sludge meaning that pits remain full and unusable or if emptied, sludge is disposed of directly into the environment contaminating water resources (Ingallinella et al., 2002). This problem has inspired the development of technology that effectively treats faecal sludge from existing OSS infrastructure.

Knowledge of the waste that enters treatment systems is a basic prerequisite for the design and development of future technology as well as determining its re-use potential. There is information on conventional water borne sewage (Henze et al., 2001; Tchobanoglous et al., 2003) but this waste has a different composition to that of faecal sludge as the material will have undergone different periods of storage under differing storage conditions as well as having different levels of dilution through flush water and grey water additions. Therefore the generation rate, the chemical, physical and biological composition of faeces, urine and different types of faecal sludge are key factors to be understood by designers of treatment systems.

Anaerobic digestion (AD) can be used as an on-site or decentralised sanitation system and has great potential as part of faecal sludge management systems that undertake the collection, transportation, treatment and reuse/disposal of residual sludge from OSS facilities (Gautam et al., 2009; Bond and Templeton, 2011; Chen et al., 2012; Song et al., 2014). Anaerobic digestion has great potential as a faecal sludge treatment mechanism for low income regions as it not only provides biogas generation but also preserves nutrients present (Daisy and Kamaraj, 2011), which provides valuable resources for these communities. However, AD is not a complete treatment tool in itself; the output products require further treatment or storage before being used for agriculture, nutrient recovery, energy generation or being discharged to the environment.

The outputs of AD treating high nitrogen loaded material such as faecal sludge contain high concentrations of ammonium-nitrogen (NH_4-N) (Parsons et al., 2001). This NH_4-N is frequently not recovered and its value is currently not fully utilised. In low income countries unplanted drying beds are a common means of secondary treatment for digested sludge (Koné and Strauss, 2004; Cofie et al., 2006), however, through this nitrogen is volatilised and lost to the atmosphere or lost to the environment through the percolate. Existing problems in the use of drying beds in a low income context include long drying times, large land use, environmental pollution through liquid percolate loss and a final dried solid that has low nutrient values limiting its value for agricultural application (Tchobanoglous et al., 2003; WSUP, 2014). Most nutrient recovery systems require the prior separation of solids/liquids before nutrient recovery processes can take place. In a low income context if these timely and costly process of solids/liquids separation could be combined with recovery of nutrients this would present a significant benefit. This research aims to integrate an adsorption/ion exchange zone into the profile of a sludge drying bed in order to recover nutrients from the percolate fraction and achieve complete nutrient recovery in the sludge drying process.

The demand for nutrient recovery in the wastewater treatment process is important due to many factors. The world demand for fertiliser nutrients is

substantial (Table 1.1), with worldwide projected growth in nutrient demand at 1.4%, 2.2% and 2.6% in N, P_2O_5 and K_2O respectively (FAO, 2015). This rise in demand is also predicted to be especially pronounced in Africa, with growth rates of 3.2%, 2.7% and 7.8% in N, P_2O_5 and K_2O respectively (FAO, 2015). The input cost of fertilisers in arable food production is therefore significant (Table 1.1), vastly increasing the input costs of food production. Yet the nutrients required to enable the production of the annual amount of grain consumed by one person (250 kg) can be found in the faeces of one person (Wolgast, 1993). There are therefore significant economic gains that can be achieved if high value fertilising elements can be harnessed from human waste. Fertiliser production through nutrient recovery from human waste could therefore help to offset the need for mineral fertiliser production and the creation of new low cost fertiliser products could help reduce reliance on high-cost artificial fertilisers.

Table 1.1 The cost of the major fertilising elements (N, P and K) as of August 2015. Prices (FWI, 2015) in the currency of British £ and are quoted delivered.

Fertiliser Product	Price (£/tonne)*
Potash (MOP)	255
Phosphate (DAP)	385
Phosphate (TSP)	300
34.5% N	230
Granules Urea (46%N)	234
Ammonium Nitrate	217

Prices as of August 2015 commodity prices (FWI, 2015)

Table 1.2 World demand for fertiliser nutrients 2014-2018 (FAO, 2015)

	2014	2015	2016	2017
N	113 147	115 100	116 514	117 953
P_2O_5	42 706	43 803	44 740	45 718
K_2O	31 042	31 829	32 680	33 519
Total (N + P_2O_5 + K_2O)	186 895	190 732	193 882	197 190

Human sewage waste produces 3% of human nitrous oxide emissions (Denman et al. 2007), resultantly contributing to global climate change. Excess nitrogen pollution to waterways is also of significant global concern, with ammonia being one of the major pollutants introduced into receiving natural waterways by industrial, domestic and agricultural wastewater discharges (Hasanoğlu et al., 2010). This has significance due to the toxic effect of ammonia on most aquatic species (Tetreault et al., 2013) as well as the biological nitrification of ammonia to nitrite and nitrates which are dangerous to human health (Cockburn et al., 2013). The loss of nitrogen from soils occurs through leaching, direct run-off, ammonia volatilisation and through denitrification. Importantly, extensive amounts of nitrogen are also removed from the soils through crop offtake (including residues), especially under intensive crop production and accounts for the majority of nitrogen that leaves the soil. For instance, an average wheat crop grown with a yield of 10 tonnes/hectare (15% moisture) will remove approximately 170 kg N/hectare from the field, even if the residual straw is returned to the soil, according to UK agricultural guidelines (DEFRA, 2010). Consequently, it is the case that most agricultural soils do not contain enough naturally occurring plant available nitrogen to meet the needs of a crop throughout a growing season and soil nitrogen supply must be continually replenished. Resultantly, agricultural productivity relies heavily on N fertilisers in order to achieve maximum crop production (Xu et al., 2012). Indeed, the world demand for nitrogen fertilisers is over double that of phosphate or potash based fertilisers (Table 1.2). Ammonium nitrate is the most commonly used compound for chemical nitrogen fertiliser production (Fertilizers Europe, 2013), however, its production consumes more than 1% of the world's power production (Kitano et al., 2012). Consequently, if the sustained recovery of N from wastewater could be achieved, this could help to decrease demand for artificial fertiliser production and offset the ecological impact of energy usage through N production (McCarty et al., 2011). Despite not being a finite resource, unlike phosphorus; the recovery of nitrogen is of significant importance due to its ecological

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significance and the strong demand present for N fertilisers makes its recovery a financially viable alternative to the current energy intensive processes.

The principle driver for this work is to develop and create a solution to the high costs of sanitation by creating a high value agricultural soil amendment or fertiliser product from the outputs of anaerobic digestion. This will in turn help to reclaim the prior costs of collection and treatment of human waste as well as preventing the discharge of human waste to the environment. In creating a high value end product the proper treatment and disposal of human excreta will be promoted by financial incentives and is likely to subsequently contribute to improving human health, environmental conditions and productivity of this large sector of population.

1.2 THESIS AIMS AND OBJECTIVES

The aim of this thesis was to design a nutrient recovery system and process for the capture of nutrients from the effluent of anaerobic digesters. Consequently, the following objectives were identified:

1.   To review current knowledge of faeces and urine characteristics in order to determine how the physical and chemical composition will impact different treatment process types and nutrient recovery potential.

2.   To establish the physical, chemical and biological characteristics of different types of faecal sludge from on-site sanitation facilities and evaluate how the results will impact anaerobic digestion as a treatment process.

3.   To assess the digestibility of faecal sludge from a range of on-site sanitation facilities and establish the ultimate methane potential of each substrate.

4.   To determine technically feasible methods in which to recover nutrients in a low income context.

5.   To examine the feasibility of using a modified sludge drying bed system for the recovery of nutrients from anaerobic digestion effluents.

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6.	To propose design values for the construction and operation of nutrient recovering drying beds.

1.3 CONTRIBUTION TO KNOWLEDGE

This thesis reports research funded by the Engineering and Physical Sciences Research Council (EPSRC) as part of a wider research group of UK universities titled "A Global Solution to Water Scarcity and Health by Transforming Waste".

Anaerobic digestion has been one of the most widely used methods for sludge treatment since the early 1990s in high-income countries. In low income countries AD has great potential for treating the residual sludge from on-site sanitation facilities that are predominantly used in these regions. However, little is known about the physical and chemical composition of these facilities and what causes variation both within and between these systems. This PhD has determined how the AD processes may be impacted by the measured characteristics of OSS facilities from a range of differing types and locations. In addition this project has identified where nutrient recovery technology can best be adapted and used in a low income context. Nutrient recovery work within this project carried out experiments to determine whether NH_4-N adsorption processes in filter beds can be operated under high solids conditions which would potentially negate the need for costly prior solids/liquids separation. The final engineering design values for the nutrient recovery system are presented in the thesis and justified through experimental work.

1.4 Case Study Locations

This thesis undertook field work at two different case study sites: an undisclosed location refered to as *location x* and Kumasi, Ghana. Links to local water utility companies, community organisations and universities were made in order to enable the field work to take place in respective communities. Field work involved the sampling and analysis of faecal sludge at multiple locations. In *location x* analysis took place of pit latrine and public toilet faecal sludge. In Kumasi, sampling and analysis of portable toilet waste was carried out. Mobile

laboratories were set up within the respective locations in order to undertake analysis that was necessary to be carried out immediately on-site. Where possible samples were then preserved and transported to the UK for advanced analysis.

1.5 THESIS PLAN

This thesis is presented as a series of chapters formatted as papers for publication which have either been published, submitted or are in preparation. All papers were written by the lead author, Chris Rose, and co-authored and edited by Dr. Alison Parker, Prof. Elise Cartmell and selected others. All sampling and laboratory work was undertaken by the lead author, Chris Rose, with the exception of Chapter 3 where sample collection in the Ghana case study site was carried out by the third author, DNA sequencing analysis was carried out by the fourth author and finally in Chapter 6 where there was laboratory support in batch adsorption isotherm studies from another co-author. The thesis outline is presented in a flow diagram in Figure 1.1.

Initially a literature review was undertaken to explore the current knowledge regarding fresh faeces and urine characteristics in order to aid on-site treatment of fresh waste material. In this review, data were extracted from the medical literature and a statistical assessment of the data were undertaken. The discussion section placed an emphasis on assessing the impact of variation on advanced treatment processes that are currently being developed (Chapter 2, Paper 1, was published in Critical Reviews in Environmental Science and Technology-*. Rose, C., Parker, A., Jefferson, B., Cartmell, E., The Characterisation of faeces and urine; a review of the literature to inform advanced treatment technology", Critical Reviews in Environmental Science and Technology, vol. 45, no. 17, pp. 1827-1879.*

In Chapter 3 the physico-chemical characteristics of faecal sludge from four different types of on-site sanitation treatment system was assessed. Two separate field sites were investigated and a set of techniques developed for the physico-chemical characterisation of faecal sludge. The results give a full

characterisation of the sludge spanning its physical composition, organic content, nutrient value, potential toxicity and digestability. The discussion section addresses how the composition of faecal sludge from these instalments may impact anaerobic treatment and points to potential problems and benefits of anaerobic treatment in this context. (Chapter 3, Paper 2, submitted to Journal of Hazardous Materials-: *Rose, C., Parker, A., Santi, M., Ijaz, U., Cruddas, P., Collins, G., Cartmell, E. The characterisation of faecal sludge from on-site sanitation systems and application to anaerobic treatment technologies in low income countries.*

Chapter 4 determines the digestibility and biochemical methane potential of faecal sludge from differing types of on-site sanitation facilities (Chapter 4, Paper 3, under review in Environmental Technology–*Rose, C., Parker A., Buamah, R., Kabika, J., Collins, G., Cartmell, E., The biochemical methane potential of faecal sludge from on-site sanitation facilities in low income countries*). The results outline the biochemical methane potential of different substrates and examine the effect of retention time of the on-site sanitation facility as well as the impact of sampling depths within the on-site sanitation facility.

Chapter 5 utilises faecal sludge characterisation data from Chapter 3 in order to provide predictions of the composition of anaerobic digestate and explores technically feasible options and opportunities for nutrient recovery (Chapter 5, Paper 4, Submitted to Journal of Water, Sanitation and Hygiene for Development - *Rose, C., Parker A., Cartmell, E., The recovery of nutrients from faecal sludge: appropriate selection and advancement of technology* - and it is under review. In this chapter NH_4-N is recognised as the prime target for nutrient recovery with the combination of secondary treatment process, such as sludge drying beds, with adsorption and ion exchange processes identified as the most suitable option for NH_4-N recovery. In addition potential adsorption and ion exchange media are explored and identified as appropriate for application and further testing.

Chapter 6 focusses on nutrient recovery, in particular NH_4-N capture from high strength waste water streams through the use of non-regenerative media in passive treatment systems. Experimental work established the most suitable media, factors that affect NH_4-N uptake and the most efficient operational configuration in regards to bed depth, hydraulic flow rate and influent concentration for recovering NH_4-N. This was done using a down-flow fixed media filter bed. (Chapter 6, Paper 5, in preparation to be submitted to Journal of Separation and Purification Technology – *Rose, C., Parker A., Ezbakhe, F., Jefferson, B., Collins, G., Cartmell, E. Passive ammonium recovery through the use of low-cost media without regeneration*). In this chapter clinoptilolite is highlighted as the most efficient media for ammonium recovery in a synthetic wastewater effluent constructed to simulate digestate without the interference of solid matter. The operation of a fixed clinoptilolite bed at long empty bed contact times (EBCT) is identified as the main factor influencing media capacity with a 12 fold increase in media capacity reported with an increase in EBCT from 20-354 minutes. This increase was attributed to greater contact time between the media and solution, allowing increased intra-particle diffusion to take place. Numerous applications for a passive NH_4-N recovery system are identified; however, the need to further understand how the presence of solids in wastewater could impact the performance of the media was identified.

Chapter 7 presents a process to recover nutrients from digestate through the integration of an adsorption/ion exchange zone into a sludge drying bed, allowing the simultaneous dewatering and recovery of nutrients from the percolate stream. The use of a sand filter to act as a solids barrier to prolong the media bed life is explored and final engineering design values for the construction, operation and nutrient recovery products created by the system are presented. (Chapter 7, Paper 6, in preparation to be submitted to Water Research – *Rose, C., Parker A., Ezbakhe, F., Jefferson, B., Collins, G., Cartmell, E. Integrated sludge drying beds and nutrient recovery utilising non-regenerative media and a sacrificial sand barrier*). In this chapter clinoptilolite demonstrated favourable performance as part of the integrated system with

high levels of NH_4-N recovery (62-99% recovery) despite high levels of solids in the percolate. This indicates the ion exchange process is not adversely impacted by the high solids nature of drying bed percolate streams. The dewatering mechanisms of a sacrificial sand barrier are investigated and applied to blocking and sludge cake filtration theory. A design utilising the scraping of dewatered solids is recommended to intensify sludge drying beds and allow complete nutrient recovery to be achieved through the blending of dewatered biosolids rich in phosphorus with the nitrogen and potassium captured by the media layers beneath.

Chapter 8 is the overall discussion of the thesis and the key findings of the study are discussed alongside implications and practicalities for the sector. Chapter 9 is the final conclusions of the research.

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Figure 1.1 Flow diagram of thesis content and structure

1.6 REFERENCES

Bond, T. and Templeton, M. R. (2011), "History and future of domestic biogas plants in the developing world", *Energy for Sustainable Development,* vol. 15, no. 4, pp. 347-354.

Chen, L., Zhao, L., Ren, C. and Wang, F. (2012), "The progress and prospects of rural biogas production in China", *Energy Policy,* vol. 51, no. 0, pp. 58-63.

Cockburn, A., Brambilla, G., Fernández, M., Arcella, D., Bordajandi, L. R., Cottrill, B., van Peteghem, C. and Dorne, J. (2013), "Nitrite in feed: From Animal health to human health", *Toxicology and applied pharmacology,* vol. 270, no. 3, pp. 209-217.

Cofie, O. O., Agbottah, S., Strauss, M., Esseku, H., Montangero, A., Awuah, E. and Kone, D. (2006), "Solid–liquid separation of faecal sludge using drying beds in Ghana: Implications for nutrient recycling in urban agriculture", *Water Research,* vol. 40, no. 1, pp. 75-82.

Daisy, A. and Kamaraj, S. (2011), "The Impact and Treatment of Night Soil in Anaerobic Digester: A Review", *Journal of Microbial and Biochemical Technology,* vol. 3, pp. 43-50.

DEFRA (2010), *Fertilser Manual (RB209),* 8th ed, Defra, London, UK.

Denman, K.L., Brasseur, G., Chidthaisong, A., Ciais, P., Cox, P.M., Dickinson, R.E., Hauglustaine, D., Heinze, C., Holland, E., Jacob, D., Lohmann, U., Ramachandran, S., Dias, P.L.d.S., Wofsy, S.C., Zhang, X. (2007) *Couplings Between Changes in the Climate System and Biogeochemistry.* In: Solomon, S., Qin, D., Manning, M., Chen, Z., Marquis, M., Averyt, K.B., M.Tignor, Miller, H.L. (eds.) *Climate Change 2007: The Physical Science Basis Contribution of Working Group I to the Fourth Assessment Report of the Intergovernmental Panel on Climate Change.* pp. 501–587. Cambridge University Press, Cambridge, United Kingdom and New York, NY, USA

Fertilizers Europe (2013)*, EU Fertilizer Market - Key Graphs*, available at:http://www.fertilizerseurope.com/fileadmin/user_upload/publications/statistics_publications/Stat_website.pdf (accessed 5th May 2015)

FAO (2015), *World fertilizer trends and outlook to 2018,* 1st ed, Food and Agricultural Organisation of the United Nations, Rome, Italy

FWI (2015)*, Fertiliser Prices Stable in a Quiet Market*, available at: http://www.fwi.co.uk/business/fertiliser-prices-stable-in-quiet-market.htm (accessed October 2015)

Gautam, R., Baral, S. and Herat, S. (2009), "Biogas as a sustainable energy source in Nepal: Present status and future challenges", *Renewable and Sustainable Energy Reviews,* vol. 13, no. 1, pp. 248-252.

Hasanoğlu, A., Romero, J., Pérez, B. and Plaza, A. (2010), "Ammonia removal from wastewater streams through membrane contactors: Experimental and theoretical analysis of operation parameters and configuration", *Chemical Engineering Journal,* vol. 160, no. 2, pp. 530-537.

Henze, M., Harremoës, P., la Cour Jansen, J. and Arvin, E. (2001), *Wastewater treatment: biological and chemical processes,* 3rd ed, Springer, Berlin, Germany.

Ingallinella, A., Sanguinetti, G., Koottatep, T., Montangero, A., Strauss, M., Jimenez, B., Spinosa, L., Odegaard, H. and Lee, D. (2002), "The challenge of faecal sludge management in urban areas- strategies, regulations and treatment options", *Water Science and Technology,* vol. 46, no. 10, pp. 285-294.

Kitano, M., Inoue, Y., Yamazaki, Y., Hayashi, F., Kanbara, S., Matsuishi, S., Yokoyama, T., Kim, S., Hara, M. and Hosono, H. (2012), "Ammonia synthesis using a stable electride as an electron donor and reversible hydrogen store", *Nature chemistry,* vol. 4, no. 11, pp. 934-940.

Koné, D. and Strauss, M. (2004), "Low-cost options for treating faecal sludges (FS) in developing countries–Challenges and performance", *9th International IWA Specialist Group Conference on Wetlands Systems for Water Pollution Control and to the 6th International IWA Specialist Group Conference on Waste Stabilisation Ponds,* Vol. 27, 27th Sept. - 1st Oct.., Avignon, France, IWA, London, UK, .

McCarty, P. L., Bae, J. and Kim, J. (2011), "Domestic wastewater treatment as a net energy producer–can this be achieved?", *Environmental Science and Technology,* vol. 45, no. 17, pp. 7100-7106.

Montangero and Strauss, M. (Dept. of Water & Sanitation in Developing Countries), (2004), *Faecal Sludge Treatment* Eawag, Swiss Federal Institute of Aquatic Science & Technology.

Parsons, S., Wall, F., Doyle, J., Oldring, K. and Churchley, J. (2001), "Assesing the potential for struvite recovery at sewage treatment works", *Environmental Technology,* vol. 22, no. 11, pp. 1279-1286.

Peletz, R., Simuyandi, M., Sarenje, K., Baisley, K., Kelly, P., Filteau, S. and Clasen, T. (2011), "Drinking Water Quality, Feeding Practices, and Diarrhea among Children under 2 Years of HIV-Positive Mothers in Peri-Urban Zambia", *The American Journal of Tropical Medicine and Hygiene,* vol. 85, no. 2, pp. 318-326.

Prüss-Üstün, A., Bos, R., Gore, F. and Bartram, J. (2008), *Safer water, better health: costs, benefits and sustainability of interventions to protect and promote health.* 1st ed, World Health Organization, Geneva.

Song, Z., Zhang, C., Yang, G., Feng, Y., Ren, G. and Han, X. (2014), "Comparison of biogas development from households and medium and large-scale biogas plants in rural China", *Renewable and Sustainable Energy Reviews,* vol. 33, no. 0, pp. 204-213.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Tetreault, G. R., Brown, C. J., Bennett, C. J., Oakes, K. D., McMaster, M. E. and Servos, M. R. (2013), "Fish community responses to multiple municipal wastewater inputs in a watershed", *Integrated environmental assessment and management,* vol. 9, no. 3, pp. 456-468.

WHO/UNICEF (2012), *Progress on drinking water and sanitation; 2012 update,* MDG assessment report, Joint Monitoring Programme for Water Supply and Sanitation., U.S.A.

WSUP (2014), *FSM sercices in x: moving up the excreta management ladder,* pn#017, Water and Sanitation for the Urban Poor, London, UK.

Xu, G., Fan, X. and Miller, A. J. (2012), "Plant nitrogen assimilation and use efficiency", *Annual review of plant biology,* vol. 63, pp. 153-182

Ziegelbauer, K., Speich, B., Mäusezahl, D., Bos, R., Keiser, J. and Utzinger, J. (2012), "Effect of Sanitation on Soil-Transmitted Helminth Infection: Systematic Review and Meta-Analysis", *PLoS Medicine,* vol. 9, no. 1, pp. e1001162.

CHAPTER 2

LITERATURE REVIEW - THE CHARACTERISATION OF FAECES AND URINE; A REVIEW OF THE LITERATURE TO INFORM ADVANCED TREATMENT TECHNOLOGY

2 THE CHARACTERISATION OF FAECES AND URINE; A REVIEW OF THE LITERATURE TO INFORM ADVANCED TREATMENT TECHNOLOGY

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ABSTRACT

The safe disposal of human excreta is of paramount importance for the health and welfare of populations living in low income countries as well as the prevention of pollution to the surrounding environment. On site sanitation (OSS) systems are the most numerous means of treating excreta in low income countries, these facilities aim at treating human waste at source and can provide a hygienic and affordable method of waste disposal. However, current OSS systems need improvement and require further research and development. Development of OSS facilities that treat excreta at, or close to, its source require knowledge of the waste stream entering the system. Data regarding the generation rate and the chemical and physical composition of fresh faeces and urine was collected from the medical literature as well as the treatability sector. The data were summarised and statistical analysis was used to quantify the major factors that were a significant cause of variability. The impact of this data on biological processes, thermal processes, physical separators and chemical processes was then assessed. Results showed that the median faecal wet mass production was 128 g/cap/day, with a median dry mass of 29 g/cap/day. Faecal output in healthy individuals was 1.20 defecations per 24 hour period and the main factor affecting faecal mass was the fibre intake of the population. Faecal wet mass values were increased by a factor of 2 in low income countries (high fibre intakes) in comparison to

values found in high income countries (low fibre intakes). Faeces had a median pH of 6.64 and were composed of 74.6% water. Bacterial biomass is the major component (25-54% of dry solids) of the organic fraction of the faeces. Undigested carbohydrate, fibre, protein and fat comprise the remainder and the amounts depend on diet and diarrhoea prevalence in the population. The inorganic component of the faeces is primarily undigested dietary elements that also depend on dietary supply. Median urine generation rates were 1.42 litres/cap/day with a dry solids content of 59 g/cap/day. Variation in the volume and composition of urine is caused by differences in physical exertion, environmental conditions as well as water, salt and high protein intakes. Urine has a pH 6.2 and contains the largest fractions of nitrogen, phosphorus and potassium released from the body. The urinary excretion of nitrogen was significant (10.98 g/cap/day) with urea the most predominant constituent making up over 50% of total organic solids. The dietary intake of food and fluid is the major cause of variation in both the faecal and urine composition and these variables should always be considered if the generation rate, physical and chemical composition of faeces and urine is to be accurately predicted.

KEYWORDS

Faeces, urine, human excreta, faecal characteristics, urine characteristics, faeces treatment.

2.1 INTRODUCTION

An estimated 2.6 billion people in the world lack access to improved sanitation, defined as the hygienic separation of human excreta from human contact (WHO/UNICEF, 2012). Diseases that are associated with inadequate sanitation are particularly associated with poverty and account for 10% of the total disease burden worldwide (Prüss-Üstün et al., 2008). Poor sanitation and faecal sludge management not only have negative impacts on human health

but also affect the environment through the contamination of water bodies, soils and food sources (Peletz et al., 2011; Ziegelbauer et al., 2012). In 2010, 72% of sanitation facilities in Sub-Saharan Africa and 59% in Southern Asia were classified as 'unimproved' (WHO/UNICEF, 2012). On-site sanitation (OSS) facilities are the predominant form of excreta disposal in urban populations of low income areas; for example in urban areas of Ghana and Tanzania 85% of inhabitants are served by OSS facilities and in urban areas of the Philippines 98% rely on OSS facilities (Montangero and Strauss, 2004). However, when these facilities need emptying, there are often inadequate facilities or financial disincentives for the proper disposal of faecal sludge meaning that pits remain full and unusable or if emptied, sludge is disposed of directly into the environment contaminating water resources (Ingallinella et al., 2002). This problem has inspired the development of OSS technologies that treat excreta directly at or close to its source, producing safe and beneficial products with no need for further transport. This factor is illustrated by a rapid rise in research and development in OSS technology, with the Bill and Melinda Gates Foundation (BMGF) funding 16 'Reinvent the Toilet Challenge' (RTTC) research projects worldwide since 2011, with the second round of grants totalling nearly US$3.4 million in 2012 (Global Development Programme, 2014). This trend is continuing with the BMGF investing in regional programmes, for example US$5 million has been awarded to Chinese research institutes to drive research and development into new OSS systems (Global Development Programme, 2014).

Knowledge of the waste that enters treatment systems is a basic prerequisite for the design and development of future technology. There is information on conventional sanitary sewage (Henze et al., 2001; Tchobanoglous et al., 2003) but this material has a different composition to fresh faeces and urine which has not undergone any degradation processes and will have substantially less water or greywater addition. Instead generation rates and the chemical composition of faeces and urine in the human population are key factors to be understood by OSS technology developers. A number of medical studies have determined the faecal and urine output of human populations,

however the data were specific to distinct populations defined by geography, age, ethnicity, disease and diet. There have so far been no attempts to summarise these data and understand the major causes of variation. The aim of this study is to review the variation, generation rate and chemical and physical composition of the solid and liquid fractions of human excreta that would supply OSS technologies in developing countries. An assessment will then be made on how the results and any variation found will impact on potential treatment technology.

2.2 METHODS

Generation rate, composition, physical and chemical nature of both faeces and urine were recorded as of Table 2.1. Each recorded datum was the mean of the data from the reported study. Some published papers reported two or more independent studies so these papers contributed more than one value to the data set. The mean and median of each variable were both calculated as measures of central tendency and data were checked for normality by calculating a coefficient of skewness (Young, 1962);

$$Skewness = n*M_3 /[(n-1)*(n-2)*\sigma^3] \tag{1}$$

$$M_3 = \Sigma(x_i - Mean_x)^3 \tag{2}$$

$\sigma = Standard\ deviation$

$n = Valid\ number\ of\ cases$

Table 2.1 Measured variables for faeces and urine.

Variable	Faeces Unit of Measure	Urine Unit of Measure
Generation	**g/cap/day**	**L/cap/day**
Frequency of defecation	motions/24hours	urinations/24hours
Water Content	% total mass	% total mass
Organic composition	% total mass	% dry mass
Components of solids	% total mass	% total mass
Inorganic composition	% dry mass	% dry mass
Daily excretion of elements	g/cap/day	g/cap/day, mg/L
Chemical Nature		
pH	pH	pH
COD and BOD	mg/g wet mass	mg/L

Physical Form	
Bristol Stool Form	Linear scale (1-7)
Diarrhoea prevalence	% of population

Box and whisker plots were created using Statistica 11 software (Statsoft Inc., 2011). Outliers of each data set were defined using a standard default outlier coefficient value (Burns et al., 2005).

*Outliers = Upper value of the 75th percentile * outlier coefficient of 1.5*   (3)

*Extreme values = Upper value of 75th percentile *2 outlier coefficient.*

No outliers were removed from the data set but were identified in the graphical output. Full statistical calculations were only conducted on variables that had at least 7 values but a median value is given for data when there were less than 7 values.

A summary of studies used in the statistical analysis are outlined in Table 2.2, including the location and number of studies. A large proportion (80%) of the data set was from studies conducted in Europe and North America. A distinction was therefore made between low and high income countries by the measure of development; using the Human Development Index (HDI), a composite index measuring average achievement in three basic dimensions of human development; life expectancy, education and income (UNDP, 2011).

Preliminary data analysis indicated that fibre intake was a major cause of variation in faecal generation and composition. There were a sufficient number of studies that had examined the effects of fibre intake on faecal output to enable further analysis to be undertaken on these data. The total dietary fibre intake was related to the generation of faeces in linear and non-linear regression analyses.

Table 2.2 The geographical location and Human Development Index ranking of studies used in statistical analysis

Country	n	HDI*	References
Africa	2	3/4[a]	Cranston and Burkitt (1975), Burkitt et al. (1980)
Australia	2	1	Birkitt, et al. (1996), Hovey et al. (2003)
Burma	1	4	Myo-Kin et al. (1994)
Canada	3	1	Habbick et al. (1978), Burkitt et al. (1980), Vuksan et al. (1999)
China	3	2	Jie et al. (2000), Chen et al. (2008), Bai and Wang (2010)
Denmark	2	1	Maclennan (1977), Jensen et al. (1982)
Developing countries	2	3/4[a]	Feacham (1978)
Europe and North America	1	1/2[b]	Feacham (1978)
European	1	1[b]	Mykkanen et al. (1998)
Finland	4	1	Reddy et al (1975), Reddy et al. (1978), Jensen et al. (1982), Mykkanen et al. (1998)
Germany	1	1	Erhardt et al. (1997)
Guatemala	1	3	Calloway and Kretsch (1978)
Holland	4	1	Stasse-Wolthius et al (1980), Van Faassen et al. (1993), Gaillard (2002), Wierdsma et al. (2011)
India	1	3	Shetty and Kurpad (1986)
Iran	1	2	Adibi et al. (2007)
Japan	7	1	Glober et al. (1977), Polprasert et al (1981), Tarida (1984), Saitoh et al. (1999), Danjo et al. (2008), Shinohara et al. (2010), Hotta and Funamizu (2009)
Kenya	1	4	Cranston and Burkitt (1975)
New Zealand	1	1	Pomare et al. (1981)
North America	1	1[b]	Vuksan (2008)
Peru	1	2	Crofts (1975)
Singapore	1	1	Chen et al. (2000)
South Africa	2	3	Burkit et al. (1972), Walker et al. (1975)
Spain	1	1	Roig Villa et al. (1993)
Sweden	4	1	Ellström et al. (1977), Reddy et al. (1978), Vineras (2002), Vineras et al. (2006)
Thailand	2	2	Danivat et al.(1988), Schouw et al (2002)
Tonga	1	2	Pomare et al. (1981)
UK	26	1	Olmsted et al. (1934), Connell et al (1965), Southgate and Durnin (1970), Burkitt et al. (1972), Goy et al. (1976), Wyman et al. (1978), Prynne and Southgate (1979), Stephen et al. (1980), Eastwood et al. (1984), Eastwood et al. (1986), Davies et al. (1986), Cummings et al. (1987), Sandler and Drossman (1987), Cummings et al. (1992), Murphy et al. (1993), Cummings et al. (1996), Lewis et al (1997), Chen et al. (1998), Reddy et al. (1998), Rivero-Marcotegui et al. (1998), Aichbichler et al. (1998), Almeida et al. (1999),Magee et al. (2000), Chaplin et al (2000), Woodmansey et al. (2004), Silvester et al. (2011).
USA	18	1	Canfield et al. (1963), Watts et al. (1963), Diem and Lentner (1970), Goldsmith and Burkitt (1975), Cummings et al. (1978), Glober et al. (1977), Goldberg et al (1977), Beyer and Flynn (1978), Reddy et al. (1978),Calloway and Kretsch (1978), Kien et al. (1981), Polprasert et al (1981),Tucker et al (1981),Schubert et al. (1984),Parker and Gallagher (1988), Zuckerman, et al. (1995), Aichbichler et al. (1998), McRorle et al. (2000).

*Human Development Index Classifications (UNDP, 2011): 1. Very high, 2. High, 3. Medium, 4.Low
[a]Classification not available, presumed to be ranking 3 or 4 and [b]Classification not available, presumed to be ranking 1 or 2.

2.3 RESULTS

2.3.1 Faeces generation

Faecal wet mass values have a median figure of 128 g/cap/day. This is from a distribution of 116 mean values from studies reporting healthy individuals, with a large minimum and maximum range of 51-796 g/cap/day (Figure 2.1). However, as mean values for each study were recorded, individual variation within these studies is not accounted for; if all values are recorded the range extends to 15-1505 g/cap/day. The data set for mean wet faecal generation had a positive skew, hence the mean was greater than the median. The low income countries data set was not as skewed as the high income countries (Table 2.3). This is likely a result of the wider range of diets that can be consumed by populations in richer countries. A statistically significant difference (t= 2.87, P<0.05) between mean values of high income countries and low income countries was found in regards to wet faecal weight. As a collective group high income countries had relatively small per capita wet faecal weights in comparison to low income countries. However, between individual studies there was a large variation of 51-796 g/cap/day, despite all studies reporting healthy individuals. For low income countries the median value of 250 g/cap/day was larger in comparison to the median value of 126 g/cap/day in high income countries.

Table 2.3 Daily wet and dry mass produced by humans from low and high income populations.

	Wet weight (g/cap/day) High Income*	Wet weight (g/cap/day) Low Income*	Dry weight (g/cap/day) High Income*	Dry weight (g/cap/day) Low Income*
Median	126	250	28	38
n	95	17	57	8
Minimum	51	75	12	18
Maximum	796	520	81	62
Skewness	4.178	0.598	2.378	0.098
Std. Error of Skewness	0.248	0.550	0.327	0.752
Mean	149	243	30	39
St Dev	95.0	130.2	11.7	14.1
Variance	9024	16960	136	201

* Classifications acquired from the 2011 HDI report (UNDP, 2011) where the four tiers were split into two sections with "very high" and "high" comprising the high income classification and "medium" and "low" comprising the low income classification.
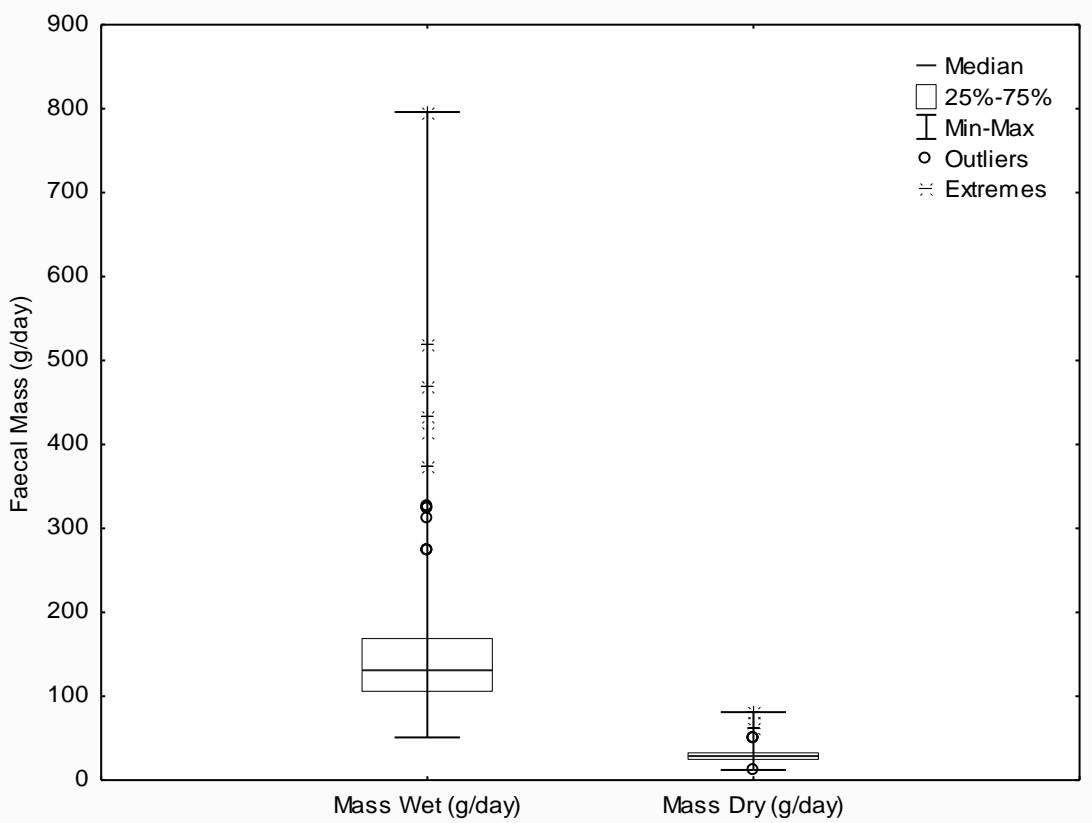
Outliers represent the upper value of the 75th percentile multiplied by the outlier coefficient (1.5), (extreme values = upper value of 75th percentile *2 outlier coefficient). Faecal wet mass generation (n=112) has a large range and was an abnormal data set. Faecal dry mass (n=61) showed a smaller range with fewer outliers and extreme values.

Figure 2.1 Daily wet and dry mass of faeces produced by human populations (g/cap/day)

The mean weight of children's faeces (3-18 years) has been recorded between 75-374 g/cap/day (Burkitt et al., 1972; Tandon and Tandon, 1975; Burkitt et al., 1980; Almeida et al., 1999; Schouw et al., 2002). Infants (1-4 years) were shown to have a mean stool weight of 85 g/cap/day with no significant difference found between the age of children in years, however, a weak correlation was found between the infants age in months and total stool weight (r=0.125, p<0.029) (Myo-Khin et al., 1994). Mean values for elderly subjects

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(aged 65 years or more) were reported at 158 g/cap/day by Mykkanen et al., (1998) and 70 g/cap/day by Woodmansey et al., (2004).

Table 2.4 The effect of diet type on faecal characteristics.

Diet type *	Fibre Intake (g/d)	Number of subjects in study	Faecal Mass wet (g/d)	Faecal Mass dry (g/d)	Stool Frequency (motions per 24hours)	Moisture (%)	Faecal pH	Reference
Omnivore	23	17	153		1			(Davies et al., 1986)
Vegetarian	37	17	168		1.2			(Davies et al., 1986)
Vegan	47	17	225		1.7			(Davies et al., 1986)
Omnivore		14			1.4	73.5		(Goldberg et al., 1977)
Vegetarian		14			1.8	73.3		(Goldberg et al., 1977)
Omnivore		66	131.9					(Lewis and Heaton, 1997)
Omnivore	16.6	22	117	30.8		72.6	6.65	(Reddy et al., 1998)
Vegetarian	16.2	22	186	36		78.9	6.18	(Reddy et al., 1998)
Vegetarian	29.3	18	160	38.4		74.6	6.55	(Reddy et al., 1998)
Omnivore[1]	12	8	129	32.8		74	7	(Silvester et al., 1997)
Omnivore[2]	11	8	118	32		70.7	7.2	(Silvester et al., 1997)
Omnivore	27.3	149	119	27.1	0.9		6.8	(Van Faassen et al., 1993)
Vegetarian	40.8	11	189	27.9	1.5		6.8	(Van Faassen et al., 1993)

*O: Omnivore, V: Vegetarian, VN: Vegan.
[1] Low meat diet (68g/d protein).
[2] High meat diet (192 g/d protein).

Median dry stool weight was 29 g/cap/day which were recorded from the mean values of 60 studies, with a range of means of 12-81 g/cap/day (Figure 2.1). Again, individual variation within these studies was not accounted for as mean values of these populations were taken; ranges of minimum and maximum values taking into account individual variation within these studies was subsequently larger at 4-102 g/cap/day dry solids. The data set was not of a normal distribution with a positive skew of 1.8. This was also due to the skewed

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distribution of values from high income countries (Table 2.3). The median dry weight of faeces is 25% of the wet weight of faeces (n=45) with values in the range of 11%-34% reported (Figure 2.1)

2.3.1.1 Factors affecting faecal mass

The major factors leading to variation in faecal generation rate are total food intake, body weight and diet. Parker and Gallagher (1992) found that mean daily stool weight was correlated (p <0.001) with calorie intake (energy intake can act as a measure of food intake); however, they found that this only accounted for 28% of the variation seen in individual stool output. Body weight also represents differing energy intake requirements; for example, as a guideline a healthy adult requires 20 -25 kcal/per kilogram of body weight (Moyes and McKee, 2008). The increasing body weight therefore reflects increasing energy intake which in turn can act as a measure of total food intake. Food intake and body weight therefore have an influence over faecal weight and this accounts for variables such as gender (Stephen et al., 1986; Lampe et al., 1993; Poullis et al., 2004) and race (Burkitt et al., 1972; Goldsmith and Burkitt, 1975) that have been observed as being significant within the literature.

Human diet is also a factor that can impact the generation rate and composition of faeces. Fibre intake is often cited for causing variation in faeces production, for example by Vuksan et al. (2008). Regression analysis of secondary data presented in 25 studies where fibre intake was recorded was conducted and results show that faecal wet mass was positively correlated with fibre intake (r= 2.96 ±1.13, p=0.017) (Figure 2.2).

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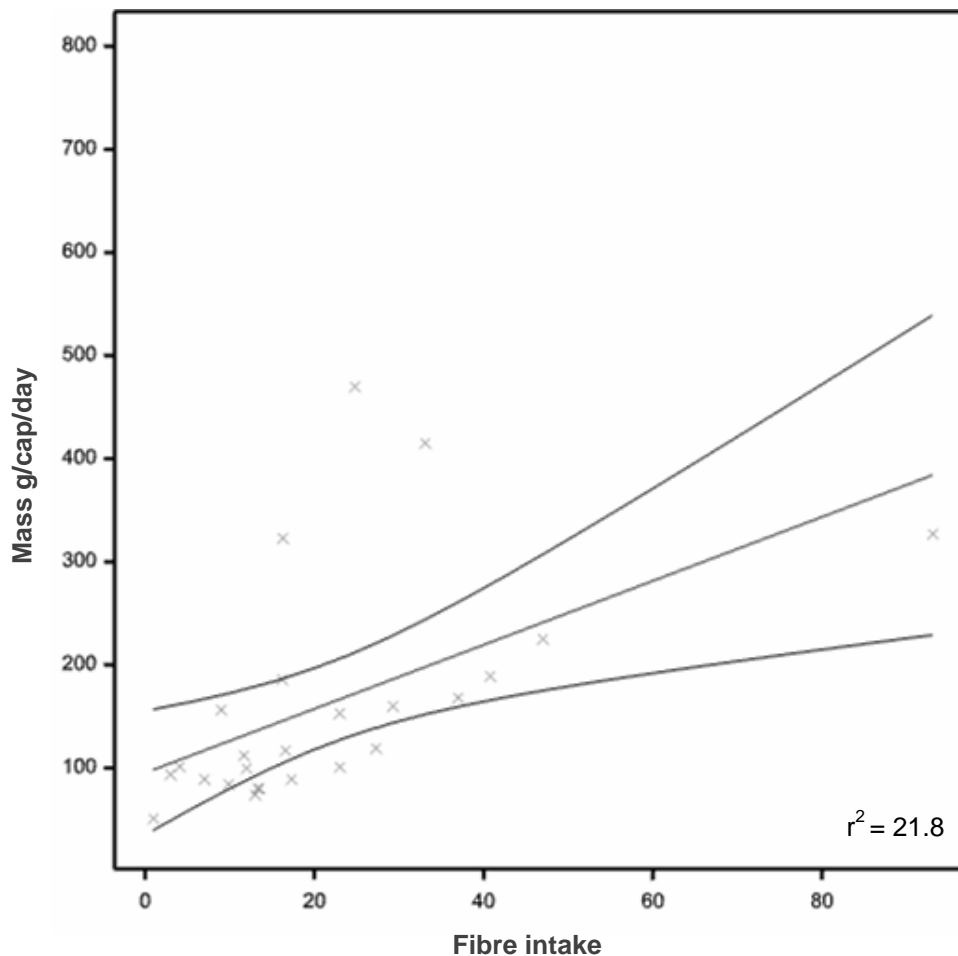
Figure 2.2 . Fitted and Observed relationship with 95% confidence limits. Values from 22 studies where fibre intake was recorded. Three large outliers were recorded, however, no reason could be found to exclude these results from the study. There was a significant correlation between dietary fibre intake and faecal output (r2 = 21.8, p=0.017) with an intercept 101.3 ± 34.3 and a regression coefficient of 2.96 ± 1.13.

The effect of dietary fibre on faecal weight is highly dependent upon the type of fibre consumed (non-degradable or degradable). Non-degradable fibre undergoes minimal changes in the digestive tract as it is relatively un-fermentable and shortens colonic transit time (Bijkerk et al., 2004); wet faecal mass has been negatively correlated with transit time, $r = -0.22$, $p<0.05$ (Eastwood et al., 1984). Non-degradable fibre has a high water holding capacity which promotes bulk and increased defecation frequency; extensive studies with non-degradable cereal fibres have shown this (Cummings et al., 1992;

Hughes et al., 2002; Vuksan et al., 2008). In a study on wheat bran by Vuksan et al. (2008) a ratio of 2.8 g stool/per g additional fibre on top of a control diet was observed. Degradable fibres can also cause an increase in faecal mass. Highly degradable types of fibre (such as cabbage fibre or oat bran) are fermented in the colon by bacteria much more than non-degradable fibres (Bijkerk et al., 2004). However, degradable fibres still increase faecal weights due to the proliferation of the bacterial component that is stimulated by the presence of a fermentable substrate (Garrow et al., 1993); the resultant increase in bacterial mass is soft, bulky and water retaining (FAO/WHO, 1997). Any alteration in the bacterial biomass component is significant as it can make up to 55% of total faecal solids (Stephen and Cummings, 1980). Therefore, the impact of dietary fibre on increasing faecal mass is dependent on the type of fibre consumed.

Polysacharides, such as Resistant Starches (RS) have similar properties to fibre and have also been shown to increase faecal wet weight in many studies (Shetty and Kurpad, 1986; Cummings et al., 1996; Silvester et al., 1997). Diets high in RS have shown a significant increase in faecal wet and dry weight; (Phillips et al., 1995) concluded that for every 1 g RS consumed (mean 34g/day) there was an increase in the faecal wet weight of 1.8 g. Undigested starch, as measured by dietary intake, reaching the colon was found to increase faecal output (g wet weight/day) by 42% (Phillips et al., 1995). This correlation can be largely attributed to increases in bacterial biomass with fermentation (Cummings et al., 1996).

2.3.1.2 Stool frequency

Defecation frequency provides an indication for design parameters relating to treatability as it provides an indication of how often a facility may be used. Stool frequency also provides an indication of the resultant texture and form of the faecal matter (see physical form section). Mean stool frequency across studies (n=39) ranged from 0.74-1.97 motions per 24 hours with a median value of 1.10 motions per 24 hour period (Figure 2.3). This represents a guideline figure for a population majority, however, within this variability exists. In a study by Parker

and Gallagher (1988) of over 25000 days worth of data, individuals had a range of means between 0.21-2.54 movements per 24 hours illustrating the variability that can occur for individuals in the same population. In a study of a UK population defecations were recorded per hour of the day; the majority of defecations, 61% and 59% in men and women respectively occurred in the morning (06:00-10:00) with peak times in men (20%) occurring between 07:00 and 08:00 and an hour later in women (21%) (Heaton et al., 1992). Another small peak in defecation timing was recorded at 17:00 and 18:00 which is a common time for the evening meal and few defecations were recorded during the night (01:00 to 05:00) (Heaton et al., 1992). The increase in defecation after meal times is primarily due to the resultant increased motor activity of the colon (Christensen, 1985).
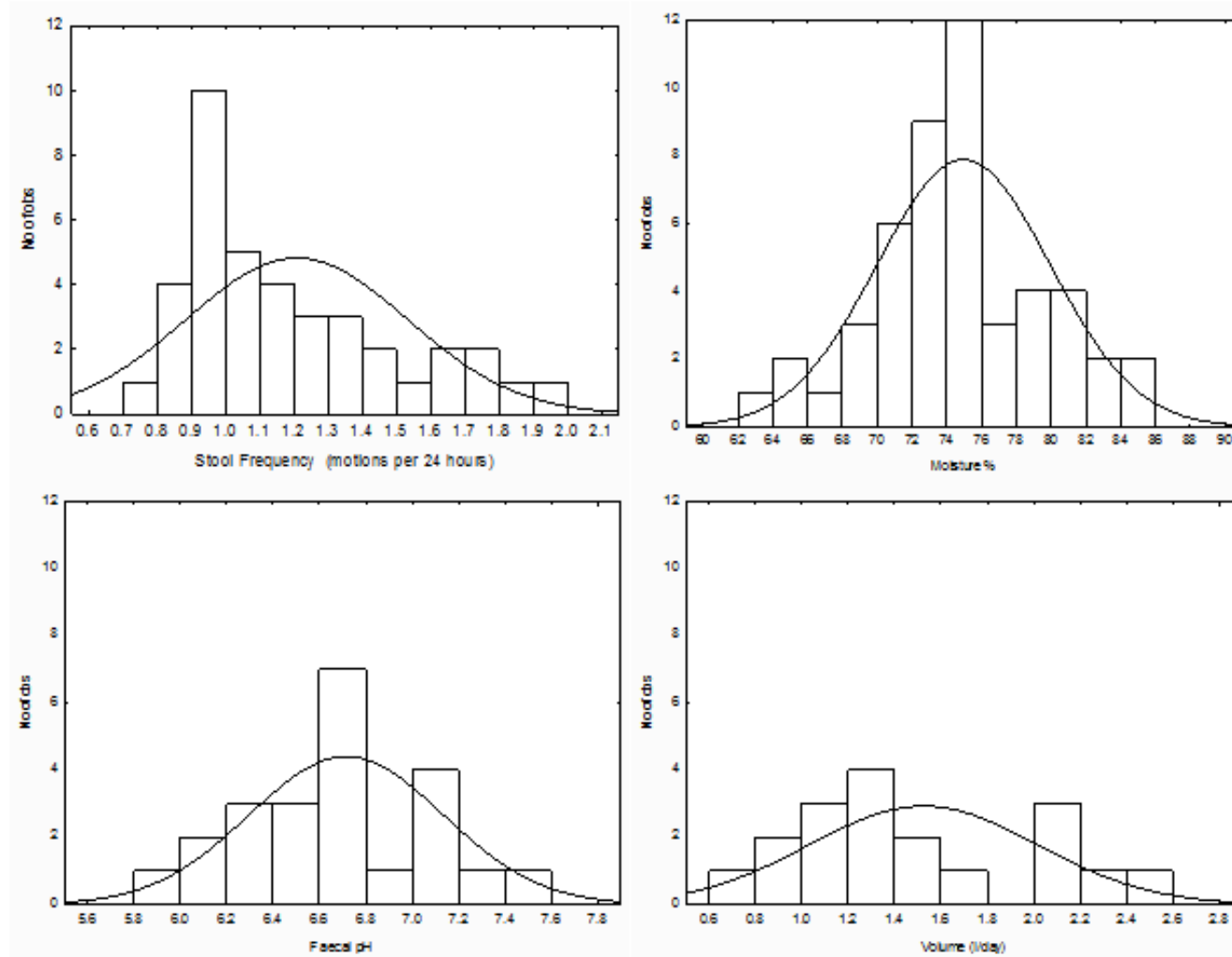
Figure 2.3 Top Left: Mean stool frequency in healthy subjects from a wide range of studies (n=39). Ranges of individuals within these studies varied from 0.21-2.54 motions per 24 hours. Top Right: Mean moisture composition of faeces (n=47). Bottom Left: Mean faecal pH values from a range of studies (n=28) consuming a variety of different diets. Bottom Right. Mean volume of total urine excreted (n=14)

Stool frequency is impacted by an individual's health (see physical form section) as well as their fibre intake which is associated with more rapid transit times (Gear et al., 1981). Fibre intake has been positively correlated with stool frequency (r = 0.8, p < 0.001 wet weight; r = 0.5, p = 0.008 dry weight) (Southgate et al., 1976). The inclusion of fibre from fruit and vegetables in the diet has been proven to decrease transit time (P < 0.05) and increase the number of defecations (P < 0.001) (Kelsay et al., 1978). For instance, in a study by Vuksan et al. (2008) high fibre breakfast cereals induced a shorter intestinal transit time and an increased stool frequency. In a meta-analysis of 5 relevant randomised controlled trials by Yang et al. (2012) dietary fibre was proven to increase stool frequency (odds ratio = 1.19; 95% CI: 0.58-1.80, *P* < 0.05).

Amongst adults no consistent relationship between frequency of defecation and age was observed (Heaton et al., 1992). Similarly amongst infants there was no significant difference in frequency of defecation between different age categories (Myo-Khin et al., 1994). A lower defecation frequency has been observed in females than in males (Van Faassen et al., 1993; Zuckerman et al., 1995; Chen et al., 2000) and this was accounted for by the longer intestinal transit time of females (p<0.02) (Gear et al., 1981). However, in children no significant difference was observed between the defecation frequency of boys (0.99/24 hours) and girls (0.96/24 hours) (Myo-Khin et al., 1994). A study by Sandler and Drossman (1987) undertaken in the U.S.A, indicated that the daily mean number of stools varied by race and by sex; whites had more frequent stools than non-whites at 1.3 vs 0.86 defecations/24 hours respectively and men had more frequent stools than women at 1.31 vs 0.96 defecations/24 hours respectively. Conversely, in a study of an Iranian population by Adibi et al. (2007) men were reported to have fewer bowel frequencies per day (1.78 vs 1.97).

2.3.2 Composition

Faeces are composed of water, protein, undigested fats, polysaccharides, bacterial biomass, ash and undigested food residues. The major elements in faeces as a percentage of wet weight are oxygen 74%, hydrogen 10%, carbon 5% and nitrogen 0.7%, including the hydrogen and oxygen present in the water fraction of the faeces (Snyder et al., 1975).

Faeces compose a median value of 75% H_2O (n=47) with a range of 63%-86% across mean values of studies (Figure 2.1), variation can be attributed to differences in fibre intake as non-degradable fibre absorbs more water in the colon (Eastwood, 1973); therefore, as shown in a study by Reddy et al. (1998) those with vegetarian diets will have a higher moisture content of 78.9% whereas those who consume less fibre and more protein will have a lower moisture content of 72.6% (p=0.001). Fibre intake also affects transit time, which has been positively correlated (r=0.4, p=0.03) with % dry matter (Silvester et al., 1997), showing the shorter the intestinal transit time the higher the water content. Variation in moisture content has been shown to vary with age; elderly people were found to excrete the highest amount of water in excreta of all age groups by Schouw et al. (2002). Further deviations from the median value can be caused by illness (see physical composition section). The mean generation rate of faecal water (n=47) is 0.1 L/cap/day. Average pH values for faecal water have been recorded at pH 6.9 with a range of pH 5.0-8.0 (Mai et al., 2009).
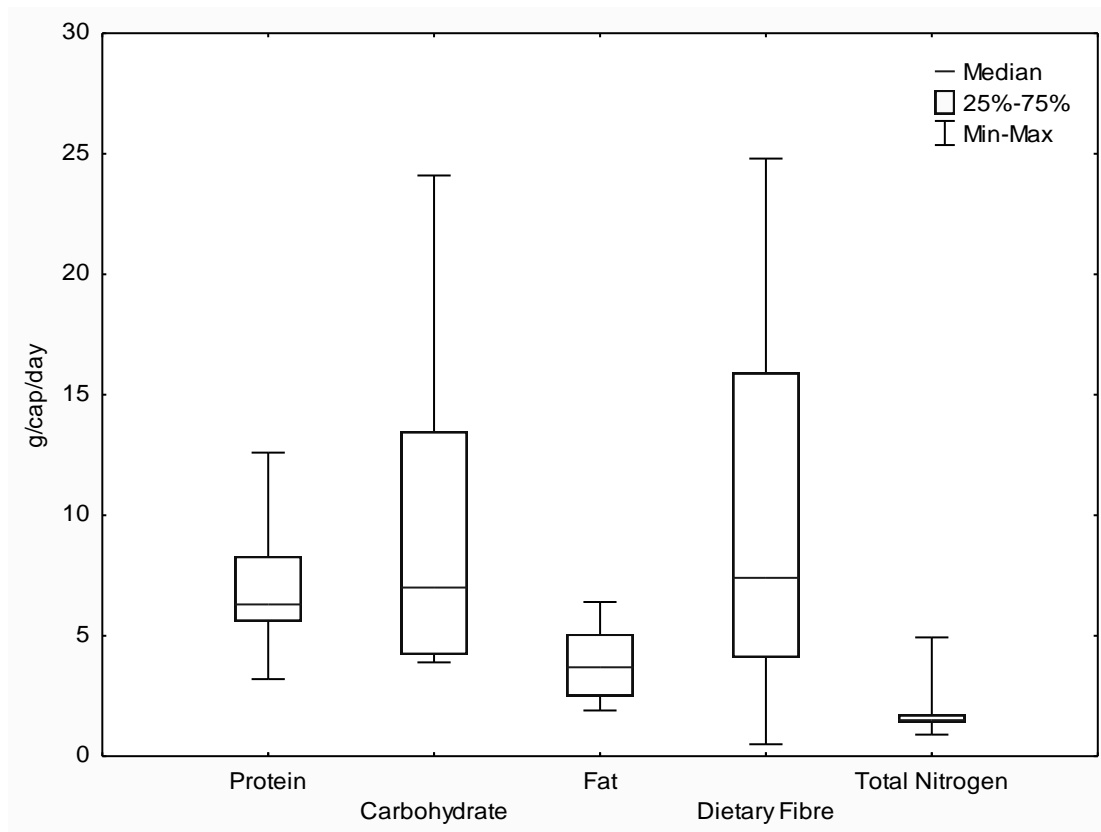
Figure 2.4 Daily per capita weights of organic fractions excreted in faeces

2.3.2.1 Organic fraction

The remaining 25% of faeces is therefore composed of solid material. Of the solid fraction organic material makes up between 84-93% (Feachem et al., 1978; Nwaneri et al., 2008; Bai and Wang, 2011). The organic solids fraction can be further broken down to the fractions of 25-54% bacterial biomass (Stephen and Cummings, 1980; Guyton and Hall, 2000), 2-25% protein or nitrogenous matter (in addition 50% of bacterial biomass is protein) (Canfield et al., 1963; Volk and Rummel, 1987)), 25% carbohydrate or any other non nitrogenous undigested plant matter (Volk and Rummel, 1987) and 2-15% undigested lipids (Kien et al., 1981; Chen et al., 1998; Wierdsma et al., 2011). These fractions are highly dependent on dietary intake and its biological availability.

The organic fraction therefore makes up the majority of dried solids. Carbon content of faeces is between 44-55% of dried solids (Feachem et al., 1978;

Strauss, 1985) or 7 g/cap/day (Snyder et al., 1975). Volatile solids were shown to comprise 92% of the total solids fraction of faeces (Fry and Merrill, 1973). The bulk organic content of faeces can also be measured by COD and BOD values (Table 2.5). Per capita daily values for BOD were between 14-33.5 g/cap/day. Values of COD were measured between 46-96 g/cap/day or 567-1671 mg/g dry faecal sample. Gas production of human faeces was placed at 0.02-0.28 per kg wet faeces (United Nations, 1984).

Table 2.5 Loading rates and concentration of BOD and COD in faeces

BOD g/cap/day	COD g/cap/day	COD mg/L	COD mg/g dry	COD mg/g wet	Reference
1223*	1668*				Vinneras et al. (2006)
		48900			Takahashi et al. (1989)
			1450		Zavala et al. (2002)
			1380		Almeida et al. (1999)
			1130		Nwaneri et al. (2008)
45					Heinss et al. (1998)
14-34	46-55				Kujawa-Roelveld and Zeeman (2006)
			567		Chaggu et al. (2007)
			1671		Bai and Wang (2010)
38	96				Choi et al. (2004)
19.3					Fourie and Ryneveld (1995)
			1448	354	Buckley et al. (2008)
32	50				Meinzinger and Oldenburg (2009)
		46230-78310			Chaggu (2004)

*includes toilet paper

2.3.2.2 Bacterial composition

A significant proportion of faecal mass consists of bacteria with estimates of combined dead and living bacteria of approximately 25-54% of dry solids (Stephen and Cummings, 1980; Guyton and Hall, 2000; Achour et al., 2006). The wide variation observed is due to differing methodology used between microscopic counting techniques and the separating of bacterial biomass. The high nitrogen content of faeces is partly due to undigested protein voided in the faeces but is also due to the significant protein content of bacterial biomass in the faeces, a figure of 50% protein was proposed by Volk and Rummel (1987); however, a more precise figure is not possible to determine due to uncertainties in the total bacterial composition of faeces. A detailed

break down of the microbial composition of faeces has been compiled by Stephen and Cummings (1980).

2.3.2.3 Nitrogen/protein

Nitrogen voided in faeces is also recorded as protein. The protein content of faeces can be estimated by multiplying the determined nitrogen content by a nitrogen-to-protein conversion factor. The Jones' factor (Jones, 1931) has been used extensively, with a standard default conversion factor of 6.25 (Mariotti et al., 2008), which is based on the average nitrogen content and composition of proteins. Data from measured mean values in faeces provides a median figure for protein daily loadings of 6.3 g/cap/day with a range of 3.2-16.2 (n=7) and for nitrogen 1.8 g/cap/day with a range of 0.9-4.9 (n=18) (Figure 2.4). Faecal nitrogen is present in the form of undigested dietary protein, nucleic acids, protein from bacteria and shed intestinal mucosal cells as well as being present in secreted mucus (Canfield et al., 1963; Bender and Bender, 1997). Nitrogen can make up 5-7% of the dried solids (Feachem et al., 1978) and of the nitrogen voided in the faeces fraction 50% is thought to be water-soluble (Montangero and Belevi, 2007).

Mean endogenous nitrogen excretion in 14 males has been measured at 0.96 g/cap/day in faeces, or 38 mg/kg body weight by Calloway and Margen (1971); this is the minimum nitrogen loading that can be expected. The safe rate of nitrogen intake to maintain nitrogen balance is 0.75 g protein/kg body weight/day (FAO/WHO/UNU, 1985) and as a guideline figure of nitrogen voided in faeces Bender and Bender (1997) concluded that when a healthy human is in nitrogen equilibrium, nitrogen excretion will be within 5% of the total nitrogen intake. Variation in the protein content of faeces is largely dependent on protein intake in the diet; however, the digestion rate of protein has been shown to vary from 69%-93% as a result of differing types of protein in the diet (Southgate and Durnin, 1970; Calloway and Kretsch, 1978). It should be noted that the majority of nitrogen output is in the urine fraction with this study showing that only 14% is voided through the faeces (1.8 g/cap/day) and the majority is excreted in urine (10.7 g/cap/day).

Concentrations of the differing nitrogenous fractions have also been recorded; Silvester et al. (1997) recorded faecal ammonia concentrations on low (68 g/day) and high (192 g/day) protein diets with values of 12mmol/kg (1.4 mmol/day) and 24mmol/kg (2.9mmol/day) respectively. Faecal nitrite levels were also found to be increased two fold on high protein diets, with values of 1678 µg/kg, in comparison to the lower protein diet with 829 µg/kg (Silvester et al., 1997).

2.3.2.4 Lipids

Fats contribute between 2.4%-8% of the wet weight of faeces (Canfield et al., 1963; Kien et al., 1981; Rivero-Marcotegui et al., 1998; Guyton and Hall, 2000; Wierdsma et al., 2011) or 8.7-16.0% of the dry weight of faeces (Calloway and Kretsch, 1978; Tarpila et al., 1978; Stephen et al., 1986). Daily loadings of fat in the faecal fraction from the mean values of 8 studies gave a median value of 4.1 g/cap/day and a range of 1.9-6.4 g/cap/day (Figure 2.4). However, it should be noted that only one out of the 8 studies was from outside Europe and North America (Guatemala): with this individual study presenting the lowest figure in the range of values (1.9 g/cap/day). Age differences have been observed, with infants voiding lower amounts of faecal fat 0.8-3.2 (Shmerling et al., 1970) and children aged 1-11 years voiding 0.9-5.9 (mean 3.0) g/cap/day of fat (Kuo and Huang, 1965). As would be expected faecal fat is positively correlated ($p<0.001$) with faecal wet mass and has also been positively correlated with fibre intake (Eastwood et al., 1984). Faecal fat excretion is dependent on dietary intake; however, even with no fat intake excretion of fat occurs. At high levels of fat intake there is no correlation between fat intake and faecal fat excretion (Gades and Stern, 2012). A significant positive correlation ($r=0.56$, $P=0.007$) between calcium intake and faecal fat excretion was found by Jacobsen et al. (2005) with faecal fat excretion on a high calcium diet increasing from 7% to 18% of dietary fat intake and an increase of 100 mg calcium resulting in an increase of 5.4 g in fat excretion. This increase is thought to be due to an interaction between calcium and fatty acids, which causes insoluble calcium fatty acids to form and resultantly reduces fat absorption and increases fat excretion (Jacobsen

et al. 2005). Fat found within faeces comes from bacteria and fat in the shredded epithelial cells as well as from the undigested dietary intake of fat (Guyton and Hall, 2000). Broadly the fat content includes substances such as fatty acids, waxes and phosphoglycerides.

2.3.2.5 Carbohydrate and energy value

The carbohydrate fraction is largely made up of undigested cellulose, vegetable fibres and pentosan (Canfield et al., 1963). Faeces do not contain large quantities of carbohydrates as the majority of what is consumed is absorbed; however, undigested and unabsorbed fractions (resistant starch) remain. A median value (n=10) of 9 g/cap/day carbohydrate in faeces was recorded with a range of 4-24 g/cap/day. The vast majority of studies were again conducted in North America and Europe with only one study in Peru presenting values in the centre of this range. The calorific content of faeces had a median value (n=14) of 132 kcal/cap/day (range: 49-347 kcal/cap/day). By using the median value of production (32 g/cap/day) a calorific value of 4115 kcal/kg dry solids can be used as a design standard for calorific value of faeces. All studies were carried out in North America and Europe therefore no correlation could be made between income and calorific value. However, the largest quantities of faecal energy are shown from diets containing a large amount of unavailable carbohydrates (Southgate and Durnin, 1970), defined as all polysaccharides not hydrolysed by the intestinal secretions of humans, as opposed to available carbohydrates such as starch and sugars which result in less faecal energy loss (Southgate, 1973).

2.3.2.6 Fibre

Human stools contain approximately 25% undigested plant matter, not including any nitrogenous material (Volk and Rummel, 1987). Fibre is present in stools due to the large linked polysaccharides that inhibit digestibility (Volk and Rummel, 1987), therefore the dietary intake will strongly influence the quantity found in faeces. The quantity of fibre found in faeces (n=8) ranged from 0.5-24.8 g/cap/day with a median value of 6 g/cap/day (Figure 2.4). Fibre consumption has also been shown to have significant effects on other

variables. It was found by Beyer and Flynn (1978) that when a high fibre diet was consumed and compared to a low fibre diet then measurements of faecal fat, protein, carbohydrate, and calories were more than doubled. Similar conclusions were made by Kelsay et al. (1978) when a high fibre diet from fruit was consumed. It was concluded that this was down to fibre consumption having a significant impact on absorption capacity in the gut.

2.3.2.7 Inorganic composition

The remaining solids compose the inorganic fraction which is predominantly made up of calcium phosphate and iron phosphate, intestinal secretions, small amounts of dried constituents of digestive juices such as shredded epithelial cells and mucus (Guyton and Hall, 2000; Iyengar et al., 1991). Fixed solids were measured at 3.13 g/cap/day by (Cummings et al., 1996) which was 2.25% of faecal wet weight and 9.02% of faecal dry weight. Fixed solids are in the range of 7.5% -16% of total solids (Feachem et al., 1978; Nwaneri et al., 2008; Bai and Wang, 2011); using the assumption of 29 g/cap/day TS then this would give a fixed solid value of between 2 g/cap/day and 4 g/cap/day.

In a healthy fully grown adult the amount of inorganic elements are in equilibrium (Kujawa-Roeleveld and Zeeman, 2006) and are not subject to any transformation within the body (Muñoz et al., 2007). Therefore it would be expected that the intake of elements would be equal to the output in human excreta. The intake of nutrients is therefore of great importance as well as the partitioning of these elements between the two excreta streams of faeces and urine. Wignarajah et al. (2003) found that the partitioning of elements between the urine and faecal fractions could be determined by looking at % absorption rates of inorganic elements in the body. Absorption rates were found to be predictable and reliable, therefore if the elemental input of the diet is known for an individual or population (alternatively it could be predicted from recommended daily allowance figures for that population), the partitioning between urine and faecal fractions could be predicted. This is because

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elements that are absorbed by the body will be excreted in the urine fraction and the remaining fraction will be voided in the faeces.

However, absorption rates are not clearly defined at high intake rates; an example cited by Wignarajah et al. (2003) is the partitioning of phosphate. The phosphate absorption rate at normal intake levels is 60%, however, at high rates of phosphate intake the absorption rate is markedly reduced to 40%. This means that at high levels of phosphate intake the relative amount of phosphate voided in faeces can be increased from 40% to 60% as the amount absorbed and excreted in urine is reduced.

Minimum and maximum values of elements (Table 2.6) can be used as an estimate of daily loading rates of elements voided in faeces; the variation is likely to be due to the differing dietary intakes which were not recorded. The intake of elements is therefore the most important variable. Therefore, factors that have an effect on this, such as heavy metal contamination of farmland or high concentrations of certain elements, such as lead in the air as a result of industrial pollution, also bear importance. Increased fibre intake has also been shown to lead to an increase in inorganic constituents, particularly Na and P (Southgate et al., 1976). Feachem et al. (1978) recorded % concentration of P, K and Ca at 3-5.4%, 1-2.5% and 4.5% respectively in the dried solid fraction. Levels of P in faeces have been shown to increase with increasing protein intake; however, protein intake had no other impact on Mg, K and Ca (Calloway and Margen, 1971). The total quantity of faeces voided will also have an impact on the quantity of constituents; Na, K, Mg, Ca, Zn were all found to be strongly correlated with faecal wet mass (Eastwood et al., 1984).

Table 2.6 Daily loadings and concentrations of elements in faeces (wet weight)

	Value (g/cap/day)	Value (g/kg)	Reference
Total P	0.35	3.40	(Vinnerås et al., 2006)
	0.5	1.83	(Czemiel, 2000)
	0.5	3.59	(Vinneras, 2002)
	0.51	1.77	(Goldblith and Wick, 1961)
	0.65-0.87	7.76-8.92	(Calloway and Margen, 1971)
	0.5	3.8	(Meinzinger and Oldenburg, 2009)
	0.69-2.5	4.80-9.86	(Chaggu, 2004)
	0.9-2.7		(Wignarajah et al., 2003)
Total K	0.20-0.24	1.78-2.14	(Calloway and Margen, 1971)
	0.47	3.10	(Goldblith and Wick, 1961)
	0.75-0.88		(Wignarajah et al., 2003)
	0.8	4.936	(Eastwood et al., 1984)
	0.8-1.0		(Kujawa-Roeleveld and Zeeman, 2006)
	0.7	3.3	(Meinzinger and Oldenburg, 2009)
	0.8-2.1	2.712	(Chaggu, 2004)
	1.48-2.52	7.16	(Vinnerås et al., 2006)
Na	0.12	0.80	(Goldblith and Wick, 1961)
	0.8 (0.3-4.1)	4.94	(Eastwood et al., 1984)
Ca	0.1-1		(Wignarajah et al., 2003)
	2.9-3.6		(Chaggu, 2004)
	0.53		(Kujawa-Roeleveld and Zeeman, 2006)
	0.61	3.77	(Eastwood et al., 1984)
	0.64	4.27	(Goldblith and Wick, 1961)
	0.96-1.12	2.68	(Calloway and Margen, 1971)
Mg	0.15	0.93	(Eastwood et al., 1984)
	0.18		(Kujawa-Roeleveld and Zeeman, 2006)
	0.20	1.33	(Goldblith and Wick, 1961)
	0.30-0.34	2.86	(Calloway and Margen, 1971)
Cl	0.09	0.6	(Goldblith and Wick, 1961)
S	0.13	0.87	(Goldblith and Wick, 1961)
	0.2		(Meinzinger and Oldenburg, 2009)
	(mg/cap/day)	**(mg/kg)**	
Cu	1.02	6.8	(Goldblith and Wick, 1961)
	1.10		(Kujawa-Roeleveld and Zeeman, 2006)
	1.5-2.1		(Wignarajah et al., 2003)
Fe	30	200	(Goldblith and Wick, 1961)
	700-1000		(Wignarajah et al., 2003)
Pb	0.03-0.07	0.12-0.27	(Schouw et al., 2002)
	0.02-0.03		(Hansen and Tjell, 1979)
	1.26	6.38	(Vinnerås et al., 2006)
Mn	24-90		(Wignarajah et al., 2003)
Mo	2-4		(Wignarajah et al., 2003)
Zn	7.85	48.46	(Eastwood et al., 1984)
	5-10		(Wignarajah et al., 2003)
	10.68		(Kujawa-Roeleveld and Zeeman, 2006)
	13.31	67.49	(Vinnerås et al., 2006)
Ni	0.08-0.09		(Hansen and Tjell, 1979)
	0.3	1.52	(Vinnerås et al., 2006)
	0.3	1.15	(Schouw et al., 2002)
Cr	0.02-0.03		(Hansen and Tjell, 1979)
	0.08	0.31	(Schouw et al., 2002)
	0.18	0.91	(Vinnerås et al., 2006)
Cd	0.07	0.27	(Schouw et al., 2002)
	1.26	6.39	(Vinnerås et al., 2006)
Hg	0.007	0.04	(Vinnerås et al., 2006)

2.3.3 Chemical nature

Faecal pH is neutral with a median value of pH 6.6 and a range of mean pH values of 5.3-7.5 (n=28) (Figure 2.5). Faecal pH not only varies between different populations but has also been proven to differ between individuals consuming the same diet and with time (Silvester et al., 1997).Van Dokkum et al. (1983) found a difference of 0.25 in the faecal pH between sampling separated by two days in the same individual when exactly the same diet was consumed.
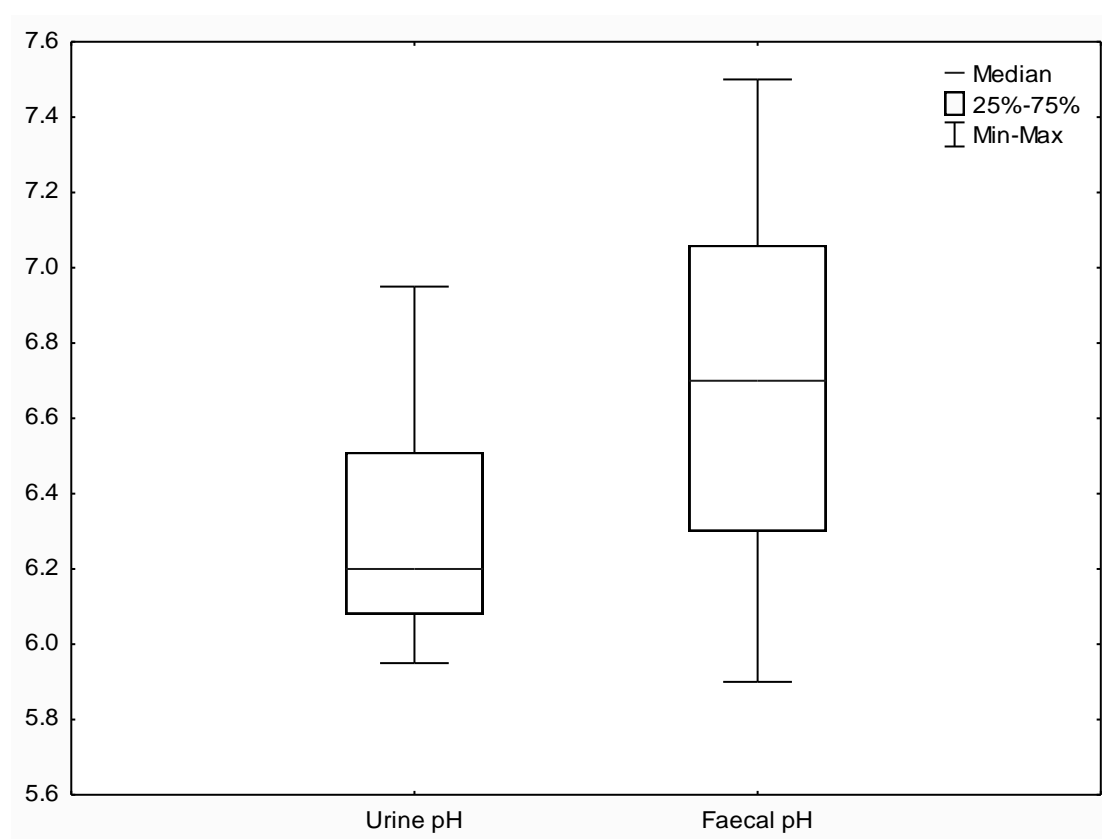
Figure 2.5 Mean pH values for urine (n=9) and faeces (n=23)

Faecal pH variation is related to diet (Thornton, 1981; Van Dokkum et al., 1983). Increased dietary fibre was suggested by Newmark and Lupton (1990) to lower faecal pH. However, not all studies have found that high fibre diets correlate with lower faecal pH. In a comparison study of omnivorous and vegetarian diets by Walker and Walker (1992), no significant difference in pH values for the stool or stool water were observed, even though the vegetarian

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diet provided considerably more fibre. Similarly in a comparative study of omnivorous and vegetarian diets by Van Faassen et al. (1993) no difference between pH values for the stool or the stool water were observed, even though the vegetarian diet again, provided considerably more fibre.

High levels of resistant starch in diets was also shown by Phillips et al. (1995) to lower faecal pH in a controlled experiment of differing resistant starch intakes, a significant inverse relationship between resistant starch intake and faecal pH was found ($r = -0.65$, $P < 0.01$). Interestingly 30% of the variance of faecal pH in a study by Van Dokkum et al. (1983) was accounted for by calcium intake, showing a significant positive correlation. Evidence of variation in faecal pH is not conclusive and variation could be due to a specific dietary intake, such as citrus fruit which has been proven to lower faecal pH (Walker et al., 1979).

2.3.4 Physical form

For the development of on-site treatment technologies an understanding of the physical form of faeces is important; this characterisation can be done through the use of visual scales or prevalence rates of diarrhoea and constipation.

2.3.4.1 Visual scale

Within the medical literature a number of linear scales have been used to characterise faeces e.g. Davies et al. (1986), however, with different scales in use cross comparison of studies is difficult. The most popular scale used is that of Lewis and Heaton (1997) who proposed the "Bristol Scale Stool Form" (Figure 2.6). This simplified visual scale provides an indication of the form of faeces expected and the variation that can be observed across a population. Stool form is considered abnormal when type 1, 6 and 7 occurs and this is 15% of the time within a healthy population (Heaton et al., 1991). The mean value for a general population sample of 66 people using the Bristol Stool Form scale have been placed at 3.6 by Lewis and Heaton (1997). The distribution of the physical form in two populations of differing countries shows that stool types 3 and 4 are most commonly reported (Figure 2.6). Variation

occurs between individuals, by age and gender (Heaton et al., 1992), although diet and health prove more important variables (Davies et al. 1985; Heaton et al. 1991). Dietary fibre is linked to stool texture, as dietary fibre increases stools become softer (Davies et al., 1986).
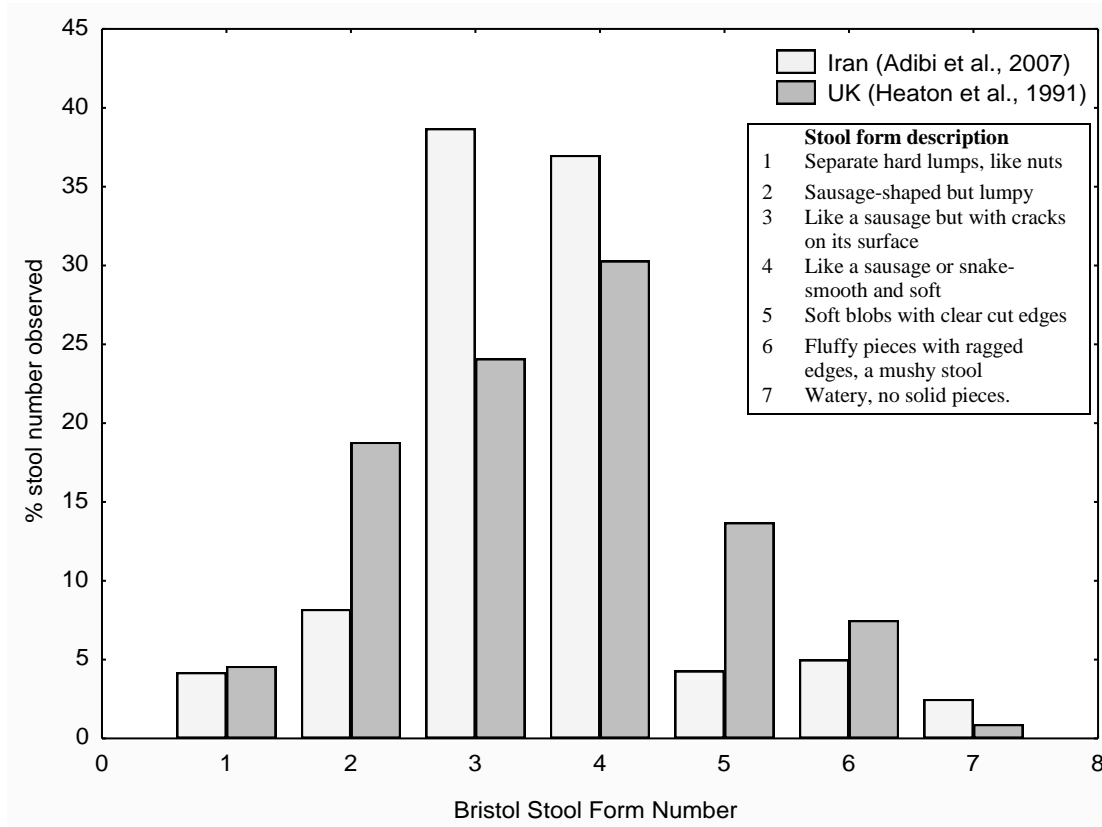
Figure 2.6. Data from two separate studies of healthy subjects (Heaton et al (1991); Adibi et al. (2007)) both use the Bristol Stool Form scale. Stool types 3 and 4 make up the most common stool type in both studies, however all types of stool are recorded in both studies.

2.3.4.2 Diarrhoea

Diarrhoea has an impact on stool production, structure, form and composition. In a controlled study by Wierdsma et al. (2011) it was found that patients in an intensive care unit with diarrhoea had over 5 times the wet faecal weight (796g/cap/day versus 157g/cap/day) compared to those without diarrhoea. Increased water losses are the predominant cause of the increase in weight; an increase in water content of 5% was shown by Wierdsma et al. (2011) and

in a study by Goy et al. (1976) faeces of patients with diarrhoea had a significantly (p<0.05) greater percentage water content compared to control subjects. Faecal water loss of more than 10 ml/kg body weight is often used as a definition of chronic diarrhoea (Auth et al., (2012). Those with diarrhoea display higher faecal protein losses of 16.2 g/cap/day versus 5.6 g/cap/day and higher faecal energy losses were also shown in comparison to patients with normal stools (Wierdsma et al., 2011). However, faecal energetic content per gram of faeces (kcal/g wet faeces) was not significantly different between subjects with and without diarrhoea (Wierdsma et al., 2011).

Diarrhoea is defined as a minimum of 3 liquid stools per day; it is further sub-divided into acute diarrhoea (defined as diarrhoea lasting up to 3 weeks) and chronic diarrhoea (lasting any longer than 3 weeks) (Patel and Thillainayagam, 2009). It has been classified as stool types 6 and 7 on the Bristol Stool Form Scale (Figure 2.6). Chronic diarrhoea prevalence rates in five studies across the UK, US and Asia show an average of 4.6% (Table 2.7) with prevalence more frequent in the elderly at rates of 14.2% (Talley et al., 1992). Acute (infectious) diarrhoea is caused most commonly by viruses, bacteria and protozoa and is commonly transmitted by the faecal-oral route through water, food and person to person contact (Farthing and Kelly, 2007). Acute diarrhoea prevalence figures have been applied to geographic areas, such as in the United States where there is an equivalent of 1.4 episodes per person per year (Herikstad et al., 2002) and in the UK with just under 1 episode per person per year (Feldman and Banatvala, 1994).

Table 2.7 Diarrhoea prevalence in a selection of 6 countries

Study	Country	n	Chronic Diarrhoea Prevalence %
Han et al. (2006)	Korea	1066	6.6
Chen et al. (2000)	Singapore	271	7
Danivat et al. (1988)	Thailand	1077	2.3
Danivat et al. (1988)	UK	301	4.7
Sandler and Drossman (1987)	UK	1128	3.6
Danivat et al. (1988)	USA	789	4.9
Tan et al. (2003)	Malaysia	84	3
Average across studies		7	4.6

Acute diarrhoea prevalence is higher in low income countries as many of the risk factors of contracting diarrhoeal illness are associated with poor socioeconomic conditions (Ahs et al., 2010). Factors that increase exposure to infectious diarrhoea include lack of access to safe water supplies, inadequate sanitation facilities and poor personal hygiene. Added to this factors that reduce resistance to infection are also important such as age, malnutrition and illnesses such as the human immuno-deficiency virus (HIV) (Ahs et al., 2010). Geographically, there is an overlap of areas with a large burden of diarrheal illness and those with a large proportion of HIV cases; some enteric pathogens have also been shown to occur more frequently in HIV-positive individuals than in the general population, including Campylobacter, Cryptosporidium and Shigella (Ahs et al., 2010). Zinc and Vitamin A deficiencies have also been shown to increase susceptibility to diarrhoea episodes, especially in children (Walker and Black, 2004).

Diarrhoea disproportionately affects children in low and middle income countries due to inadequate water and sanitation facilities and nutritional risk factors (Fischer Walker et al., 2012). In a systematic review by Fischer Walker et al. (2012) diarrhoea prevalence rates in children were estimated at 2.9 episodes/child year, with incidence rates the highest among infants aged 6-11 months. In an overview report by the World Bank, data collected by a Demographic and Health Survey (DHS) project between 1990-2005 was presented by Gwatkin et al. (2007) with prevalence measured according to the % of children under 5 who had diarrhoea in the 2 weeks prior to the survey; population averages for the regions of South Asia (15.3%), Sub-Saharan Africa (19.7%), East Asia and the Pacific (13%) were recorded (Gwatkin et al., 2007). Infectious diarrhoea is also more common among elderly populations due to increased incidence of immunodeficiency and resultantly an increased likelihood of bacteria in the blood (DuPont, 1997).

Seasonality affects the prevalence rates of diarrhoea. It has been observed that acute diarrhoea becomes an epidemic in the rainy season in places such

as Kathmandu (Karki and Tiwari, 2007) this is largely due to the problem of water supply contamination. However, in a cross sectional study of diarrhoea in children under 5, a negative association between rainfall and diarrhoea rates was found by Lloyd et al. (2007) with a 4% increase in diarrhoea incidence (95 % confidence interval, CI: 1-7 %, p = 0.02) for each 10 mm month^{-1} decrease in rainfall, this was thought to be due to the use of unprotected water sources during water scarcity.

2.3.4.3 Constipation

Constipation has prevalence rates that can range from 1.9%-27.2% in an American population (Higgins and Johanson, 2004); however, it is commonly found at 6-12% in a general population (Heaton et al., 1992; Talley et al., 1993; Thompson et al., 2000). Constipation increases with increasing age, particularly after the age of 65 (Higgins and Johanson, 2004). Only one comparative study (Aichbichler et al., 1998) of faecal characteristics of constipated and non-constipated subjects was found; concluding that stool weight per week was markedly reduced in constipated subjects due to a reduction in stool water and total solids output. There are numerous other studies that report faecal weights of constipated subjects, e.g. (Ashraf et al., 1996; Chen et al., 2008) these studies report daily per capita weights that fall within the study range presented (for example in a study of constipated subjects by Chen et al. (2008) values of 108.3 g/cap/day were recorded, in comparison to the median value of 128 g/cap/day reported in this study); however, shorter experimental studies can often be misleading and it is often the case that over prolonged study periods of weeks or even months stool weights can be considerably decreased (Aichbichler et al., 1998).

2.3.5 Urine

In contrast to faeces, the characteristics of urine have been studied extensively (Diem and Lentner, 1970; Kirchmann and Pettersson, 1994; Karak and Bhattacharyya, 2011). Urine as a potential fertiliser has attracted much attention in the treatability sector with a large range of literature exploring the agricultural fertiliser potential (Palmquist and Jönsson, 2004; Karak and

Bhattacharyya, 2011; AdeOluwa and Cofie, 2012). Urine presents less danger to human health in comparison to faeces and contains few enteric microorganisms, however, some human pathogen microorganisms such as *Schistosoma haematobium, Salmonella typhi, Salmonella paratyphi and Leptospira interrogans* as well as helminth eggs can be found in the urine fraction (Feachem et al., 1978; Heinonen-Tanski and van Wijk-Sijbesma, 2005).

2.3.5.1 Liquid generation

Human urine is a liquid that is secreted by the kidneys, collected within the bladder and excreted through the urethra. Urine is composed of 91-96% water (Drangert, 1998; Höglund et al., 2000; Heinonen-Tanski et al., 2007) and the remainder can be broadly characterised into inorganic salts, urea, organic compounds and organic ammonium salts (Putnam, 1971).

Liquid generation from humans is dependent on the water balance of individuals. Liquid output is in the form of urine, faecal water, from the skin through sweating and from the lungs through respiration. A median volume of 1.4 litres/cap/day urine is excreted with mean values ranging from 0.6-2.6 L/cap/day (n=14). In medicine, urine output is used to assess circulatory adequacy with inadequate urine output considered at <0.5 mL/kg body weight/hour for adults (Suen et al., 1998) and at 1-1.5mL/kg body weight/hour in children (Yowler and Fratianne, 2000). This indicates the minimal urine output that can be expected.

Variation in total urine output (Table 2.10) is primarily due to fluid intake and in a study by Parker and Gallagher (1992) accounted for 78% of the variation observed in a sample of 11748 days' worth of data. It was noted by Garrow et al. (1993) that the volume of water drunk as fluid is generally equal to the volume of urine produced. Body size is inevitably important when assessing a human's urinary output; when assigning loading rates in wastewater, Almeida et al. (1999) reduced urinary output by 33% for children such that Karak and Bhattacharyya (2011) stated that children urinate about half that of the volume excreted by adults. Urine output therefore increases with body size. Other

factors leading to variation such as excessive exercising or sweating will have an effect on the quantity of urine generated as they will impact hydration. Variation in urine output according to race has been proven significant with the urine volume of black women 0.24 L/day less than white women (p=0.001) (Taylor and Curhan, 2007). It was also observed by Clark et al. (2011) that higher volumes of urine tended to be from subjects who were older, were more likely to be obese or taking medication.

Information regarding the number of times urination takes place over a 24 hour period is sparse and is likely to vary greatly due to fluid intake, biological factors and health of the individual. Schouw et al. (2003) recorded a figure of 5.4 urinations per day in a boy's prison in Thailand and Bael et al. (2007) reported a median figure of 6 urinations/24 hours (range of 2-11 urinations/24 hours) in a study of children aged 6-12 years. A figure of 8 urinations per 24 hour period was recorded for a population sample in the United States (n=17) (Clare et al., 2009). The diurnal variation of urinary output is not commonly recorded, however, a control sample of 15 healthy adult subjects showed that 60% of total urine volume was excreted during the daytime (09:00-21:00) and 40% was excreted at night time (21:00-09:00) (Hineno et al., 1994).

2.3.5.2 Composition

Urine composition varies due to differences in physical exercise, environmental conditions as well as water, salt and high protein intakes. Urine osmolarity is a measure of the water distribution amongst fluid components. It can vary between 50-1200 mOsmol/kg, with the average urinary excretion of solute 1000 mOsmol/cap/day (Garrow et al., 1993; Callis et al., 1999; Callis et al., 1999). This solute is excreted in a median volume of 1.4 L/cap/day of urine. The quantity of solute varies between individuals and with differing diets; for example the high consumption of meat leads to larger volumes of solutes as meat is a major source of urea (the largest solute fraction) as well as potassium and phosphates, whereas vegetarian diets are likely to lead to reduced solute production as most energy is derived from carbohydrate (Garrow et al., 1993).

The median value of mean total urine solids loading rates is 59 g/cap/day (n=7) and mean values range from 57-64 g/cap/day. The dry matter of urine was measured at 4.7-10.4 g/L by Heinonen-Tanski and van Wijk-Sijbesma (2005). The concentration of total suspended solids has been recorded at 21 mg/L (Almeida et al., 1999) and total dissolved solids have been recorded at 31.4 mg/g (Putnam, 1971). Organic matter makes up between 65-85% of urine dry solids (Strauss, 1985), with volatile solids comprising 75-85% of total solids (Fry and Merrill, 1973; House, 1981). Urea is the most predominant constituent making up over 50% of total organic solids, and is produced through the metabolism of protein. The other major solutes excreted in urine are Na and K, which are largely derived from dietary intake.

2.3.5.3 Chemical composition

Dry urine solids are composed of 14-18% N, 13% C, 3.7% P and 3.7% K (Strauss, 1985). Concentrations of major elements in urine were recorded at 6.87 g/L carbon, 8.12 g/L nitrogen, 8.25 g/L oxygen and 1.51 g/L hydrogen by Putnam (1971). Of the faeces and urine fractions, urine contains the largest proportion of N (90%), P (50-65%) and K (50-80%) released from the body (Heinonen-Tanski and van Wijk-Sijbesma, 2005).

Nitrogen is predominantly in the form of organic nitrogen and mostly in the form of urea (Beler-Baykal et al., 2011). Median values of total N excretion of 11 g/cap/day were recorded (n=8) with a range of mean values from 2-35 g/cap/day. Endogenous total N excretion of 13 men with the absence of protein in the diet was 2.41 g/cap/day, with no correlation with body weight found (r=0.450) (Calloway and Margen, 1971). This therefore provides a minimum figure for N excretion. The dietary intake of protein is the most predominant factor effecting N excretion. Urinary N components increase with increasing levels of protein in the diet; a positive correlation (r^2) between urinary N and protein intake (intake ranging from 51-212 g/day) was found to be 0.91 (Magee et al., 2004). In a meta-analysis of data by Kipnis et al. (2001) it was found that urinary N is 80% of dietary intake on average

Table 2.8 Per capita generation of components in urine

Variable	Range (median) (g/cap/day)	Reference
Total N (n=8)	2-35 (11)	
Urea	10.00-35.00	(Bender and Bender, 1997)
	1.36-6.77	(Calloway and Margen, 1971)
Ammonia	0.34-1.2	(Bender and Bender, 1997)
Creatine	0-0.15	(Dong, 1999)
	<0.10	(Bender and Bender, 1997)
Creatinine	0.001-0.002	(Bender and Bender, 1997)
	1.640	(Dong, 1999)
	1-1.800	(Harper et al., 1977)
Uric Acid	0.25-0.75	(Bender and Bender, 1997)
	0.86	(Dong, 1999)
	0.50-0.80	(Harper et al., 1977)
Total P	0.93	(Jönsson et al., 2005)
	0.62-0.74	(Taylor and Curhan, 2006)
	0.45-0.71	(Borawski et al., 2008)
	1.15-1.30	(Calloway and Margen, 1971)
Total K	0.78-2.50	(Wignarajah et al., 2003)
	2.5	(Del Porto and Steinfeld, 1999)
	0.027-0.036	(Borawski et al., 2008)
	2.51-2.87	(Calloway and Margen, 1971)
Na	3.45-4.53	(Wignarajah et al., 2003)
	0.082-0.163	(Borawski et al., 2008)
SO_2-4	1.34-1.63	(Taylor and Curhan, 2006)
Ca	0.20-0.50	(Wignarajah et al., 2003)
	0.118-0.113	(Taylor and Curhan, 2006)
	0.057-0.134	(Borawski et al., 2008)
	0.14-0.25	(Calloway and Margen, 1971)
Mg	0.19-0.21	(Calloway and Margen, 1971)

Of the nitrogenous fractions urea is the most predominant, making up between 75-90% (Lentner, 1981). Urea concentrations range from 9.3-23.3 g/L (Putnam, 1971; Otterpohl et al., 2002; Jönsson, 2005), with daily loadings of 1.4-35.0 g/cap/day (Calloway and Margen, 1971; Bender and Bender, 1997). Creatinine is a significant nitrogenous fraction in urine. Endogenous creatinine was measured at 1.59 g/cap/day and was correlated with body weight (22 ±4 mg/kg, r =0.918) and is also dependent on age and muscle mass (Calloway and Margen, 1971). Concentrations can vary according to gender with male subjects recording higher (P=0.001) creatinine values than female subjects, 1.9 and 1.4 respectively (Newman et al., 2000). Concentrations of creatinine in urine also decreases when increasing volumes of urine are excreted over a 24 hour period (R^2= 0.618, r=0.786, P<0.001)

(Newman et al., 2000). If there has been incomplete sampling over 24 hours an internal standard against the creatinine value can be used, with standards of creatinine excretion set at 1.7 g/d in men and 1.0 g/d in women (Jackson, 1966). Nitrate concentrations in urine are low, with measured values at 1.07 mmol/L and 2.06 mmol/day when a high protein diet is consumed (192 g/d) and 1.09mmol/L and 2.23mmol/day when a lower protein diet is consumed (68g/d) (Silvester et al., 1997).

Protein intake is the predominant cause for variation in nitrogen concentrations of urine. In addition to this, protein intake has also been shown to impact other mineral constituents in urine. For example, in very low protein diets P and K were shown to be increased, Ca was reduced in very low protein diets but protein intake had no effect on Mg concentrations in urine (Calloway and Margen, 1971).

Differences in chemical composition have been observed according to race by Taylor and Curhan (2007) with black women (n=146) excreting 65 mg less Ca (P<0.001), 351 mg less K (P<0.001), 11 mg less Mg (P<0.001) and 120 mg less P (P<0.001) per day than white women (n=330); these observations were consistent even after adjustment for age and Body Mass Index (BMI). Animal protein in the diet has been shown to lead to increased levels of urinary calcium, with calcium excretion at 21% of intake whereas with higher levels of vegetable protein calcium excretion is 16% of intake (Taylor and Curhan, 2007). Positive associations were found between BMI and urinary calcium excretion, however, it was concluded that this was due to differences in animal protein and sodium intake (Taylor and Curhan, 2006).

Table 2.9 Concentration of key components in fresh urine

Variable	Concentration Range (mg/L)	Reference
Electrical Conductivity EC	160 mS/cm	(Jana et al., 2012)
	270 mS/cm	(Jönsson et al., 1997)
Osmolarity	1025 mosmol/kg	(Callis et al., 1999)
	50-1200 mosmol/kg	(Garrow et al., 1993)
COD	17500	(Putnam, 1971; Almeida et al., 1999)
	6270-10600	(Putnam, 1971)
Total N	8000	(Ban and Dave, 2004)
	5000	(Jönsson et al., 2004)
	11000-13900	(Jönsson et al., 2004; Southgate and Durnin, 1970)
	4000	(Jönsson et al., 1997)
	12000	(Mojtahedi et al., 2002)
	11700	(Beler-Baykal et al., 2004)
TKN	9220	(Beler-Baykal et al., 2011)
	5580-7900	(Putnam, 1971)
Urea	21400	(Jönsson, 2005)
	9300-23300	(Putnam, 1971)
	10000	(Otterpohl et al., 2002)
NH_4-N	125	(Jana et al., 2012)
	600	(Beler-Baykal et al., 2004)
NH_3-N	480	(Tilley et al., 2008a; Diem and Lentner, 1970)
	200-730	(Putnam, 1971)
	300	(Tilley et al., 2008c)
Total P	350	(Jönsson et al., 1997)
	800-2500	(Wignarajah et al., 2003)
	1000	(Del Porto and Steinfeld, 1999)
	1800	(Ban and Dave, 2004)
PO_4-P	205	(Tilley et al., 2008c; Diem and Lentner, 1970; Jana et al., 2012)
	450	(Tilley et al., 2008c)
	760	(Diem and Lentner, 1970)
K	966-1446	(Beler-Baykal et al., 2004)
	1200	(Jönsson et al., 1997)
	750-2610	(Putnam, 1971)
Ca	230	(Diem and Lentner, 1970)
	32	(Jana et al., 2012)
	70	(Tilley et al., 2008c)
Mg	120	(Diem and Lentner, 1970)
	70	(Tilley et al., 2008c)
Creatine	0-890	(Putnam, 1971)
Creatinine	311-2150	(Putnam, 1971)
Uric Acid	40	(Putnam, 1971)
	152-858	(Jen et al., 2002)
	856	(Dong, 1999)

3.6.4 CHEMICAL NATURE

The pH of fresh urine is largely neutral with a median of pH 6.2, with a range of mean pH values of 5.5-7.0 based on a large subject sample size across nine individual studies (Figure 2.5). There are numerous factors that can lead to changes in urinary pH but diet once again provides a key variable. Urinary pH is reduced by high protein intake through meat and dairy produce as well as through alcohol consumption (Kanbara et al., 2012). However urine is more alkaline with the ingestion of potassium and organic acids which are increased in diets with high consumption of vegetables and fruit. Taylor and Curhan (2007) found that black women had a higher urinary pH than white women by 0.11 units (p=0.03) even when adjusted for differences in diet, BMI and age. Further, an inverse relationship between BMI and urine pH (p=0.02) was found by Taylor and Curhan (2006). Factors leading to a lower urinary pH include a higher weight, old age and increased dietary acid intake (Hesse et al., 1986; Maalouf et al., 2004; Taylor and Curhan, 2007).

The specific gravity of urine ranged from 1.002-1.037 in spot samples of 534 subjects (aged 18-68) with a high correlation (r=0.82, P<0.001) observed between creatinine and specific gravity (Carrieri et al., 2000). The COD levels of 8-17 g/L found in urine are low (Table 9); this is likely to be because most of the organics excreted are small molecules. The mean calorific content of urine was measured at 100 kcal/day (range: 91-117) by Southgate and Durnin (1970): using the median value of urine solids produced daily (59.0 g/cap/day) a design value of 1707 kcal/kg can be used.

2.3.6 Additional influences on treatment systems

Both faecal solids (29 g/cap/day) and urine solids (58-64 g/cap/day) are produced daily in large quantities. A mixed stream treatment system at source will therefore have to deal with a large quantity of solids from both faeces and urine. However, it is also the case that faeces and urine are likely not to be the only additions to a treatment system. A treatment system may also have to deal with additional material from human behavioural practices such as the use of toilet paper or the addition of sanitary items (Table 2.10). A similar principle applies to water addition; a large liquid

fraction is produced daily through urine and faecal output; however this may be further increased by additional water inputs such as pour flush toilet systems or anal cleansing practices.

Table 2.10 Components and generation rate of human excreta waste streams and possible additional inputs.

Component of solids fraction	Generation rate (g/cap/day)	Component of liquid fraction (L/cap/day)	Generation rate
Stool mean (range) g/cap/day	32 (4-102)	Stool water mean (range)	0.101 (0.053-0.265)
Urine	61 (50-75)	Urine median (range)	1.42 (0.8-2.45)
Toilet paper use average	11.68-19.4 [bc]	Anal cleansing L/wash	0.35-3 [de]
Toilet paper use men	6 -10.3 [abc]	Pour flush toilet water L/flush	1-3 [f]
Toilet paper use women	17.9 -36 [abc]		
Menstrual pads and flow	34 [a]		
Sanitary Items. refuse item/cap/day	0.16 [b]		

[a] (Parker and Gallagher, 1992) [b] (Friedler et al., 1996) [c] (Almeida et al., 1999) [d] (Strauss, 1985) [e] (Tilley et al., 2008a) [f] (Cairncross and Feachem, 1993)

2.4 DISCUSSION

Existing OSS facilities are often poorly designed, constructed and maintained which regularly results in inadequate sanitation facilities in many low income regions. This problem has given rise to research into the on-site treatment and/or resource recovery from faeces and urine within a low income context. This trend has accelerated with the challenge presented to researchers by the Bill and Melinda Gates Foundation to 'Reinvent the Toilet' (Global Development Program, 2013). A large proportion of this research aims to treat faeces and urine as a fresh waste stream on the site of production, giving a need to understand the production, composition and any variation around these factors in order to determine how this may impact these technologies. In this discussion all types of conventional treatment processes were considered alongside recent research funded by the Bill and Melinda Gates Foundation (BMGF). These grants (Sustainable Sanitation Alliance, 2013) were grouped according to their treatment pathways comprising; biological processes (17), physical separators (7), chemical processes (3) and thermal processes (8) (Table 2.11). The principle aim of this discussion is to understand how the production rates, physical and chemical composition of faeces and urine can

lead to an improved understanding of potential treatment pathways that are either currently in use or under development in the OSS technology sector.

Table 2.11 Classifications of broad treatment pathways in wastewater treatment

Process Type	Examples	Resource Recovery
Biological	Anaerobic digestion	Biogas
	Decoupled HRT and SRT	Digestate/Biosolids/liquid fraction
	UASB	Biofuel production
	Wet and dry composting	Compost fertiliser
Thermal Processes	Pyrolysis/gasification	Energy/Char
	Incineration	Energy/Ash
Separation	Biofiltration	Pathogen free water
	Membrane pervaporation	Irrigation water
Chemical Processes	Electrochemical disinfection	Pathogen free products
	Ammonia disinfection	NPK irrigation water/fertiliser
	Struvite	Phosphorus
	Ammonia stripping	Fertiliser
	Biochemical Fuel Cells	Electricity

2.4.1 Biological processes

The predominant factors likely to impact biological processes to the greatest extent are solids loading, energy content, protein and fat concentration in the faeces and the high urea concentrations in urine.

The high solids loading rate associated with fresh faeces (~25% wt.) when viewed as an individual waste stream presents a potential barrier to the successful implementation of high rate anaerobic systems in relation to their solids handling and rheological impacts on mixing and pumping (Speece, 2008). Accordingly, high solids anaerobic digestion processes (operating with solids concentrations greater than 15% w/w) represent a more appropriate match due to the significantly lower impact associated with mixing. Operation at the higher solids loadings will translate to smaller reactor volumes, lower energy requirements and less material handling than traditionally encountered with standard anaerobic digestion (Guendouz et al., 2008) but would most likely result in a reduced rate and lower biogas yields. For biological processes such as aerobic composting the optimum moisture content is 30-60% (Liang et al., 2003): the moisture content of faeces was greater than this (75%) increasing the potential for anaerobic conditions to develop due to water logging (Tiquia et al., 1996). Therefore, incorporation of dewatering pre-treatment or a co-

composting feedstock should be considered in order to establish resilient conditions to maximise the efficacy of the desired aerobic degradation pathways. Importantly, the fluctuating levels of moisture content reported in faeces (63-86%) means that amendment strategies need to be appropriately flexible and robust and are likely to require a degree of bespoke commissioning.

Based on the COD values collected in this study each 66 g/cap/day COD added and removed by a digester could theoretically produce $0.0175m^3$ of methane at standard temperatures and pressures (Grady et al., 1999). Practical delivery of such potential is dependent on anaerobic reactor type, retention time and biodegradability such that actual conversion of the available organic matter to biogas is expected to range between 40-90% (Mang and Li, 2010). For instance, a key variable is associated with the fibre content of faeces which was found to vary widely (Figure 2.4); especially in populations consuming high fibre diets (such as diets consumed in low income countries). The importance of this relates to the relatively lower biodegradation rate of the fibrous material resulting in reduced COD conversions. Importantly, increased wet mass production rates above the average (128 g/cap/day) are commonly associated with increased levels of indigestible fibre in the faeces. Accordingly there is a poor correlation between wet mass loading and energy production. Whilst this places a risk of overestimation during design for such systems the impact can be readily accounted for as the fibre content of faeces is directly dependent on the non-degradable fibre intake of the population within the associated catchment. Consequently, the fibre composition of faeces for a given population can be predicted if diet is known and accounted for in such calculations.

Potential biogas production from faeces could therefore be significant, however, the relatively small quantities of solids produced per cap/day should be noted and may mean that in order for significant quantities of methane to be produced a large population would be required or an additional co-digestion feedstock. This factor is likely to be problematic to small household or community anaerobic digester designs that cite methane production as a key driver for gaining energy neutral systems or for additional cost recovery.

The efficacy of biological processes for the treatment of faeces and urine, in either aerobic or anaerobic processes, may be inhibited through imbalances in the macro nutrient composition of such streams. For instance, anaerobic digestion proceeds optimally when the C: N ratio is around 20:1 to 30:1 (Parkin and Owen, 1986); this is not the case in faeces (8:1), urine (0.8:1) or as a combined waste stream (2.3:1). Similarly, in aerobic systems the recommended ratio for C:N:P (100:10:1 to 100:5:1) (Tchobanoglous et al., 2003) would not be reached. However, imbalances in the macro nutrient composition could be rectified through the use of organic waste substrates that are frequently locally available and could be a simple means of increasing the viability of biological systems.

Potential chronic toxicity for treatment by anaerobic processes can be assessed according to the moderately inhibitory and strongly inhibitory concentration classifications according to Parkin and Owen (1986). Faeces as a single waste stream showed concentrations of Na^+, K^+, Ca^{2+}, Mg^{2+} that were of moderately inhibitory concentrations with values of K^+ reaching levels defined as strongly inhibitory on occasions. Toxic metals such as Cu, Ni, Cr and Pb were not of significant concentrations to inhibit anaerobic processes of a faeces waste stream. However, the high concentrations of sulphide reported have the potential to exhibit toxicity to methanogenic bacteria (Speece, 2008); this will only occur when high levels of sulphate are entering digesters along with sulphate reducing bacteria. Relatively high levels of sulphate 1.34-1.63 g/cap/day were recorded in urine but with very small amounts of elemental Sulphur found (0.16 g/cap/day) in the faeces fraction.

Nitrogen excreted in urine and voided in faeces was shown to vary according to diet (primarily levels of protein intake) and combined median daily losses (13 g/cap/day) could have the potential to lead to ammonia toxicity problems. Ammonia (NH_3-N) concentration is a function of ammonium (NH_4-N) concentration, temperature and pH (Speece, 1996); thresholds in anaerobic systems can be found at concentrations of 100-500 mg/L depending on adjustment time (Metcalf and Eddy, 2004). Measurements of ammonia in faeces are within this range (204-409 mg/kg), although a significant proportion of protein (29 g/kg) was found in the faeces fraction that will degrade to produce additional ammonia, dependent on storage time and

conditions. The addition of urine to this waste stream (urine comprises 80% of total N losses) could lead to ammonia threshold limits being exceeded in an undiluted waste stream. This is because a large proportion (> 80%) of the nitrogenous fraction of urine is in the form of urea, which in turn breaks down into ammonia. Therefore, ammonia toxicity (resulting from urea toxicity) is likely to be problematic when faeces and urine are treated as a combined waste stream and significant dilution could be necessary. Toxicity from the urine fraction could have negative impacts on biological systems as relatively large volumes of urine are collected in relation to faeces (daily urine: faeces ratio on a weight basis of 11:1). Accordingly it is suggested that smaller household systems that treat a combined faeces and urine waste stream need to especially consider such issues and may be enhanced through inclusion of source separation. Source separation could be carried out through the use of urine diverting toilets in which the faeces and urine fractions are collected separately within the toilet bowl.

2.4.2 **Physical separators**

There are numerous different types of separating technologies; however, the majority are likely to be predominantly influenced by variation in the solids content, physical form as well as levels of protein and fat in faeces.

For technologies based on separation the lack of a standard faeces shape, structure and water content may be one of the greatest challenges. This could impact bound water removal from different stool types and also the different particle sizes that make up faeces. This uncertainty could be problematic when selecting process types and optimisation operating conditions. In addition to this faeces show a low proportion of fixed to volatile solids which could make dewatering challenging and require the addition of increasing amounts of chemicals or conditioning agents in order to gain adequate separation without pre-treatment.

Significant levels of protein in the faeces fraction (29 g/kg) and the potential for fluctuations in this value (range of 19 to 122 g/kg) may be unfavourable to separation processes such as membrane and other surface filter systems. Layers of protein that form on the outside of particles could lead to clogging and its deposition and adhesion to membrane surfaces may cause fouling (Chan and Chen, 2004).

Similarly fat can be problematic to separation technologies as it can act as a binder for particles (Nguyen et al., 2012). Fat content in faeces shows variation across studies (Figure 2.4) but remains within a narrow region (5.8 to 49.1 g/kg). The concentration of fat in faeces (median of 25 g/kg) is comparatively low in comparison to conventional types of wastewater sludge such as primary sludge which has much higher levels of fats, oils and greases: this is usually due to the discharge of these products in the sewage system. Nevertheless, shock loads due to variation in the fat content of faeces may be large enough to cause the clogging of pores and impact dewatering properties.

Information regarding the physical structure immediately after voiding provides an indication as to how the structure of faeces may change over short time periods, for example in the Bristol Stool Form scale a number of 1 or 2 would suggest a faeces structure that holds its shape to a much greater extent than others in the scale. Studies were found regarding the settling and thickening of excreta from septage and public toilet tanks (Heinss et al., 1999) but in this review no studies were found regarding the change in the physical structure of faeces once voided over shorter time scales. This lack of data regarding the change in physical structure over time is limiting current ability to fully understand technology needs. Importantly, the time required to lose the initial consolidated identity of the fresh faecal material is required to understand the potential virtue of utilising fast separation processes that could benefit from the initial cohesion of the solid material. However, such development must also take into account looser faecal material that will also enter such systems and is likely to be significantly less effectively removed by physical processes. Accordingly, understanding the kinetics of the structural change in faecal material during the initial periods after generation remains a critical area for future research activity that could inform novel low cost technology development.

2.4.3 Chemical processes

Chemical treatment processes can be wide ranging and are dependent on the end use and initial purpose of treatment and include processes such as chemical precipitation, disinfection, oxidation, neutralisation and stabilisation.

Perhaps the most obvious process relates to precipitation of the available phosphorus, magnesium, calcium and sulphur along with the other micronutrients that exist within faecal material and urine (Table 2.6 and Table 2.9), in particular the use of source separation to enable recovery of the high content of P in urine (0.4 - 2.5 g/cap/day) through struvite precipitation. The pH of faeces and urine are both slightly acidic in nature (Figure 2.5), however, the pH level is likely to increase over short time periods which helps drive the precipitation reactions. Indeed, this self induced onset of precipitation can be detrimental to treatment technology through the precipitation of unwanted scale forming crystals and is considered a particular problem in the supernatant following solid/liquid separation. Nevertheless, the nutrient potential of faeces should not be underestimated, with 50% of N being water-soluble as well as 40% of total P excretion being voided in the faeces.

2.4.4 Thermal processes

Efficient thermal technologies have been the focus of much development because of their potential for energy saving and cost recovery. However, although there is great potential for energy production there is the negative aspect of the loss of nutrients present within faeces and urine as the majority are made unavailable for agriculture use. The cost efficiency of the process is primarily dependent on the water content of excreta and its calorific value.

The total solids (TS) content of faeces and urine is likely to be the most important factor impacting thermal treatment technology, with TS content of faeces (25%) and urine (1%). The TS content and its variation will determine the financial viability of thermal processes and whether it can be a viable feedstock. However, the TS content of faeces (25% TS), is in a similar range to that of dewatered sludge (typically 22-36% TS) from conventional sewage treatment works using belt-filter press, filter press and centrifuge dewatering (Tchobanoglous et al., 2003). This is important as it highlights that when faeces are voided the material is already at the level of de-watered sludge if it could avoid being diluted. This could therefore mean that thermal treatment technologies could potentially be used without prior dewatering processes and this factor could promote collection practices that involve less dilution of the waste stream highlighting again the need to understand the time

related change in faecal identity that occurs during the initial periods after being voided.

Variation in water content (Figure 2.1) was significant with a range of 63% - 86%. Diet was the predominant cause for variation in water content (predominantly fibre intake) in healthy subjects, however, in unhealthy subjects this range can further increase due to the prevalence of diarrhoea. Chronic and acute diarrhoea within populations could have a significant impact on treatment technology as faeces of those with diarrhoea showed increases in water content and a change in physical structure. Global averages of diarrhoea prevalence are significant in developed countries; therefore, this should be accounted for and amplified for technologies aimed at low income regions where both the chronic and acute diarrhoea prevalence rates are likely to be significantly greater. In contrast to diarrhoea, constipation decreases the water content of faeces and is equally prevalent in the developed world. Scales relating to the physical form of faeces also provides a further estimation of the solids composition by providing approximate estimations of the TS content of faeces across large sectors of populations. Research being carried out by Wooley et al. (2013) into assigning a TS value to the Bristol Stool Form scale will be of further benefit to technology development in this respect. Extremes in solids composition may cancel each other out in an averaging effect; however, thermal systems would have to be capable of dealing with this wide range and potential fluctuations in water content.

The calorific value can be used as a metric of potential energy that can be produced during combustion of excreta. Calorific value of faeces (4115 kcal/kg) shows lower values in comparison to animal manure feed-stocks such as swine (4634 kcal/kg), similar values to cattle manure (4211 kcal/kg) but greater than poultry litter (3611 kcal/kg) (Cantrell et al., 2012). Human faeces therefore could present an economically viable option for energy creation through combustion. However, humans will consume a much more varied diet then animals, leading to greater deviation from median values than would be seen in manure feedstock. For example, although there is variation in the calorific value of swine manure from different sites (e.g. 4660 – 7887 kcal/kg (Cao et al., 2010; Xiu et al., 2010) variation within these sites is limited as the animals are kept under the same conditions and

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are being fed the same diet. In contrast, variation in the energy value of faeces is quite substantial (1523 kcal/kg - 10875 kcal/kg). This variation is predominantly caused by the varying presence of unavailable carbohydrates in the diet, the larger the quantity of unavailable carbohydrates the higher the energy value of faeces voided. This has significance, as in lower income countries foodstuffs may often have more unavailable carbohydrates, therefore, faeces of subjects in lower income countries may have faecal energy values higher than the values presented in this study suggest. As a guideline for calorific values faecal dry mass can be used as an estimate for energy losses in faeces (reflecting unavailable carbohydrate intake) and energy adsorption by the body is correlated significantly with faecal dry weights (-0.911) (Calloway and Kretsch, 1978).

The high TS concentration of faeces gives a good case for the source separation of faeces and urine as the addition of urine could add the further problem of dewatering and could resultantly increase costs of thermal treatment processes. Nevertheless a sizeable proportion of urine solids are produced by humans (59 g/cap/day) and the calorific value of urine (1701 kcal/kg) could contribute to energy production if efficient dewatering technologies were available.

Other factors that may be significant for thermal process regard the potential emissions from any thermal treatment process. Levels of sulphur are low in faeces but slightly higher levels are observed in the urine fraction, this could be significant as sulphur in oxygen starved conditions is reacted in the form H_2S (Kang et al., 2011).

2.4.5 Nutrient recovery processes

The nutrients in faeces and urine originate from the food ingested and if recovered and recycled can help contribute to the nutrient requirements of food production in agriculture and subsequently reduce the need for chemical fertiliser manufacturing. Following adequate stabilisation and pathogen destruction the recycling of nutrients to agricultural soils can take place through the direct application of faeces and urine to land or through nutrient removal, concentration and recovery by secondary processes.

The application of human faeces and urine to land following biological processes presents the benefit of providing key macro (N, P, K, S, Ca and Mg) and micro (such as B, Zn, Cu, Fe) nutrients (Table 2.6 and Table 2.8) required for agricultural production as well as the addition of organic matter (44-55% C in faeces) to soils which has multiple benefits to soil composition (House, 1980). The presence of potentially toxic elements in sewage sludge has traditionally been a problematic issue in sewage application to land (Tchobanoglous et al., 2003). However, heavy metal concentrations in faeces and urine (such as Ni (1.15-1.52 g/kg), Pb (0.12-0.27 g/kg) and Cr (0.31-0.91 g/kg)) are all present in very low concentrations and will not be an issue due to the self-regulating aspect of human consumption rarely exceeding potentially toxic concentrations in soils.

The greatest proportion of N losses are through the urine fraction (80%), of which the vast proportion is present in the form of urea (9300-23300 mg/L) which is quickly transformed to NH_4^+. The consequently high NH_4-N concentration presents a valuable resource for nutrient recovery. Ion exchange is a potentially viable mechanism for NH_4^+ capture (Beller-Baykal, 2004), however, competing cationic ions (K^+, Na^+, Ca^{2+}, Mg^{2+}) present in significant concentrations (Table 2.9) will hinder ion exchange processes and may limit its effectiveness in a urine waste stream.

Table 2.12 Summary table of faeces and urine characteristics providing on site sanitation design criteria

Key design criteria	Median value
Faeces	
Faecal wet weight (g/cap/day)	128
Faecal dry weight (g/cap/day)	29
Stool Frequency (motions/24 hours)	1.1
Total Solids (%)	25
VS (% of TS)	89
COD (g/cap/day)	71
Nitrogen (g/cap/day)	1.8
Protein (g/cap/day)	6.3
Lipids (g/cap/day)	4.1
Carbohydrate (g/cap/day)	9
Fibre (g/cap/day)	6
Calorific value (kcal/cap/day)	132
pH	6.6
Urine	
Urine wet weight (L/cap/day)	1.4
Urine dry weight (g/cap/day)	59
Urination frequency (urinations/24 hours)	6
Nitrogen (g/cap/day)	11
Calorific value (kcal/cap/day)	1701
pH	6.2

2.5 CONCLUSIONS

This review aimed to characterise faeces and urine and determine the extent and causes of variation seen and its subsequent impact on technologies treating faeces and urine as a fresh waste stream. Table 2.12 provides a summary of the key criteria and values that will assist in not only the operation of existing OSS systems but will help advance research and development into new OSS technologies.

The generation rate of faeces and urine shows significant variation across a wide range of studies presenting difficulties assigning standard design values for treatment technology processes. The values presented are based upon a large database of values from studies worldwide. The median generation rate of faeces has been calculated at 128 g/cap/day wet mass and 29 g/cap/day dry mass; however, caution should remain when using these central tendency figures as the data sets were highly skewed. The largest factor leading to variability in faecal mass

is the indigestible fibre content of dietary intake; this explains the reason why faecal wet mass values were increased by a factor of 2 in low income countries. A urine generation rate of 1.42 L/cap/day was recorded with the water balance of the body highlighted as the main cause of variation in volume.

Variation in the chemical and physical composition of faeces and urine was widespread throughout the study; this means that technology developments must be robust and flexible in order to deal with this uncertainty. It can be concluded however that the composition of faeces and urine is highly dependent on the dietary intake of subjects. The predominant factor leading to variation in key parameters in faeces was the dietary intake of non-degradable fibre which was shown to impact production rate, stool frequency, TS, fat, protein and the energy value of faeces. In the urine fraction, protein intake was one of the key factors leading to variation in urea concentration as well as impacting concentrations of P, K and Ca in urine.

Biological treatment processes are likely to be effective at treating faeces as a waste stream and a large proportion of the faeces are likely to digest readily. However, high non-degradable fibre content of faeces may reduce digestibility and with a combined waste stream of faeces and urine the anaerobic digestion process may be limited with potential problems such as ammonia toxicity. Technologies based on separation will predominantly be impacted by the variation in TS concentration as well as fluctuating levels of protein and fat found within the faeces. Chemical processes and nutrient recovery will be largely influenced by variation in the diet consumed by subjects, leading to fluctuations in nitrogen and phosphorus loads which could be influential on pH levels, precipitation, ion exchange and nutrient recovery. Thermal treatment processes will similarly be most influenced by variation in TS as well as the energy content of these solids, once again the intake of fibre proved most influential in predicting these factors.

The source separation of faeces and urine could prove beneficial for biological treatment such as anaerobic digestion where large urea concentrations in the urine stream could prove problematic and cause ammonia toxicity. However, high concentrations of ammonium in urine could prove a significant opportunity for nutrient recovery through ion exchange. The separation of the two streams could

also increase the efficiency of the dewatering process and make thermal processes increasingly attractive. In addition to this the largest proportion of nutrients (e.g. N, P and K) are found within the urine fraction making nutrient recovery from urine more attractive from this more easily accessible stream. It is therefore evident that source separation could be beneficial to many treatment technologies.

This study has illustrated that there is significant variation in both the production values as well as the physico-chemical composition of faeces and urine. Therefore, there are limitations in using standard design values in the development of treatment technology. Consequently it is important that treatment technology is robust and flexible enough to deal with the variation exposed. It is however possible to make more appropriate decisions about values of production and composition through the assessment of a target population's diet. Through this a range of dietary factors can be assessed in order to make more informed decisions about design values that specifically target individual populations. Additional data, especially information regarding how the structure of faeces changes over time, would be of further benefit to technology development but there is nevertheless no shortage of data regarding the production and composition of faeces and urine.

2.6 REFERENCES

Abbasi, T. and Abbasi, S. A. (2012), "Formation and impact of granules in fostering clean energy production and wastewater treatment in upflow anaerobic sludge blanket (UASB) reactors", *Renewable and Sustainable Energy Reviews,* vol. 16, no. 3, pp. 1696-1708.

Achour, L., Nancey, S., Moussata, D., Graber, I., Messing, B. and Flourie, B. (2006), "Faecal bacterial mass and energetic losses in healthy humans and patients with a short bowel syndrome", *European Journal of Clinical Nutrition,* vol. 61, no. 2, pp. 233-238.

AdeOluwa, O. O. and Cofie, O. (2012), "Urine as an alternative fertilizer in agriculture: Effects in amaranths (Amaranthus caudatus) production", *Renewable Agriculture and Food Systems,* vol. 27, no. 4, pp. 287-294.

Adibi, P., Behzad, E., Pirzadeh, S. and Mohseni, M. (2007), "Bowel habit reference values and abnormalities in young Iranian healthy adults", *Digestive Diseases and Sciences,* vol. 52, no. 8, pp. 1810-1813.

Aguilera, Y., Cartmell, E., McAdam, E. J. and Lester, J. N. (2012), "Understanding the criticality of granule integrity to resilience of anaerobic wastewater treatment", *IWA World Congress on Water Climate & Energy,* Vol. 1, 13-18 May 2012, Dublin, Ireland, IWA, London, pp. 1.

Ahs, J. W., Tao, W., Löfgren, J. and Forsberg, B. C. (2010), "Diarrheal Diseases in Low- and Middle-Income Countries: Incidence, Prevention and Management", *The Open Infectious Diseases Journal,* vol. 4, pp. 113-124.

Aichbichler, B., Wenzl, H., Ana, C. A. S., Porter, J., Schiller, L. and Fordtran, J. (1998), "A comparison of stool characteristics from normal and constipated people", *Digestive Diseases and Sciences,* vol. 43, no. 11, pp. 2353-2362.

Almeida, M. C., Butler, D. and Friedler, E. (1999), "At-source domestic wastewater quality", *Urban Water,* vol. 1, no. 1, pp. 49-55.

Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J., Guwy, A., Kalyuzhnyi, S., Jenicek, P. and Lier, J. v. (2009), "Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays", .

APHA (2005), *Standard Methods for the Examination of Water and Waste Water,* American Public Health Association, Baltimore, U.S.A.

Ashraf, W., Park, F., Lof, J. and Quigley, E. (1996), "An examination of the reliability of reported stool frequency in the diagnosis of idiopathic constipation", *The American Journal of Gastroenterology,* vol. 91, no. 1, pp. 26-32.

Auth, M. K. H., Vora, R. and Kokai, G. K. (2012), "Investigations of chronic diarrhoea", *Paediatrics and Child Health,* vol. 22, no. 10, pp. 409-418.

Aziz, H. A., Adlan, M. N., Zahari, M. S. and Alias, S. (2004), "Removal of ammoniacal nitrogen (N-NH3) from municipal solid waste leachate by using activated carbon and limestone", *Waste management & research : the journal of the International Solid Wastes and Public Cleansing Association, ISWA,* vol. 22, no. 5, pp. 371-375.

Bael, A. M., Lax, H., Hirche, H., Gäbel, E., Winkler, P., Hellström, A. -., Van Zon, R., Janhsen, E., Güntek, S., Renson, C. and Van Gool, J. D. (2007), "Self-reported urinary incontinence, voiding frequency, voided volume and pad-test results: Variables in a prospective study in children", *BJU International,* vol. 100, no. 3, pp. 651-656.

Bai, F. and Wang, X. (2010), "Nitrogen-retaining property of compost in an aerobic thermophilic composting reactor for the sanitary disposal of human feces", *Frontiers of Environmental Science & Engineering in China,* vol. 4, no. 2, pp. 228-234.

Bai, F. and Wang, X. (2011), "Study on dynamics model of Biodegration of organic matter in aerobic mesophilic composting reactor for sanitary disposal of human feces", *Environmental, Biomedical and Biotechnology,* vol. 3, pp. 11-16.

Ban, Z. S. and Dave, G. (2004), "Laboratory studies on recovery of n and p from human urine through struvite crystallisation and zeolite adsorption", *Environmental Technology,* vol. 25, no. 1, pp. 111-121.

Bassan, M., Tchonda, T., Yiougo, L., Zoellig, H., Mahamane, I., Mbeguere, M. and Strande, L. (2013), "Characterisation of faecal sludge during dry and rainy seasons in Ouagadougou, Burkino Faso", *36th WEDEC International Conference, Nakuru, Kenya, 2013. Delivering water, sanitation and hygiene services in an uncertain environment,* 1st-5th July 2013, Nakuru, Kenya, WEDEC, Loughborough University, UK, pp. 1814.

Beler-Baykal, B., Allar, A. and Bayram, S. (2011), "Nitrogen recovery from source-separated human urine using clinoptilolite and preliminary results of its use as fertilizer", *Water Science and Technology,* vol. 63, no. 4, pp. 811-817.

Beler-Baykal, B., Bayram, S., Akkaymak, E. and Cinar, S. (2004), "Removal of ammonium from human urine through ion exchange with clinoptilolite and its recovery for further reuse", *Water Science and Technology,* vol. 50, no. 6, pp. 149-156.

Bender, D. A. and Bender, A. E. (1997), *Nutrition; a reference handbook,* 1st ed, Oxford University Press Inc., New York, USA.

Beyer, P. L. and Flynn, M. A. (1978), "Effects of high- and low-fiber diets on human feces", *Journal of the American Dietetic Association,* vol. 72, no. 3, pp. 271-277.

Bijkerk, C., Muris, J., Knottnerus, J., Hoes, A. and De Wit, N. (2004), "Systematic review: the role of different types of fibre in the treatment of irritable bowel syndrome", *Alimentary Pharmacology & Therapeutics,* vol. 19, no. 3, pp. 245-251.

Bishop, G., Burns, R., Shepherd, T., Moody, L., Gooch, C., Spajić, R. and Pronto, J. (2009), "Evaluation of laboratory biochemical methane potentials as a predictor of anaerobic dairy manure digester biogas and methane production", *ASABE Paper, ,* no. 096769.

Bond, T. and Templeton, M. R. (2011), "History and future of domestic biogas plants in the developing world", *Energy for Sustainable Development,* vol. 15, no. 4, pp. 347-354.

Borawski, K. M., Sur, R. L., Miller, O. F., Pak, C. Y. C., Preminger, G. M. and Kolon, T. F. (2008), "Urinary Reference Values for Stone Risk Factors in Children", *Journal of Urology,* vol. 179, no. 1, pp. 290-294.

Bryce, D., Croshaw, B., Hall, J., Holland, V. and Lessel, B. (1978), "The activity and safety of the antimicrobial agent bronopol (2-bromo-2-nitropropan-1, 3-diol)", *Journal of the Society of Cosmetic Chemists,* vol. 29, no. 1, pp. 3-24.

Buckley, C., Foxon, K. M., Broukaert, C. J., Rodda, N., Nwaneri, C., Balboni, E., Couderc, A. and Magagna, D. (2008), *Scientific support for the design and operation of Ventilated Improved Pit latrines (VIPs) and the efficacy of pit latrine additives,* WRC project no K5/1630, Water Research Commission, Durban, South Africa.

Burkitt, D., Morley, D. and Walker, A. (1980), "Dietary fibre in under-and overnutrition in childhood", *Archives of Disease in Childhood,* vol. 55, no. 10, pp. 803-807.

Burkitt, D. P., Walker, A. R. P. and Painter, N. S. (1972), "Effect of dietary fibre on stools and transit-times, and its role in the causation of disease", *The Lancet,* vol. 300, no. 7792, pp. 1408-1411.

Burns, M. J., Nixon, G. J., Foy, C. A. and Harris, N. (2005), "Standardisation of data from real-time quantitative PCR methods–evaluation of outliers and comparison of calibration curves", *BMC Biotechnology,* vol. 5, no. 1, pp. 31-44.

Cairncross, S. and Feachem, R. (1993), *Environmental health engineering in the tropics: an introductory text.* John Wiley & Sons Ltd., Chichester, England.

Callis, L., Vila, A., Carrera, M. and Nieto, J. (1999), "Long-term effects of cyclosporine A in Alport's syndrome", *Kidney International,* vol. 55, no. 3, pp. 1051-1056.

Calloway, D. H. and Margen, S. (1971), "Variation in endogenous nitrogen excretion and dietary nitrogen utilization as determinants of human protein requirement", *The Journal of Nutrition,* vol. 101, no. 2, pp. 205-216.

Calloway, D. and Kretsch, M. (1978), "Protein and energy utilization in men given a rural Guatemalan diet and egg formulas with and without added oat bran", *The American Journal of Clinical Nutrition,* vol. 31, no. 7, pp. 1118-1126.

Canfield, J., Goldner, B. and Lutwack, R. (1963), *Research on applied bioelectrochemistry; First quarterly progress report,* NASA-CR-71834, NASA, Anaheim, U.S.A.

Cantrell, K. B., Hunt, P. G., Uchimiya, M., Novak, J. M. and Ro, K. S. (2012), "Impact of pyrolysis temperature and manure source on physicochemical characteristics of biochar", *Bioresource Technology,* vol. 107, no. 0, pp. 419-428.

Cao, X., Ro, K. S., Chappell, M., Li, Y. and Mao, J. (2010), "Chemical Structures of Swine-Manure Chars Produced under Different Carbonization Conditions Investigated by Advanced Solid-State 13C Nuclear Magnetic Resonance (NMR) Spectroscopy†", *Energy & Fuels,* vol. 25, no. 1, pp. 388-397.

Carrieri, M., Trevisan, A. and Bartolucci, G. B. (2000), "Adjustment to concentration-dilution of spot urine samples: correlation between specific gravity and creatinine", *International archives of occupational and environmental health,* vol. 74, no. 1, pp. 63-67.

Chaggu, E. J. (2004), *Sustainable environmental protection using modified pit-latrines* (unpublished PhD thesis), Wageningen Universiteit, Wageningen, Holland.

Chaggu, E. J., Sanders, W. and Lettinga, G. (2007), "Demonstration of anaerobic stabilization of black water in accumulation systems under tropical conditions", *Bioresource Technology,* vol. 98, no. 16, pp. 3090-3097.

Chan, R. and Chen, V. (2004), "Characterization of protein fouling on membranes: opportunities and challenges", *Journal of Membrane Science,* vol. 242, no. 1, pp. 169-188.

Chen, H. L., Cheng, H. C., Wu, W. T., Liu, Y. J. and Liu, S. Y. (2008), "Supplementation of konjac glucomannan into a low-fiber Chinese diet promoted bowel movement and improved colonic ecology in constipated adults: a placebo-controlled, diet-controlled trial", *Journal of the American College of Nutrition,* vol. 27, no. 1, pp. 102-108.

Chen, H. L., Haack, V. S., Janecky, C. W., Vollendorf, N. W. and Marlett, J. A. (1998), "Mechanisms by which wheat bran and oat bran increase stool weight in humans", *The American Journal of Clinical Nutrition,* vol. 68, no. 3, pp. 711-719.

Chen, L., Ho, K. and Phua, K. (2000), "Normal bowel habits and prevalence of functional bowel disorders in Singaporean adults-findings from a community based study in Bishan", *Singapore Medical Journal,* vol. 41, no. 6, pp. 255-258.

Chen, L., Zhao, L., Ren, C. and Wang, F. (2012), "The progress and prospects of rural biogas production in China", *Energy Policy,* vol. 51, no. 0, pp. 58-63.

Choi, E., Eum, Y., Gil, K. I. and Oa, S. W. (2004), "High strength nitrogen removal from nightsoil and piggery wastes", *Water Science and Technology,* vol. 49, no. 5-6, pp. 97-104.

Chong, S., Sen, T. K., Kayaalp, A. and Ang, H. M. (2012), "The performance enhancements of upflow anaerobic sludge blanket (UASB) reactors for domestic sludge treatment - A State-of-the-art review", *Water Research,* vol. 46, no. 11, pp. 3434-3470.

Christensen, J. (1985), "The response of the colon to eating", *The American Journal of Clinical Nutrition,* vol. 42, no. 5, pp. 1025-1032.

Chynoweth, D. P., Turick, C. E., Owens, J. M., Jerger, D. E. and Peck, M. W. (1993), "Biochemical methane potential of biomass and waste feedstocks", *Biomass and Bioenergy,* vol. 5, no. 1, pp. 95-111.

Clare, B. A., Conroy, R. S. and Spelman, K. (2009), "The diuretic effect in human subjects of an extract of Taraxacum officinale folium over a single day", *The Journal of Alternative and Complementary Medicine,* vol. 15, no. 8, pp. 929-934.

Clark, W. F., Sontrop, J. M., Macnab, J. J., Suri, R. S., Moist, L., Salvadori, M. and Garg, A. X. (2011), "Urine volume and change in estimated GFR in a community-based cohort study", *Clinical Journal of the American Society of Nephrology,* vol. 6, no. 11, pp. 2634-2641.

Clean Team (2012)*, Clean Team Toilets*, available at: http://www.cleanteamtoilets.com/ (accessed August 2014).

Coetzee, M., Van der Merwe, M. and Badenhorst, J. (2011), "Effect of nitrogen loading rates on nitrogen removal by using a biological filter proposed for ventilated improved pit latrines", *International Journal of Environment Science and Technology,* vol. 8, no. 2, pp. 363-372.

Cofie, O., Kone, D., Rothenberger, S., Moser, D. and Zubruegg, C. (2009), "Co-composting of faecal sludge and organic solid waste for agriculture: Process dynamics", *Water Research,* vol. 43, no. 18, pp. 4665-4675.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd Water Research Conference,* Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Cooney, E. L., Booker, N. A., Shallcross, D. C. and Stevens, G. W. (1999), "Ammonia removal from wastewaters using natural Australian zeolite. I. Characterization of the zeolite", *Separation Science and Technology,* vol. 34, no. 12, pp. 2307-2327.

Cummings, J. H., Beatty, E. R., Kingman, S. M., Bingham, S. A. and Englyst, H. N. (1996), "Digestion and physiological properties of resistant starch in the human large bowel", *British Journal of Nutrition,* vol. 75, no. 05, pp. 733-747.

Cummings, J., Bingham, S., Heaton, K. and Eastwood, M. (1992), "Fecal weight, colon cancer risk, and dietary intake of nonstarch polysaccharides (dietary fiber)", *Gastroenterology,* vol. 103, no. 6, pp. 1783-1789.

Czemiel, J. (2000), "Phosphorus and nitrogen in sanitary systems in Kalmar", *Urban Water,* vol. 2, no. 1, pp. 63-69.

Danivat, D., Tankeyoon, M. and Sriratanaban, A. (1988), "Prevalence of irritable bowel syndrome in a non-Western population", *British medical journal (Clinical research ed.),* vol. 296, no. 6638, pp. 1710-1710.

Davies, G., Crowder, M., Reid, B. and Dickerson, J. (1986), "Bowel function measurements of individuals with different eating patterns", *Gut,* vol. 27, no. 2, pp. 164-169.

Del Porto, D. and Steinfeld, C. (1999), *The composting toilet system book: a practical guide to choosing, planning and maintaining composting toilet systems, a water-saving, pollution-preventing alternative,* 1st ed, The Center for Ecological Pollution Protection, Vermont, Canada.

Diem, K. and Lentner, C. (1970), *Scientific Tables: Excerpts from the 7th Ed,* Ciba-Geigy.

Diener, S., Semiyaga, S., Niwagaba, C. B., Muspratt, A. M., Gning, J. B., Mbéguéré, M., Ennin, J. E., Zurbrugg, C. and Strande, L. (2014), "A value proposition: Resource recovery from faecal sludge—Can it be the driver for improved sanitation?", *Resources, Conservation and Recycling,* vol. 88, no. 0, pp. 32-38.

Doku, I. A. (2003), *The potential for the use of upflow anaerobic sludge blanket (UASB) reactor for the treatment of faecal sludges in Ghana,* Assainissement A06, Ministère des Affaires Etrangères, Training Research and Networking for Development Group, Kumasi, Ghana.

Dong, Y. (1999), "Simultaneous determination of creatine, uric ccid, creatinine and hippuric acid in urine by high performance liquid chromatography", *Biomedical Chromatography,* vol. 12, no. 2, pp. 47-49.

Drangert, J. (1998), "Fighting the urine blindness to provide more sanitation options", *Water SA,* vol. 24, pp. 157-164.

Drechsel, P., Scott, C. A., Raschid-Sally, L., Redwood, M. and Bahri, A. (eds.) (2009), *Wastewater Irrigation and Health: Assessing and Mitigating Risk in Low Income Countries,* 1st ed, Earthscan/IWMI/IDRC, London.

Drosg, B., Braun, R., Bochmann, G. and Al Saedi, T. (2013), "Analysis and characterisation of biogas feedstocks", *The biogas handbook: Science, production and applications,* , pp. 52-84.

DuPont, H. L. (1997), "Guidelines on Acute Infectious Diarrhea in Adults", *The American Journal of Gastroenterology,* vol. 92, no. 11, pp. 1962-1974.

Eastwood, M. A., Brydon, W. G., Baird, J., Elton, R., Helliwell, S., Smith, J. and Pritchard, J. (1984), "Fecal weight and composition, serum lipids, and diet among subjects aged 18 to 80 years not seeking health care", *The American Journal of Clinical Nutrition,* vol. 40, no. 3, pp. 628-634.

Eastwood, M. (1973), "Vegetable fibre: its physical properties", *Proceedings of the Nutrition Society,* vol. 32, no. 03, pp. 137-143.

Elbeshbishy, E., Nakhla, G. and Hafez, H. (2012), "Biochemical methane potential (BMP) of food waste and primary sludge: Influence of inoculum pre-incubation and inoculum source", *Bioresource technology,* vol. 110, no. 0, pp. 18-25.

FAO/WHO (1997), *Carbohydrates in human nutrition,* FAO Food and Nutrition Paper 66, Food and Agriculture Organization of the United Nations, Rome.

FAO/WHO/UNU (1985), *Energy and Protein Requirments,* Report of a joint expert consultation, WHO Technical Report Series, Geneva.

Farthing, M. J. G. and Kelly, P. (2007), "Infectious diarrhoea", *Medicine,* vol. 35, no. 5, pp. 251-256.

Feachem, D. J., Bradley, H. :., H. and Mara, D. D. (1978), *Health Aspects of Excreta and Wastewater Management,* 1st ed, The World Bank, Washington, DC. U.S.A.

Feldman, R. A. and Banatvala, N. (1994), "The frequency of culturing stools from adults with diarrhoea in Great Britain", *Epidemiology and infection,* vol. 113, no. 1, pp. 41-44.

Fischer Walker, C. L., Perin, J., Aryee, M. J., Boschi-Pinto, C. and Black, R. E. (2012), "Diarrhea incidence in low- and middle-income countries in 1990 and 2010: A systematic review", *BMC Public Health,* vol. 12, no. 1.

Fourie, A. and Van Ryneveld, M. (1995), "The fate in the subsurface of contaminants associated with on-site sanitation: A review", *Water S. A.,* vol. 21, no. 2, pp. 101-111.

Friedler, E., Brown, D. M. and Butler, D. (1996), "A study of WC derived sewer solids", *Water Science and Technology,* vol. 33, no. 9, pp. 17-24.

Fry, L. J. and Merrill, R. (1973), *Methane digesters for fuel gas and fertilizer,* 1st ed, New Alchemy Institute, Massachusetts, USA.

Fytili, D. and Zabaniotou, A. (2008), "Utilization of sewage sludge in EU application of old and new methods-A review", *Renewable and Sustainable Energy Reviews,* vol. 12, no. 1, pp. 116-140.

Gades, M. D. and Stern, J. S. (2012), "Chitosan supplementation and fecal fat excretion in men", *Obesity Research,* vol. 11, no. 5, pp. 683-688.

Garrow, J. S., James, W. P. T. and Ralph, A. (eds.) (1993), *Human Nutrition and Dietetics,* 9th ed, Longman Group, Edinburgh, UK.

Gautam, R., Baral, S. and Herat, S. (2009), "Biogas as a sustainable energy source in Nepal: Present status and future challenges", *Renewable and Sustainable Energy Reviews,* vol. 13, no. 1, pp. 248-252.

Gear, J., Brodribb, A., Ware, A. and Mannt, J. (1981), "Fibre and bowel transit times", *British Journal of Nutrition,* vol. 45, no. 01, pp. 77-82.

Global Development Program (2013)*, Water ,Sanitation adn Hygiene: Strategy Overview*, available at: http://www.gatesfoundation.org/What-We-Do/Global-Development/Water-Sanitation-and-Hygiene (accessed May 28).

Goldberg, M. J., Smith, J. W. and Nichols, R. L. (1977), "Comparison of the fecal microflora of Seventh-Day Adventists with individuals consuming a general diet. Implications concerning colonic carcinoma.", *Annals of Surgery,* vol. 186, no. 1, pp. 97.

Goldblith, S. A. and Wick, E. L. (1961), *Analysis of human fecal components and study of methods for their recovery in space systems,* ASO-TR-61-419, NASA, Washington DC.

Goldsmith, H. S. and Burkitt, D. P. (1975), "Letter: Stool characteristics of Black and White Americans", *Lancet,* vol. 2, no. 7931, pp. 407.

Gonzalez Gil, G., Seghezzo, L., Lettinga, G. and Kleerebezem, R. (2001), "Kinetics and mass-transfer phenomena in anaerobic granular sludge", *Biotechnology and Bioengineering,* vol. 73, no. 2, pp. 125-134.

Goy, J., Eastwood, M., Mitchell, W., Pritchard, J. and Smith, A. (1976), "Fecal characteristics contrasted in the irritable bowel syndrome and diverticular disease", *The American Journal of Clinical Nutrition,* vol. 29, no. 12, pp. 1480-1484.

Grady, J., Leslie, C. P., Daigger, G. T. and Lim, H. C. (1999), *Biological Wastewater Treatment,* 2nd ed, Marcel Dekker, Inc., New York, USA.

Graham, J. P. and Polizzotto, M. L. (2013), "Pit Latrines and Their Impacts on Groundwater Quality: A Systematic Review", *Environmental Health Perspectives,* vol. 121, no. 5, pp. 521.

Guendouz, J., Buffiere, P., Cacho, J., Carrere, M. and Delgenes, J. (2008), "High-solids anaerobic digestion: comparison of three pilot scales", *Water Science and Technology,* vol. 58, no. 9, pp. 1757-1763.

Guštin, S. and Marinšek-Logar, R. (2011), "Effect of pH, temperature and air flow rate on the continuous ammonia stripping of the anaerobic digestion effluent", *Process Safety and Environmental Protection,* vol. 89, no. 1, pp. 61-66.

Guyton, A. C. and Hall, J. (eds.) (2000), *Textbook of medical physiology,* 12th ed, WB Saunders Co, Philadelphia, USA.

Gwatkin, D. R., Rutstein, S., Johnson, K., Suliman, E. and Wagstaff, A. (2007), *Socio-Economic Differences in Health, Nutrition, and Population,* , The World Bank, Washington, D.C.

Han, S. H., Lee, O. Y., Bae, S. C., Lee, S. H., Chang, Y. K., Yang, S. Y., Yoon, B. C., Choi, H. S., Hahm, J. S. and Lee, M. H. (2006), "Prevalence of irritable bowel syndrome in Korea: Population-based survey using the Rome II criteria", *Journal of gastroenterology and hepatology,* vol. 21, no. 11, pp. 1687-1692.

Hankins, N., Pliankarom, S. and Hilal, N. (2005), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

Hansen, J. A. and Tjell, J. C. (1979), *Human Excretion of heavy metals and other elements,* pp1-15, Technical University of Denmark, Denmark.

Harper, H. A., Rodwell, V. W. and Mayes, P. A. (1977), "Review of Physiological Chemistry", in 16th ed, Lang Medical Publications, California, pp. 625.

He, Z., Zhang, H., Toor, G. S., Dou, Z., Honeycutt, C. W., Haggard, B. E. and Reiter, M. S. (2010), "Phosphorus distribution in sequentially extracted fractions of biosolids, poultry litter, and granulated products", *Soil Science,* vol. 175, no. 4, pp. 154-161.

Heaton, K., Radvan, J., Cripps, H., Mountford, R., Braddon, F. and Hughes, A. (1992), "Defecation frequency and timing, and stool form in the general population: a prospective study.", *Gut,* vol. 33, no. 6, pp. 818-824.

Heinonen-Tanski, H., Sjöblom, A., Fabritius, H. and Karinen, P. (2007), "Pure human urine is a good fertiliser for cucumbers", *Bioresource Technology,* vol. 98, no. 1, pp. 214-217.

Heinonen-Tanski, H. and van Wijk-Sijbesma, C. (2005), "Human excreta for plant production", *Bioresource Technology,* vol. 96, no. 4, pp. 403-411.

Heinss, U., Larmie, S. A. and Strauss, M. (1998), *Solids separation and pond systems for the treatment of faecal sludges in the tropics: lessons learnt and recommendations for preliminary design,* 05/98, EAWAG/SANDEC, Dubendorf, Switzerland.

Heinss, U., Larmie, S. A. and Strauss, M. (1999), *Characteristics of faecal sludges and their solids-liquid separation,* 1, EAWAG/SANDEC, Zurich, Switzerland.

Henze, M., Harremoës, P., la Cour Jansen, J. and Arvin, E. (2001), *Wastewater treatment: biological and chemical processes,* 3rd ed, Springer, Berlin, Germany.

Herikstad, H., Yang, S., Van Gilder, T., Vugia, D., Hadler, J., Blake, P., Deneen, V., Shiferaw, B. and Angulo, F. (2002), "A population-based estimate of the burden of diarrhoeal illness in the United States: FoodNet, 1996-7", *Epidemiology and Infection,* vol. 129, no. 1, pp. 9-17.

Hesse, A., Classen, A., Knoll, M., Timmermann, F. and Vahlensieck, W. (1986), "Dependence of urine composition on the age and sex of healthy subjects", *Clinica Chimica Acta,* vol. 160, no. 2, pp. 79-86.

Higgins, P. D. and Johanson, J. F. (2004), "Epidemiology of constipation in North America: a systematic review", *The American Journal of Gastroenterology,* vol. 99, no. 4, pp. 750-759.

Hineno, T., Mizobuchi, M., Nishimatsu, O., Horiguchi, J. and Kakimoto, Y. (1994), "Day-Night Variation of Urine Volume in Parkinson's Disease", *Psychiatry and clinical neurosciences,* vol. 48, no. 3, pp. 583-587.

Hoffmann, J. P. (1998), "Wastewater treatment with suspended and nonsuspended algae", *Journal of Phycology,* vol. 34, no. 5, pp. 757-763.

Höglund, C., Vinneras, B., Stenström, T. A. and Jönsson, H. (2000), "Variation of chemical and microbial parameters in collection and storage tanks for source separated human urine", *Journal of Environmental Science and Health,* vol. 35, no. 8, pp. 1463-1475.

Horan, N. J., Gohar, H. and Hill, B. (1997), "Application of a granular activated carbon-biological fluidised bed for the treatment of landfill leachates containing high concentrations of ammonia", *Water Science and Technology,* vol. 36, no. 2–3, pp. 369-375.

Hounkpe, S. P., Adjovi, E. C., Crapper, M. and Awuah, E. (2014), "Wastewater Management in Third World Cities: Case Study of Cotonou, Benin", *Journal of Environmental Protection,* vol. 5, no. 05, pp. 387.

House, D. (1981), *Biogas Handbook,* 2nd ed, Peace Press Inc, California, U.S.A.

Hughes, R., Pollock, J. R. A. and Bingham, S. (2002), "Effect of vegetables, tea, and soy on endogenous N-nitrosation, fecal ammonia, and fecal water genotoxicity during a high red meat diet in humans", *Nutrition and Cancer,* vol. 42, no. 1, pp. 70-77.

Ingallinella, A., Sanguinetti, G., Koottatep, T., Montangero, A., Strauss, M., Jimenez, B., Spinosa, L., Odegaard, H. and Lee, D. (2002), "The challenge of faecal sludge management in urban areas- strategies, regulations and treatment options", *Water Science and Technology,* vol. 46, no. 10, pp. 285-294.

Issah, A., Aklaku, E. D. and Salifu, T. (2012), "Comparative study of effluent for pollutionindicators and indicator pathogenic organisms from anerobic digesters from human and fruit wastes", *ARPN Journal of Agricultural and Biological Science,* vol. 6, no. 6, pp. 416-420.

Iyengar, V., Albaugh, G., Lohani, A. and Nair, P. (1991), "Human stools as a source of viable colonic epithelial cells.", *The FASEB Journal,* vol. 5, no. 13, pp. 2856-2859.

Jackson, S. (1966), "Creatinine in urine as an index of urinary excretion rate", *Health physics,* vol. 12, no. 6, pp. 843-850.

Jacobsen, R., Lorenzen, J., Toubro, S., Krog-Mikkelsen, I. and Astrup, A. (2005), "Effect of short-term high dietary calcium intake on 24-h energy expenditure, fat oxidation, and fecal fat excretion", *International journal of obesity,* vol. 29, no. 3, pp. 292-301.

Jana, B. B., Rana, S. and Bag, S. K. (2012), "Use of human urine in phytoplankton production as a tool for ecological sanitation", *Water Science and Technology,* vol. 65, no. 8, pp. 1350-1356.

Jeffery, S., Bezemer, T. M., Cornelissen, G., Kuyper, T. W., Lehmann, J., Mommer, L., Sohi, S. P., Voorde, T. F., Wardle, D. A. and Groenigen, J. W. (2015), "The way forward in biochar research: targeting trade-offs between the potential wins", *GCB Bioenergy,* vol. 7, no. 1, pp. 1-13.

Jen, J., Hsiao, S. L. and Liu, K. H. (2002), "Simultaneous determination of uric acid and creatinine in urine by an eco-friendly solvent-free high performance liquid chromatographic method", *Talanta,* vol. 58, no. 4, pp. 711-717.

Jones, D. B. (1931), *Factors for converting percentages of nitrogen in foods and feeds into percentages of proteins,* US Department of Agriculture Washington, DC.

Jönsson, H. (2005), *Composition of urine, faeces, greywater and biowaste for utilisation in the URWARE model,* Urban Water, Chalmers University of Technology.

Jönsson, H., Baky, A., Jeppsson, U., Hellström, D. and Kärrman, E. (2005), *Composition of urine, faeces, greywater and bio-waste-for utilisation in the URWARE model.* Urban Water Report 2005: 6, Chalmers University of Technology, Gothenburg, Sweden.

Jönsson, H., Stenström, T. A., Svensson, J. and Sundin, A. (1997), "Source separated urine-nutrient and heavy metal content, water saving and faecal contamination", *Water Science and Technology,* vol. 35, no. 9, pp. 145-152.

Jönsson, H., Stintzing, A. R., Vinnerås, B. and Salomon, E. (2004)*, Guidelines on the use of urine and faeces in crop production*, available at: http://www.ecosanres.org/pdf_files/ESR-factsheet-06.pdf (accessed 06.11.2012).

Kanbara, A., Miura, Y., Hyogo, H., Chayama, K. and Seyama, I. (2012), "Effect of urine pH changed by dietary intervention on uric acid clearance mechanism of pH-dependent excretion of urinary uric acid", *Nutrition Journal,* vol. 11, no. 1, pp. 39.

Kang, S., Dong, J., Kim, J., Lee, W. and Hwang, W. (2011), "Gasification and its emission characteristics for dried sewage sludge utilizing a fluidized bed gasifier", *Journal of Material Cycles and Waste Management,* vol. 13, no. 3, pp. 180-185.

Karak, T. and Bhattacharyya, P. (2011), "Human urine as a source of alternative natural fertilizer in agriculture: A flight of fancy or an achievable reality", *Resources, Conservation and Recycling,* vol. 55, no. 4, pp. 400-408.

Karki, A. and Tiwari, B. R. (2007), "Prevalence of acute diarrhoea in Kathmandu valley", *Journal of the Nepal Medical Association,* vol. 46, no. 168, pp. 175-179.

Katukiza, A. Y., Ronteltap, M., Niwagaba, C. B., Foppen, J. W. A., Kansiime, F. and Lens, P. N. L. (2012), "Sustainable sanitation technology options for urban slums", *Biotechnology Advances,* vol. 30, no. 5, pp. 964-978.

Kelsay, J., Behall, K. and Prather, E. (1978), "Effect of fiber from fruits and vegetables on metabolic responses of human subjects I. Bowel transit time, number of defecations, fecal weight, urinary excretions of energy and nitrogen and apparent digestibilities of energy, nitrogen, and fat.", *The American Journal of Clinical Nutrition,* vol. 31, no. 7, pp. 1149-1153.

Kennedy-Walker, R., Amezaga, J. and Patterson, C. (2015), "Planning for improved faecal sludge management for informal settlements in x", *The 3rd International Faecal Sludge Management Conference,* 18th-21st January, Hanoi, Vietnam, FSM3, EAWAG, pp. 1.

Keraita, B., Drechsel, P. and Amoah, P. (2003), "Influence of urban wastewater on stream water quality and agriculture in and around Kumasi, Ghana", *Environment and Urbanization,* vol. 15, no. 2, pp. 171-178.

Kerstens, S., Legowo, H. and Gupta, I. H. (2012), "Evaluation of DEWATS in Java, Indonesia", *Journal of Water, Sanitation and Hygiene for Development,* vol. 2, no. 4, pp. 254-265.

Kien, C., Cordano, A., Cook, D. and Young, V. (1981), "Fecal characteristics in healthy young adults consuming defined liquid diets or a free-choice diet", *The American Journal of Clinical Nutrition,* vol. 34, no. 3, pp. 357-361.

Kipnis, V., Midthune, D., Freedman, L. S., Bingham, S., Schatzkin, A., Subar, A. and Carroll, R. J. (2001), "Empirical evidence of correlated biases in dietary assessment instruments and its implications", *American Journal of Epidemiology,* vol. 153, no. 4, pp. 394-403.

Kirchmann, H. and Pettersson, S. (1994), "Human urine-chemical composition and fertilizer use efficiency", *Nutrient Cycling in Agroecosystems,* vol. 40, no. 2, pp. 149-154.

Kone, D. (2010), "Making urban excreta and wastewater management contribute to cities' economic development: a paradigm shift", *Water Policy,* vol. 12, no. 4, pp. 602-610.

Koné, D. and Strauss, M. (2004), "Low-cost options for treating faecal sludges (FS) in developing countries–Challenges and performance", *9th International IWA Specialist Group Conference on Wetlands Systems for Water Pollution Control and to the 6th International IWA Specialist Group Conference on Waste Stabilisation Ponds,* Vol. 27, 27 Sept. – 1 Oct., 2004, Avignon, France, IWA, London, UK, .

Kone, D., Cofie, O., Zurbrügg, C., Gallizzi, K., Moser, D., Drescher, S. and Strauss, M. (2007), "Helminth eggs inactivation efficiency by faecal sludge dewatering and co-composting in tropical climates", *Water research,* vol. 41, no. 19, pp. 4397-4402.

Kujawa-Roeleveld, K. and Zeeman, G. (2006), "Anaerobic Treatment in Decentralised and Source-Separation-Based Sanitation Concepts", *Reviews in Environmental Science and Biotechnology,* vol. 5, no. 1, pp. 115-139.

Kuo, P. and Huang, N. (1965), "The effect of medium chain triglyceride upon fat absorption and plasma lipid and depot fat of children with cystic fibrosis of the pancreas.", *Journal of Clinical Investigation,* vol. 44, no. 11, pp. 1924.

Lalander, C., Diener, S., Magri, M. E., Zurbrügg, C., Lindström, A. and Vinnerås, B. (2013), "Faecal sludge management with the larvae of the black soldier fly (Hermetia illucens) — From a hygiene aspect", *Science of The Total Environment,* vol. 458–460, no. 0, pp. 312-318.

Lampe, J., Fredstrom, S., Slavin, J. and Potter, J. (1993), "Sex differences in colonic function: a randomised trial.", *Gut,* vol. 34, no. 4, pp. 531-536.

Ledda, C., Schievano, A., Salati, S. and Adani, F. (2013), "Nitrogen and water recovery from animal slurries by a new integrated ultrafiltration, reverse osmosis and cold stripping process: A case study", *Water research,* vol. 47, no. 16, pp. 6157-6166.

Lentner, C. (1981), *Geigy Scientific Tables:-1: Units of Measurement, Body Fluids, Composition of the Body,* 8th ed, CIBA-Geigy, Basle, Switzerland.

Leung, H. (2001), "Ecotoxicology of glutaraldehyde: review of environmental fate and effects studies", *Ecotoxicology and Environmental Safety,* vol. 49, no. 1, pp. 26-39.

Lewis, S. and Heaton, K. (1997), "Stool form scale as a useful guide to intestinal transit time", *Scandinavian Journal of Gastroenterology,* vol. 32, no. 9, pp. 920-924.

Li, A. F., Ning, Y. Y., Xu, W. J. and Pan, T. (2014), "Air Stripping of Ammoniacal Nitrogen from Fecal Sewage Using a Fluidized Bed", *Advanced Materials Research,* Vol. 926, Trans Tech Publ, pp. 4209.

Liang, C., Das, K. C. and McClendon, R. W. (2003), "The influence of temperature and moisture contents regimes on the aerobic microbial activity of a biosolids composting blend", *Bioresource technology,* vol. 86, no. 2, pp. 131-137.

Lim, J. W. (2011), "Anaerobic Co-digestion of Brown Water and Food Waste for Energy Recovery", *11th edition of the World Wide Workshop for Young Environmental Scientists (WWW-YES-2011)-Urban Waters: resource or risks?* 5th-10th June 2011, Accueil, France, UPEC/NPEC, France, pp. 1.

Lloyd, S. J., Kovats, R. S. and Armstrong, B. G. (2007), "Global diarrhoea morbidity, weather and climate", *Climate Research,* vol. 34, no. 2, pp. 119-127.

Lopez Zavala, M. A. Funamizu, N. Takakuwa, T (2002), "Characterization of Feces for Describing the Aerobic Biodegradation of Feces", *PROCEEDINGS- JAPAN SOCIETY OF CIVIL ENGINEERS,* , no. 720, pp. 99-105.

Lu, Q., He, Z. L. and Stoffella, P. J. (2012), "Land application of biosolids in the USA: A review", *Applied and Environmental Soil Science,* vol. 2012, no. Article ID 201462, pp. 1-11.

X Water and Sewage Company (2013), *Environmental and social impact assessment report: drainage report,* 1, USACE Europe District/Millennium Challenge Corporation, Ministry of Local Government and Housing.

Maalouf, N. M., Sakhaee, K., Parks, J. H., Coe, F. L., Adams-Huet, B. and Pak, C. Y. C. (2004), "Association of urinary pH with body weight in nephrolithiasis", *Kidney international,* vol. 65, no. 4, pp. 1422-1425.

Magee, E. A., Curno, R., Edmond, L. M. and Cummings, J. H. (2004), "Contribution of dietary protein and inorganic sulfur to urinary sulfate: toward a biomarker of inorganic sulfur intake", *The American Journal of Clinical Nutrition,* vol. 80, no. 1, pp. 137-142.

Mai, V., McCrary, Q. M., Sinha, R. and Glei, M. (2009), "Associations between dietary habits and body mass index with gut microbiota composition and fecal water genotoxicity: An observational study in African American and Caucasian American volunteers", *Nutrition Journal,* vol. 8, no. 1.

Mang, H. P. and Li, Z. (2010), *Technology review of biogas sanitation - Biogas sanitation for blackwater, brown water or for excreta and organic household waste treatment and reuse in developing countries,* , Deutsche Gesellschaft für Internationale Zusammenarbeit GmbH (GIZ), Eschborn, Germany.

Mantovi, P., Baldoni, G. and Toderi, G. (2005), "Reuse of liquid, dewatered, and composted sewage sludge on agricultural land: effects of long-term application on soil and crop", *Water research,* vol. 39, no. 2–3, pp. 289-296.

Mariotti, F., Tomé, D. and Mirand, P. P. (2008), "Converting nitrogen into protein—beyond 6.25 and Jones' factors", *Critical reviews in food science and nutrition,* vol. 48, no. 2, pp. 177-184.

Martin, J. H. (2005), *An evaluation of a Mesophilic, modified plug flow anaerobic digester for dairy cattle manure,* EPA Contract No. GS 10F-0036K Work Assignment/Task Order 9, Eastern Research Group, Inc, Lexington, USA.

Meinzinger, F. and Oldenburg, M. (2009), "Characteristics of source-separated household wastewater flows: a statistical assessment", *Water Science and Technology,* vol. 59, no. 9, pp. 1785-1791.

Miles, A. and Ellis, T. G. (2001), "Struvite precipitation potential for nutrient recovery from anaerobically treated wastes", *Water Science & Technology,* vol. 43, no. 11, pp. 259-266.

Mojtahedi, M., De Groot, L. C. P. G. M., Boekholt, H. A. and Van Raaij, J. M. A. (2002), "Nitrogen balance of healthy Dutch women before and during pregnancy", *American Journal of Clinical Nutrition,* vol. 75, no. 6, pp. 1078-1083.

Mondor, M., Masse, L., Ippersiel, D., Lamarche, F. and Massé, D. I. (2008), "Use of electrodialysis and reverse osmosis for the recovery and concentration of ammonia from swine manure", *Bioresource technology,* vol. 99, no. 15, pp. 7363-7368.

Montangero and Strauss, M. (Dept. of Water & Sanitation in Developing Countries), (2004), *Faecal Sludge Treatment* Eawag, Swiss Federal Institute of Aquatic Science & Technology.

Montangero, A. and Belevi, H. (2007), "Assessing nutrient flows in septic tanks by eliciting expert judgement: A promising method in the context of developing countries", *Water research,* vol. 41, no. 5, pp. 1052-1064.

Moyes, L. and McKee, R. (2008), "A review of surgical nutrition", *Scottish Medical Journal,* vol. 53, no. 1, pp. 38-43.

Mulder, A. (2003), "The quest for sustainable nitrogen removal technologies", *Water science & technology,* vol. 48, no. 1, pp. 67-75.

Muñoz, I., i Canals, L. M., Clift, R. and Doka, G. (2007), *A simple model to include human excretion and wastewater treatment in Life Cycle Assessment of food products,* CES Working Paper 01/07, Centre for Environmental Strategy, University of Surrey, Guildford, UK.

Mykkänen, H., Laiho, K. and Salminen, S. (1998), "Variations in faecal bacterial enzyme activities and associations with bowel function and diet in elderly subjects", *Journal of applied microbiology,* vol. 85, no. 1, pp. 37-41.

Myo-Khin, Kyaw-Hla, S. and Bolin, T. (1994), "A prospective study on defecation frequency, stool weight, and consistency.", *Archives of Disease in Childhood,* vol. 71, no. 4, pp. 311-314.

Nelson, N. O., Mikkelsen, R. L. and Hesterberg, D. L. (2003), "Struvite precipitation in anaerobic swine lagoon liquid: effect of pH and Mg: P ratio and determination of rate constant", *Bioresource technology,* vol. 89, no. 3, pp. 229-236.

Newman, D. J., Pugia, M. J., Lott, J. A., Wallace, J. F. and Hiar, A. M. (2000), "Urinary protein and albumin excretion corrected by creatinine and specific gravity.", *Clinica chimica acta; international journal of clinical chemistry,* vol. 294, no. 1-2, pp. 139.

Newmark, H. L. and Lupton, J. R. (1990), "Determinants and consequences of colonic luminal pH: implications for colon cancer", *Nutrition and Cancer,* vol. 14, no. 3-4, pp. 161-173.

Nguyen, T., Roddick, F. A. and Fan, L. (2012), "Biofouling of Water Treatment Membranes: A Review of the Underlying Causes, Monitoring Techniques and Control Measures", *Membranes,* vol. 2, no. 4, pp. 804-840.

Nwaneri, C., Foxon, K., Bakare, B. and Buckley, C. (2008), "Biological degradation processes within a pit latrine", *WISA,* Vol. 1, 18-22 May 2008, Sun CIty, WISA, South Africa, pp. 1.

Otterpohl, R., Braun, U. and Oldenburg, M. (2002), "Innovative technologies for decentralised wastewater management in urban and peri-urban areas", *BERICHTE-WASSERGUTE UND ABFALLWIRTSCHAFT TECHNISCHE UNIVERSITAT MUNCHEN BERICHTSHEFT,* vol. 173, pp. 109-126.

Owens, J. and Chynoweth, D. (1993), "Biochemical methane potential of municipal solid waste (MSW) components", *Water Science and Technology,* vol. 27, no. 2, pp. 1-14.

Palmquist, H. and Jönsson, H. (2004), "Urine, faeces, greywater and biodegradable solid waste as potential fertilisers", *The 2nd International Symposium on Ecological Sanitation, Incorporating the 1st IWA Specialist Group Conference on Sustainable Sanitation,* 7th–11th April, Lübeck, Germany, pp. 825.

Parker, A., Kennedy-Walker, R., Rose, C., Cartmell, E. and Collins, G. (2013), "A novel, high-rate, anaerobic digester to treat high solids waste ensuring re-use and good sanitation planning", *Delivering water, sanitation and hygiene services in an uncertain environment,* July 1st-5th, Nakkuru, Water Engineering and Development Center (WEDC)), Loughborough University, pp. 1.

Parker, D. and Gallagher, S. (1992), "Distribution of Human Waste Samples in Relation to Sizing Waste Processing in Space", *Lunar Bases and Space Activities of the 21st Century,* Vol. 1, pp. 563.

Parker, D. and Gallagher, S. (1988), "Distribution of human waste samples in relation to sizing waste processing in space", *LPI Contributions,* vol. 652, pp. 188.

Parkin, G. F. and Owen, W. F. (1986), "Fundamentals of anaerobic digestion of wastewater sludges", *Journal of Environmental Engineering,* vol. 112, no. 5, pp. 867-920.

Patel, K. and Thillainayagam, A. V. (2009), "Diarrhoea", *Medicine,* vol. 37, no. 1, pp. 23-27.

Pawełczyk, A. and Popowicz, A. (2006), "Application of natural zeolites in the production of controlledrelease fertilizers", *Book of Abstracts,* pp. 203.

Phillips, J., Muir, J. G., Birkett, A., Lu, Z. X., Jones, G. P., O'Dea, K. and Young, G. P. (1995), "Effect of resistant starch on fecal bulk and fermentation-dependent events in humans.", *The American Journal of Clinical Nutrition,* vol. 62, no. 1, pp. 121-130.

Poullis, A., Foster, R., Shetty, A., Fagerhol, M. K. and Mendall, M. A. (2004), "Bowel inflammation as measured by fecal calprotectin", *Cancer Epidemiology Biomarkers and Prevention,* vol. 13, no. 2, pp. 279-284.

Prüss-Üstün, A., Bos, R., Gore, F. and Bartram, J. (2008), *Safer water, better health: costs, benefits and sustainability of interventions to protect and promote health.* 1st ed, World Health Organization, Geneva.

Putnam, D. F. (1971), *Composition and concentrative properties of human urine,* CR-1802, NASA, Washington DC, USA.

Rajagopal, R., Lim, J. W., Mao, Y., Chen, C. and Wang, J. (2013), "Anaerobic co-digestion of source segregated brown water (feces-without-urine) and food waste: For Singapore context", *Science of the Total Environment,* vol. 443, pp. 877-886.

Reddy, S., Sanders, T., Owen, R. and Thompson, M. (1998), "Faecal pH, bile acid and sterol concentrations in premenopausal Indian and white vegetarians compared with white omnivores", *British Journal of Nutrition,* vol. 79, no. 06, pp. 495-500.

Rivero-Marcotegui, A., Olivera-Olmedo, J. E., Valverde-Visus, F. S., Palacios-Sarrasqueta, M., Grijalba-Uche, A. and García-Merlo, S. (1998), "Water, fat, nitrogen, and sugar content in feces: reference intervals in children", *Clinical Chemistry,* vol. 44, no. 7, pp. 1540-1544.

Rose, C., Parker, A., Jefferson, B. and Cartmell, E. (2015), "The Characterisation of faeces and urine; a review of the literature to inform advanced treatment technology", *Critical Reviews in Environmental Science and Technology,* vol. 45, no. 17, pp. 1827-1879.

Rožić, M., Cerjan-Stefanović, Š, Kurajica, S., Vančina, V. and Hodžić, E. (2000), "Ammoniacal nitrogen removal from water by treatment with clays and zeolites", *Water research,* vol. 34, no. 14, pp. 3675-3681.

Rydin, Y., Bleahu, A., Davies, M., Dávila, J. D., Friel, S., De Grandis, G., Groce, N., Hallal, P. C., Hamilton, I. and Howden-Chapman, P. (2012), "Shaping cities for health: complexity and the planning of urban environments in the 21st century", *The Lancet,* vol. 379, no. 9831, pp. 2079-2108.

Sandler, R. S. and Drossman, D. A. (1987), "Bowel habits in young adults not seeking health care", *Digestive Diseases and Sciences,* vol. 32, no. 8, pp. 841-845.

Sarkhot, D., Ghezzehei, T. and Berhe, A. (2013), "Effectiveness of biochar for sorption of ammonium and phosphate from dairy effluent", *Journal of environmental quality,* vol. 42, no. 5, pp. 1545-1554.

Schouw, N. L., Danteravanich, S., Mosbaek, H. and Tjell, J. C. (2002), "Composition of human excreta — a case study from Southern Thailand", *Science of The Total Environment,* vol. 286, no. 1–3, pp. 155-166.

Serna-Maza, A., Heaven, S. and Banks, C. J. (2014), "Ammonia removal in food waste anaerobic digestion using a side-stream stripping process", *Bioresource technology,* vol. 152, no. 0, pp. 307-315.

Shetty, P. and Kurpad, A. (1986), "Increasing starch intake in the human diet increases fecal bulking", *The American Journal of Clinical Nutrition,* vol. 43, no. 2, pp. 210-212.

Shmerling, D., Forrer, J. and Prader, A. (1970), "Fecal fat and nitrogen in healthy children and in children with malabsorption or maldigestion", *Pediatrics,* vol. 46, no. 5, pp. 690-695.

Silvester, K. R., Bingham, S. A., Pollock, J. R. A., Cummings, J. H. and O'Neill, I. K. (1997), "Effect of meat and resistant starch on fecal excretion of apparent N-nitroso compounds and ammonia from the human large bowel", *Nutrition and Cancer,* vol. 29, no. 1, pp. 13-23.

Smith, K., Metcalfe, P., Grylls, J., Jeffrey, W. and Sinclair, A. (2007), *Nutrient value of digestate from farm-based biogas plants in Scotland.Report for Scottish Executive Environment and Rural Affairs Department-ADA/009/06,* .

Smith, S., Lang, N., Cheung, K. and Spanoudaki, K. (2005), "Factors controlling pathogen destruction during anaerobic digestion of biowastes", *Waste Management,* vol. 25, no. 4, pp. 417-425.

Snyder, W., Cook, M., Tipton, I., Nasset, E., Karhausen, L. and Howells, G. (1975), *Reference Man: Anatomical, Physiological and Metabolic Characteristics,* Report of Task Group on Reference Man-International Commission on Radiological Protection, Pergamon Press Ltd, New York.

Song, Z., Zhang, C., Yang, G., Feng, Y., Ren, G. and Han, X. (2014), "Comparison of biogas development from households and medium and large-scale biogas plants in rural China", *Renewable and Sustainable Energy Reviews,* vol. 33, no. 0, pp. 204-213.

Southgate, D. (1973), "Fibre and the other unavailable carbohydrates and their effects on the energy value of the diet", *Proceedings of the Nutrition Society,* vol. 32, no. 03, pp. 131-136.

Southgate, D. and Durnin, J. (1970), "Calorie conversion factors. An experimental reassessment of the factors used in the calculation of the energy value of human diets", *British Journal of Nutrition,* vol. 24, no. 02, pp. 517-535.

Southgate, D. A. T., Branch, W. J., Hill, M. J., Drasar, B. S., Walters, R. L., Davies, P. S. and Baird, I. M. (1976), "Metabolic responses to dietary supplements of bran", *Metabolism,* vol. 25, no. 10, pp. 1129-1135.

Speece, R. (1996), *Anaerobic Biotechnology for Industrial Wastewaters,* Archae Press, Tennessee, USA.

Speece, R. E. (2008), *Anaerobic biotechnology and odor/corrosion control for municipalities and industries,* 1st ed, Archae Press, Tennessee, USA.

Stephen, A. M., Wiggins, H., Englyst, H., Cole, T., Wayman, B. and Cummings, J. (1986), "The effect of age, sex and level of intake of dietary fibre from wheat on

large-bowel function in thirty healthy subjects", *British Journal of Nutrition,* vol. 56, no. 02, pp. 349-361.

Stephen, A. M. and Cummings, J. H. (1980), "The microbial contribution to human faecal mass", *Journal of Medical Microbiology,* vol. 13, no. 1, pp. 45-56.

Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) (2014), *Faecal Sludge Management: Systems Approach for Implementation and Operation,* 1st ed, IWA Publishing, London, UK.

Strauss, M. (1985), *Health aspects of nightsoil and sludge use in agriculture and aquaculture. Part II: pathogen survival,* 04/85, International Reference Centre of Waste Disposal, Duebendorf, Switzerland.

Suen, W. S., Mok, C. K., Chiu, S. W., Cheung, K. L., Lee, W. T., Cheung, D., Das, S. R. and He, G. W. (1998), "Risk factors for development of acute renal failure (ARF) requiring dialysis in patients undergoing cardiac surgery", *Angiology,* vol. 49, no. 9, pp. 789-800.

Sustainable Sanitation Alliance (2013)*, Research projects funded by Bill and Melinda Gates Foundation*, available at: http://www.susana.org/lang-en/research/funded-by-bill-a-melinda-gates-foundation (accessed January 20th 2014).

Takahashi, Y., Wydeven, T. and Koo, C. (1989), "Subcritical and supercritical water oxidation of CELSS model wastes", *Advances in Space Research,* vol. 9, no. 8, pp. 99-110.

Talley, N., Weaver, A., Zinsmeister, A. and Melton 3rd, L. (1993), "Functional constipation and outlet delay: a population-based study.", *Gastroenterology,* vol. 105, no. 3, pp. 781.

Talley, N. J., O'Keefe, E. A., Zinsmeister, A. R. and Melton III, L. J. (1992), "Prevalence of gastrointestinal symptoms in the elderly: A population-based study", *Gastroenterology,* vol. 102, no. 3, pp. 895-901.

Tan, Y., Goh, K. L., Muhiyahah, R., Ooi, C. L. and Salem, O. (2003), "Prevalence of irritable bowel syndrome in young adult Malaysians: A survey among medical students", *Journal of gastroenterology and hepatology,* vol. 18, no. 12, pp. 1412-1416.

Tandon, R. K. and Tandon, B. N. (1975), "Letter: Stool weights in north Indians", *Lancet,* vol. 2, no. 7934, pp. 560-561.

Tarpila, S., Miettinen, T. and Metsäranta, L. (1978), "Effects of bran on serum cholesterol, faecal mass, fat, bile acids and neutral sterols, and biliary lipids in patients with diverticular disease of the colon", *Gut,* vol. 19, no. 2, pp. 137-145.

Taylor, E. N. and Curhan, G. C. (2006), "Body size and 24-hour urine composition", *Am J Kidney Dis,* vol. 48, no. 6, pp. 905-915.

Taylor, E. N. and Curhan, G. C. (2007), "Differences in 24-hour urine composition between black and white women", *Journal of the American Society of Nephrology,* vol. 18, no. 2, pp. 654-659.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Thompson, W., Heaton, K., Smyth, G. and Smyth, C. (2000), "Irritable bowel syndrome in general practice: prevalence, characteristics, and referral", *Gut,* vol. 46, no. 1, pp. 78-82.

Thornton, J. (1981), "High colonic pH promotes colorectal cancer", *The Lancet,* vol. 317, no. 8229, pp. 1081-1083.

Tilley, E., Supply, W. and Council, S. C. (2008a), *Compendium of sanitation systems and technologies,* 1st ed, Swiss Federal Institute of Aquatic Science and Technology, Geneva, Switzerland.

Tilley, E., Supply, W. and Council, S. C. (2008b), *Compendium of sanitation systems and technologies,* Eawag Dübendorf, Switzerland.

Tilley, E., Atwater, J. and Mavinic, D. (2008c), "Recovery of struvite from stored human urine", *Environmental Technology,* vol. 29, no. 7, pp. 797-806.

Tilmans, S., Russel, K., Sklar, R., Page, L., Kramer, S. and Davis, J. (2015), "Container-based sanitation: assessing costs and effectiveness of excreta management in Cap Haitien, Haiti", *Environment and Urbanization,* vol. 27, no. 1, pp. 89-104.

Tiquia, S., Tam, N. and Hodgkiss, I. (1996), "Microbial activities during composting of spent pig-manure sawdust litter at different moisture contents", *Bioresource technology,* vol. 55, no. 3, pp. 201-206.

Triolo, J. M., Sommer, S. G. and Pedersen, L. (2014), "Influence of freezing/thawing and drying/milling on Biochemical Methane Potential", *Environmental Engineering and Management Journal,* .

UNDP (2011), *Human Development Report 2011; Sustainability and Equity,* 1, United Nations, Washington DC, USA.

United Nations (1984), *Updated guidebook on biogas development,* 1st ed, United Nations, New York.

Van Dokkum, W., De Boer, B., Van Faassen, A., Pikaar, N. and Hermus, R. (1983), "Diet, faecal pH and colorectal cancer.", *British Journal of Cancer,* vol. 48, no. 1, pp. 109-110.

Van Faassen, A., Hazen, M. J., van den Brandt, P. A., van den Bogaard, A. E., Hermus, R. and Janknegt, R. (1993), "Bile acids and pH values in total feces and in fecal water from habitually omnivorous and vegetarian subjects", *The American Journal of Clinical Nutrition,* vol. 58, no. 6, pp. 917-922.

Vassileva, P., Tzvetkova, P. and Nickolov, R. (2009), "Removal of ammonium ions from aqueous solutions with coal-based activated carbons modified by oxidation", *Fuel,* vol. 88, no. 2, pp. 387-390.

Vinnerås, B., Palmquist, H., Balmér, P. and Jönsson, H. (2006), "The characteristics of household wastewater and biodegradable solid waste—a proposal for new Swedish design values", *Urban Water Journal,* vol. 3, no. 1, pp. 3-11.

Vinneras, B. (2002), *Possibilities for sustainable nutrient recycling by faecal seperation with urine diversion* (Doctoral Thesis thesis), Swedish University of Agricultural Sciences, Uppsala, Sweden.

Vögeli, Y., Riu, C., Amalia, L., Diener, S. and Zurbrügg, C. (2014), *Anaerobic digestion of biowaste in developing countries,* , Eawag – Swiss Federal Institute of Aquatic Science and Technology, Dübendorf, Switzerland.

Volk, T. and Rummel, J. D. (1987), "Mass balances for a biological life support system simulation model", *Advances in Space Research,* vol. 7, no. 4, pp. 141-148.

Vuksan, V., Jenkins, A. L., Jenkins, D. J. A., Rogovik, A. L., Sievenpiper, J. L. and Jovanovski, E. (2008), "Using cereal to increase dietary fiber intake to the recommended level and the effect of fiber on bowel function in healthy persons consuming North American diets", *The American Journal of Clinical Nutrition,* vol. 88, no. 5, pp. 1256-1262.

Walker, A. and Walker, B. (1992), "Faecal pH and colon cancer.", *Gut,* vol. 33, no. 4, pp. 572-572.

Walker, A., Walker, B. and Segal, I. (1979), "Faecal pH value and its modification by dietary means in South African black and white schoolchildren", *South African Medical Journal,* vol. 55, no. 13, pp. 495-498.

Walker, C. F. and Black, R. E. (2004), "Zinc and the risk for infectious disease", *Annu.Rev.Nutr.,* vol. 24, pp. 255-275.

Wang, S. and Wu, H. (2006), "Environmental-benign utilisation of fly ash as low-cost adsorbents", *Journal of hazardous materials,* vol. 136, no. 3, pp. 482-501.

Wang, Y., Liu, S., Xu, Z., Han, T., Chuan, S. and Zhu, T. (2006), "Ammonia removal from leachate solution using natural Chinese clinoptilolite", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 735-740.

Ward, A. J., Hobbs, P. J., Holliman, P. J. and Jones, D. L. (2008), "Optimisation of the anaerobic digestion of agricultural resources", *Bioresource technology,* vol. 99, no. 17, pp. 7928-7940.

WHO/UNICEF (2012), *Progress on drinking water and sanitation; 2012 update,* MDG assessment report, Joint Monitoring Programme for Water Supply and Sanitation., U.S.A.

Wierdsma, N. J., Peters, J. H. C., Weijs, P. J. M., Keur, M. B., Girbes, A. R. J., van Bodegraven, A. A. and Beishuizen, A. (2011), "Malabsorption and nutritional balance in the ICU: fecal weight as a biomarker: a prospective observational pilot study", *Critical Care,* vol. 15, no. 6, pp. R264.

Wignarajah, K., Pisharody, S. and Fisher, J. W. (2003), *Investigating the partitioning of inorganic elements consumed by humans between the various fractions of human wastes: An alternative approach,* 2003-01-2371, SAE International, Moffett Field, California, USA.

Wilkie, A. C. and Mulbry, W. W. (2002), "Recovery of dairy manure nutrients by benthic freshwater algae", *Bioresource technology,* vol. 84, no. 1, pp. 81-91.

Williams, J. B., Clarkson, C., Mant, C., Drinkwater, A. and May, E. (2012), "Fat, oil and grease deposits in sewers: Characterisation of deposits and formation mechanisms", *Water research,* vol. 46, no. 19, pp. 6319-6328.

Woodmansey, E. J., McMurdo, M. E. T., Macfarlane, G. T. and Macfarlane, S. (2004), "Comparison of compositions and metabolic activities of fecal microbiotas in young adults and in antibiotic-treated and non-antibiotic-treated elderly subjects", *Applied and Environmental Microbiology,* vol. 70, no. 10, pp. 6113-6122.

Wooley, S. M., Cottingham, R. S., Pocock, J. and Buckley, C. A. (2013), "Some rheological properties of fresh human faeces with a variation in moisture content", *Water S. A.,* vol. In Press.

WRAP (2012), *Using quality anaerobic digestate to benefit crops,* 1, Waste and resource action plan, UK.

WSUP (2014), *FSM sercices in x: moving up the excreta management ladder,* pn#017, Water and Sanitation for the Urban Poor, London, UK.

Xiu, S., Shahbazi, A., Shirley, V. and Cheng, D. (2010), "Hydrothermal pyrolysis of swine manure to bio-oil: Effects of operating parameters on products yield and characterization of bio-oil", *Journal of Analytical and Applied Pyrolysis,* vol. 88, no. 1, pp. 73-79.

Yang, J., Wang, H., Zhou, L. and Xu, C. (2012), "Effect of dietary fiber on constipation: A meta analysis", *World Journal of Gastroenterology: WJG,* vol. 18, no. 48, pp. 7378.

Young, H. D. (1962), *Statistical treatment of experimental data,* 1st ed, McGraw-Hill, New York, USA.

Yowler, C. and Fratianne, R. (2000), "Current status of burn resuscitation.", *Clinics in plastic surgery,* vol. 27, no. 1, pp. 1.

Zuckerman, M. J., Guerra, L. G., Drossman, D. A., Foland, J. A. and Gregory, G. G. (1995), "Comparison of bowel patterns in Hispanics and non-Hispanic whites", *Digestive Diseases and Sciences,* vol. 40, no. 8, pp. 1763-1769.

CHAPTER 3

THE CHARACTERISATION OF FAECAL SLUDGE FROM ON-SITE SANITATION SYSTEMS AND APPLICATION TO ANAEROBIC TREATMENT TECHNOLOGIES IN LOW INCOME COUNTRIES

3 THE CHARACTERISATION OF FAECAL SLUDGE FROM ON-SITE SANITATION SYSTEMS AND APPLICATION TO ANAEROBIC TREATMENT TECHNOLOGIES IN LOW INCOME COUNTRIES

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ABSTRACT

Anaerobic digestion as a treatment option for high solids waste streams from on-site sanitation systems was assessed. Faecal sludge was collected and characterised from pit latrines, unsewered public toilets, portable chemical toilets, and a portable chemical toilet holding tank. The readily degradable organic fraction of pit latrine sludge was lower than other faecal sludge types as illustrated by biochemical methane potential assays (49 and 281 mL CH_4/g

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VS$_{added}$ in pit latrines and portable toilets respectively). The highest species richness and diversity was observed in pit latrine samples, which also displayed the greatest degree of sameness in comparison to public toilets. Despite low methane production values, pit latrine sludge was treatable using anaerobic digestion with a C: N ratio of 12:1 and had significant nutrient recovery potential (1853 mg NH$_4$-N L^{-1}). Portable toilet waste had significant potential for anaerobic treatment; however, current use of odour suppressing chemical additives introduces heavy metals to the waste stream (27 g/kg TS Cu) and the active ingredients can supress methane production with IC$_{50}$ values of 100 mg L^{-1} glutaraldhyde and ≥50 mg L^{-1} bronopol.

KEYWORDS

Faecal sludge, pit latrine, faecal sludge treatment, anaerobic digestion, portable toilets, sanitation.

HIGHLIGHTS

- Comprehensive characterisation of four different types of faecal sludge completed.

- High solids pit latrine sludge (19%TS) with large NH_4-N concentration (1853 mg L^{-1}).

- Chemical additives cause inhibition of CH_4 production and introduce heavy metals.

- Species diversity greatest in pit latrines with high degree of similarity between sites

- Anaerobic digestion is suited to treatment of faecal sludge in low income countries.

3.1 INTRODUCTION

The predominant form of excreta disposal in urban areas of much of Africa and Asia is through on-site sanitation (OSS) facilities (Strauss et al., 2000), which are unsewered systems in use as public or household facilities. However, these systems eventually reach capacity and require emptying; but poor design coupled with a dense urban environment makes this process challenging (Buckley et al., 2008). Faecal sludge (FS) from sanitation facilities that are emptied is often disposed of untreated due to the lack of sustainable sludge management and treatment options (Strande et al., 2014). Low cost and efficient treatment mechanisms for FS in low income countries remains a challenge. This stems in part from the variability and limited knowledge of physical, chemical and biological characteristics of FS from different forms of sanitation facilities. A full knowledge of the characteristics of FS is therefore essential for not only the design and sizing of subsequent treatment plants but also for the advancement and development of technologies to suit this context..

Wastewater characterisation information is available on faeces and urine as a fresh waste stream (Meinzinger and Oldenburg, 2009; Rose et al., 2015), sanitary sewage from water-borne sewage networks, as well as from the resultant sewage sludge following the treatment of water-borne sanitary sewage (such as primary and waste activated sludge) (Henze et al., 2001; Tchobanoglous et al., 2003). However, this material is substantially different to that of FS from OSS systems as the material will have undergone different periods of storage and storage conditions as well as different levels of dilution through flush-water and grey-water additions.

Therefore, FS from pit latrines, unsewered public toilets and portable toilets were investigated in this study. Pit latrines are the most commonly used form of OSS systems in developing countries with an estimated 1.77 billion people around the world using some form of pit latrine (Graham and Polizzotto, 2013). Public toilet and ablution blocks where toilet and grey-water waste is collected in a sealed holding tank and emptied on a regular basis are also in widespread use (Drechsel et al., 2009). Finally, a new but additional expanding sector of OSS services is the provision of portable toilets to households which are then collected regularly (for example *Clean Team* in Ghana (Clean Team, 2012); these toilets provide another example of FS from a different OSS facility.

The safe treatment of FS from OSS facilities is one of the most important factors in effective faecal sludge management in low income countries (Muspratt et al., 2014). Anaerobic digestion (AD) is commonly used for treating sewage sludge from wastewater treatment works in high income countries and there is significant potential for AD as a treatment mechanism in a low income context (Strande et al., 2014). However, many factors impact the performance of AD and the physical and chemical characteristics of the feedstock is vital information for the design and operation of AD facilities as it will affect biogas production and process stability. Consequently, understanding how FS characteristics of a wide range of OSS facilities will impact the AD process is of great significance.

The main objective of this work was to undertake a full characterisation of pit latrine waste, unsewered public toilet holding tank waste, portable toilet waste as well as portable toilet sludge after storage in an IBC tank all of which may become an increasingly common feedstock for AD in low income countries. Characterisation analysis was conducted to help define physico-chemical and microbial composition variability, gross energy values and methane production potential in order to assess the treatability of FS by AD. In addition, analysis was undertaken to support AD design and operation as well as to assess the health and environmental risks that could influence the handling and disposal of FS.

3.2 MATERIALS AND METHODS

3.2.1 Survey sites and study population

The peri-urban district of *location x* was used for the collection of pit latrine (n=11) and unsewered public toilet (n=3) samples in February/March 2013. Pit latrines in this region were raised above ground level and had no vent pipes present. Community ablution blocks consisted of 4-12 flush toilets, urinals as well as running water used for washing and showering. This waste was then stored in lined holding tanks that were set below ground level.

A pilot scheme (Clean Team, 2012) using specially designed portable toilets and a regular collection service was used as a site for portable toilet sludge characterisation in Kumasi, Ghana in July/August 2013. Portable toilets (n=36) were designed to be urine diverting toilets with only the faeces fraction collected, in a detachable container at the base of the toilet structure, and collections took place at least once every 4 days from households (average household users 4.2). Faeces were collected in this container which contained a commercially available toilet chemical additive (Active ingredients: Pentane 1,5 diol 5-10%, 2-Bromo-2-Nitropropane-1,3-diol <5%) which was diluted in 5 -7 L of water in order to prevent odour issues. Collected portable toilet waste was subsequently emptied into an IBC (Intermediate Bulk Container, 1 m^3 volume) (n=1) where it was stored for approximately 1 month prior to final treatment and disposal.

Institutional ethical approval (*Cranfield University 109-2013)* was obtained for the sampling and analysis of faecal sludge in both case study locations: *location x* and Kumasi, Ghana.

3.2.2 Analytical methods

On-site measurements of pH and temperature were recorded using a portable YSI G3 60 meter (YSI Ltd., Hampshire, UK) and the oxidation reduction potential (ORP) was measured using an ORP15 pen (model number I662-0121, VWR International, Leuven, Belgium). Bulk FS samples were taken (containing a combined mixture of municipal solid waste and faecal material) and aliquots of 2 L were put into sealed plastic bags and stored on ice in a cool bag before being transported to the laboratory (approximate journey time of 1 hour) where they were refrigerated ($4^{o}C \pm 0.5°C$) on arrival. A plastic sample bag was also filled with FS and was immediately frozen ($-20°C \pm 0.5°C$) once at the laboratory.

In the laboratory set up at each case study location 2 L of each bulk sample was homogenised using a mechanical blender according to the methods of Buckley et al. (2008), any material that could not be blended was discarded and classified as municipal solid waste (MSW). All samples were analysed in triplicate and the mean and standard deviation recorded. Total solids (TS), volatile solids (VS), total nitrogen (TN), ammonium (NH_4-N) and faecal coliforms were all determined on the homogenised wet sample. In addition a frozen aliquot of the complete sludge sample was stored and used for biochemical methane potential (BMP). The TS and VS were measured according to standard methods (APHA, 2005), TN was determined after digestion according to the Koroleff method (analogous to EN ISO 11905-1) and was measured photo-metrically (analogous to DIN 38405 D9) and NH_4-N was measured according to DIN 38406 E5.

The homogenised sludge sample was also centrifuged for 10 minutes at 5000 g (Hettich Zentrifugen, Tuttlingen, Germany) after which the supernatant was filtered through a 0.45µm filter (Sartorious, Epsom, UK) in order to give the soluble solid free fraction. Analysis was undertaken for COD_{sol}, nitrate (NO_3-N),

total phosphorus and potassium according to standard methods (APHA, 2005). An aliquot of 5 mL was immediately frozen (-20ºC) awaiting analysis for soluble magnesium and volatile fatty acid concentrations.

A range of six volatile fatty acids (VFA) concentrations were measured and determined on the soluble solid free fraction using high pressure liquid chromatography (HPLC) (535 Kontron, Bio-TEK, UK) with a Bio-Rad fermentation column (Cat 125-0115) 300 x 7.8 mm maintained at 65ºC, with a UV detector at 210 nm. The sample volume was 50µl. All samples for VFA analysis were first acidified with concentrated H_2SO_4 to pH <2 according to Parawira et al. (2004) and a hydraulic flow rate of 0.8 mL/min used. An external multi-level calibration range from 0.1 g/L to 5 g L was used to quantify acetic, propionic, n-butyric, i-valeric and n-valeric acids.

The solid fraction was obtained by drying c.0.5 L of FS at 40ºC±0.5 for 48 hours, to avoid potential volatilisation of nutrients and prevent further degradation of the samples. The samples were then sealed in plastic bags to await subsequent analysis. Quantitative elementary analysis was undertaken using a Vario EL (Elementar, Hanau, Germany) for % C, H and N according to ISO 10694. Total P in the dried solids fraction was measured according to US EPA Method 3051 and determined photometrically (Helios Gamma, New Brunswick, USA). Potassium (K), magnesium (Mg) and calcium (Ca) as well as heavy metals were determined after microwave digestion with a nitric/hydrochloric acid mixture and was measured using Atomic Adsorption Spectroscopy (AAnalyst 800, Perkin Elmer, Waltham, USA) according to APHA (2005).

Biochemical methane potential (BMP) assays were carried out according to Owens and Chynoweth (1993) and Angelidaki at al. (2009). The anaerobic inoculum was collected from a mesophilic digester at a sewage treatment works (population equivalent (PE) of 288,000) and a stock solution of micronutrients (according to Gonzalez-Gil et al. (2001)) was added to each assay. Each assay was flushed with N_2/CO_2 (80:20% as volume) after transfer of inoculum and substrate before being anaerobically incubated for 32 days in a temperature

controlled shaker (37.5^{o}C ± 0.5: 150 rev min^{-1})((Excella E24, New Brunswick Scientific, Edison, USA). Cellulose was used as a positive control in all experiments and assays were carried out in quadruplicates. Blanks were run with inoculum only with no substrate addition and biogas production from the inoculum was subtracted from methane production values of tested substrates. Total gas volumes were calculated by water displacement. The production of biogas was given at standard temperature and pressure according to Angelidaki et al. (2009). The % methane (CH_4) in biogas samples was measured using a Servomex 1440 (Zoetermere, Netherlands) gas analyser.

Potential methane inhibition caused by the chemical toilet additive in use was investigated by determining the IC50 values of the two main active ingredients of the chemical additive (Glutaraldehyde (Pentane 1,5 Dial) and Bronopol (2-Brono-2-Nitropropane-1,3 Dial). In order to test the inhibitory effect of these chemicals a primary sludge from the 288,000 PE sewage treatment plant was used as a substrate and a range of inhibitory and sub-inhibitory concentrations of Glutaraldehyde (10, 50, 100, 500, 5000 and 10000 mg L^{-1}) and Bronopol (10, 50, 100, 500, 1000, 10000 mg L^{-1}) were added to the BMP assays completed as outlined above. Any inhibitory effect was determined by assessing the IC_{50} value and was compared to a control group with no chemical addition.

Thermotolerant coliform determination, DNA extraction and sequencing were carried out on pit latrine and portable toilet sludge samples only. Thermotolerant coliforms were determined within 4 hours of sample collection using membrane filtration and incubated on membrane lauryl sulphate broth at 44°C ±0.5°C for 14 hours following the standard method for detection and enumeration of coliforms (APHA, 2005). DNA extraction and sequencing was conducted on homogenous sludge samples taken from 11 pit latrines and 3 public toilets as well as additional depth samples from the top (0.05 m), middle (1 m) and bottom (2.5 m) (total pit latrine depth 2.9 m) from one of the pit latrines (S863). One pit latrine was a dry urine diversion pit latrine and was identified as an ECOSAN latrine. All samples were stored at -20°C ± 0.5°C prior to DNA extraction. Methods of DNA extraction, next generation sequencing library construction and PCR amplification are provided in detail in Appendix B. The pooled multiplexed

library was sequenced using the Illumina Miseq (San Diego, USA) bench-top sequencer at the Centre for Genomic Research, Liverpool.

3.3 RESULTS AND DISCUSSION

3.3.1 Physical composition of faecal sludge

Pit latrines had the highest TS concentration of the differing FS types with a mean of 19.24% TS (Std. Err.1.63). Within pit latrines a higher TS concentration (27.34% TS) was recorded in the top layer of the latrine vault in comparison to the bottom section of the pit latrine (19.21% TS) (Table 3.1). The high TS concentration is unusual in comparison to FS from other OSS facilities, such as septic tanks, where the TS concentrations are routinely lower (0.88-11.4% TS: Table 3.2). Nevertheless the TS of the pit latrine FS is within the upper range of literature values for pit latrines (Table 3.2). The higher than average TS concentrations are likely to be due to the unlined nature of most pit latrines in this study. Percolation out of the latrine vault will only take place if pit latrines are cited above the groundwater level meaning a net movement of water away from the latrine vault: in this study all pit latrines were raised above ground level due to a high water table existing in the area, this means that the majority of the latrine vault is likely to be above groundwater levels, consequently leading to net water loss through the side walls. However, the seepage of water out of pit latrines is likely to be retarded significantly by clogging of soil pores at the pit:soil interface, nevertheless infiltration will still take place and contribute to high TS concentrations. An additional factor for the high TS observed in pit latrine FS may also be due to the "wiping" anal cleansing practices of the area (the use of newspaper and/or rags) as well as the pit latrine emptying methods used (the use of shovels) meaning a large proportion of the material removed was likely to be of a spadeable consistency (15-20% TS).

Table 3.1 Comparative analysis of the top and bottom layers within a pit latrine (*n*=1) with mean values presented (standard deviation).

n = 1	Top Layer	Bottom Section
Total Solids (% g/wet mass)	27.34 (2.34)	19.21 (0.67)
Volatile Solids (% g/TS)	36.23 (0.83)	35.02 (1.34)
NH_4-N (mg L^{-1})	2570 (129)	3100 (264)

COD$_{tot}$ (mg L^{-1})	274900 (33300)	126600 (3600)
COD$_{sol}$ (mg L^{-1})	17780 (260)	25380 (1060)
Total P$_{sol}$ (mg L^{-1})	119 (1)	120 (2)

Table 3.2 Literature values of the physio-chemical characteristics for similar FS types to this study.

	Production (per capita L.day)	Production (per capita g.day)	BOD (mg L^{-1})	COD (mg L^{-1})	TS (%)	VS (% of TS)	Total N (mg L^{-1})	pH	References
Nightsoil[a]	1-2.55	940-1800	10588-3200	20000-175000	2.242-5.1	71-89.6	950-9520	5.75-8.5	(Fenner et al., 2007; Doku, 2003; Panuvatvanich et al., 2009; Meinzinger and Oldenburg, 2009; Czemiel, 2000; Heinss et al., 1998; Choi et al., 2004; Pradt, 1971; Issah, 2011; Takahashi et al., 1989; Gajurel et al., 2003; Strauss, 1985; Schouw et al., 2002)
Pit Latrine	0.07-1.5	*	7600-15000	49000-103300	5.25-57	41-69	3000-5000	7.9	(Heinss et al., 1999; Doku, 2003; Chaggu, 2004; Coetzee et al., 2011; Hawkins, 1982; Foxon et al., 2009; Salisbury et al., 2009; Buckley et al., 2008; Norris, 2000)
Septage	0.3-1	*	600-5500	4200-76000	0.887-11.4	60-76	190-2100	6.7-8	(Heinss et al., 1999; Koottatep, T., Surinkul, N., Paochaiyangyuen, R., Suebsao, W., Sherpa, M., Liangwannaphorn, C., Panuwatvanich, A., 2012; Koné and Strauss, 2004; Norris, 2000; Choi et al., 2004)

*Values not available

[a] Nightsoil defined as any human derived waste collected at regular intervals as a fresh waste stream

Public toilet sludge had a mean value of 7.01% TS (Std. Err. 2.63) with values ranging from 2.94 - 11.94 %TS, which is within the range of values reported in the literature for FS from septic tanks (Table 3.2). The relatively low TS concentration in comparison to pit latrines (19.24 %TS) is due to the extensive dilution by flushing toilets as well as the entirely sealed nature of the tanks which meant minimal percolation into the surrounding soil took place.

Due to the design and operation (with faeces contained in 5-7 L of liquid), as expected the portable toilets had the lowest TS content at 4.56% (Std. Err. 2.17) of all FS types studied. However, when TS was recorded in the IBC tank this increased to 9.50% TS, although the IBC was capped when not in use, moisture loss may have occurred periodically when open. A thicker layer was observed at the top of the IBC, however, no correlation between TS and depth within the IBC was observed using *Student's T-test* (p>0.05).

3.3.2 Organic composition of faecal sludge

Portable toilet sludge was collected on a regular basis (≤ 3 day intervals) and was therefore a relatively fresh waste stream that had undergone little degradation, this was reflected by high VS values (78.87% of TS). This waste stream therefore had strong similarities to organic composition of faeces at point of release (89% VS on average (Rose et al., 2015)). Values of COD_{sol} were higher in the IBC tank (25.50 g COD_{sol} L^{-1}) than within individual toilets (24.09 g COD_{sol} L^{-1}) which is due to the longer retention time in the IBC where hydrolysis/fermentation will start to occur. In pit latrines a low proportion of VS:TS (VS 48% of TS) was recorded indicating partial stabilisation of FS was occurring within latrine vaults, which coincides with findings by Buckley et al. (2008). The low proportion of VS:TS in pit latrines is not reflected in other studies. For example, values of 58-69% VS were reported by Heinss et al. (1999). The lower VS in this study is most likely caused by the protracted period of storage of the FS prior to emptying with most latrine owners reporting longer periods (≥5 years) of time since construction or the latrine's previous emptying.

The portable toilet IBC holding tank had the largest concentration of volatile fatty acids (VFA) (9023 mg VFA L^{-1}) of FS types with portable toilets exhibiting similarly high VFA concentrations at 7182 mg L^{-1}. Total VFA concentrations in pit latrines were lower with a mean of 3736 mg L^{-1} but with a larger range of 302-7077 mg L^{-1} and the lowest VFA concentrations were observed in public toilet sludge (996 mg L^{-1}). The high VFA build up in portable toilet waste is commensurate with the shorter retention time of the sludge in comparison to other FS types: the greater the retention time in the OSS facility, the more VFA will be consumed or converted to methane. In anaerobic treatment processes the design of hydrolysis/fermentation is based on a hydraulic retention time of typically 1-3 days (Tchobanoglous et al., 2003), which is a similar retention time to the portable toilets. The accumulation of VFAs in pit latrine FS is likely to have occurred because the rate of hydrolysis is faster than the rate of onward conversion of the acids. The vast proportion of VFAs in all FS types were found in acetic and propionic forms (1511 and 2101 mg L^{-1} in portable toilets and 1790 and 795 mg L^{-1} in pit latrines respectively), with the proportion of acetic acid highest in pit latrines (55% of total VFA concentration).

3.3.3 Methane potential and biodegradation of sludge

The theoretical maximum energy that could be obtained from the FS is demonstrated by gross energy values (Table 3.3) with portable toilet waste (22.24 MJ/kg TS) over double that of pit latrine waste. The portable toilet and public toilet FS had a similar calorific value to primary sludge (following primary settlement of sanitary wastewater) with Speece (2008) recording values between 23-29 MJ/kg TS. The gross energy values of pit latrines (10.24 MJ/kg TS) are comparatively lower than other studies such as Muspratt et al. (2014) who reported figures averaging 16.2 MJ/kg TS in similar partially lined pit latrines in Uganda. The lower values measured in this study in *location x* are likely to be due to the extensive period of time by which pit latrines were emptied (most owners reported irregular emptying of >3 years in frequency) indicating a well digested sludge type.

Due to the younger age of portable toilet waste (3-4 days old), BMP values were significantly greater than that of pit latrine FS (>3 years old) (Figure 1). The average BMP value of portable toilet waste (281 mL CH_4/g VS_{added}) falls within the range of values reported for faeces (260-300 mL CH_4/g VS_{added}) by Lim et al. (2011) and Rajagopal et al. (2013), however, the range of portable toilet values in this study was considerably greater (97 to 604 mL CH_4/g VS_{added}) which could be due to variation in factors such as the number of users or the interval between collections. Cumulative CH_4 production values in pit latrines are considerably lower than that of any other FS substrate in this study (Figure 1) but also when compared to piped sanitary sewage sludge with values reported between 230-590 mL CH_4/g VS_{added} for a sludge following primary settlement (Chynoweth et al., 1993; Elbeshbishy et al., 2012). This illustrates that the majority of the readily degradable organics have already been broken down in the pit latrine.
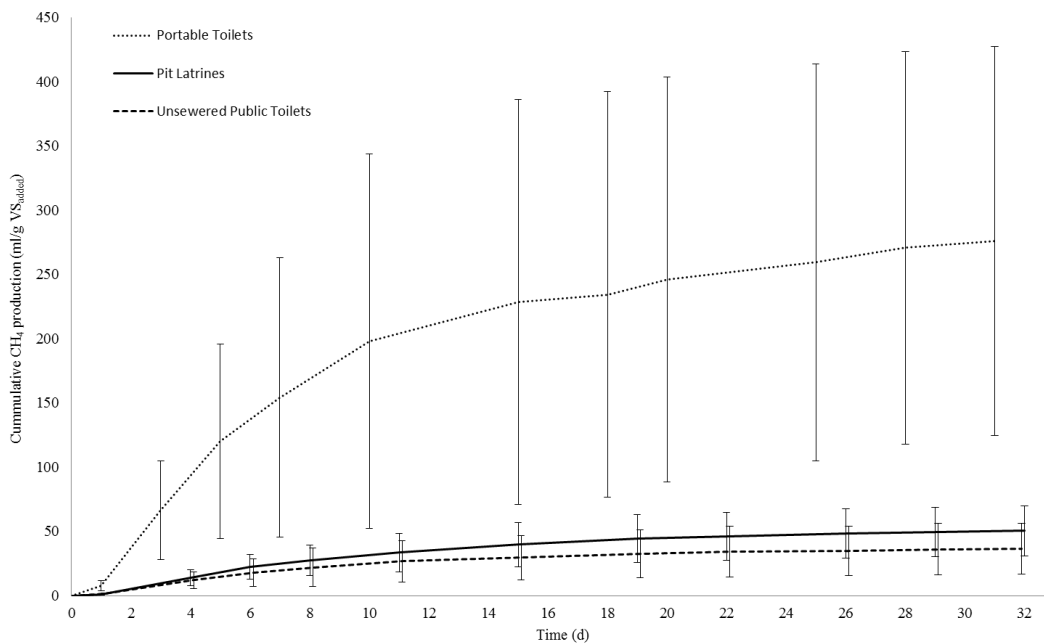
Figure 3.1 The mean biochemical methane potential of pit latrines (n =11) and unsewered public toilets (n=3) in *location x* as well as individual portable toilets (n = 7) in Ghana. Analysis of each site was undertaken in quadruplicates with the mean value of all sites presented (with standard deviation).

Methane potential tests of portable toilet sludge found that 30% of samples had extremely low biogas production values, giving cumulative methane production values of 5, 1 and 6 mL/ g VS_{added} in comparison to the average value of the other assays for this FS type (281 mL CH_4/g VS_{added}). These values were not dismissed as anomalies due to the relatively low standard deviations of the quadruplicates (2.6, 4.1, and 10.1) and high frequency within the samples (30% of samples). It was therefore hypothesised that the toilet chemical additive (used as an odour suppressant) was likely to be causing inhibition. The toxicity of the active ingredients of the chemical additive were subsequently tested and it was found that glutaraldehyde caused inhibition (IC_{50}) at concentrations of $\geq$100 mg L^{-1} and bronopol caused inhibition at concentrations of $\geq$50 mg L^{-1}. Taking into account the levels of dilution that were in use at the time of sampling, estimated concentrations in the portable toilet FS material was 200 mg L^{-1} glutaraldehyde and 100 mg L^{-1} bronopol which is above the IC_{50} levels determined. Similarly, Leung (2001) found that glutaraldehyde inhibited the metabolic activity of sewage at concentrations greater than 16 mg L^{-1}. This was considerably lower than the glutaraldehyde IC_{50} value found in this study (200 mg L^{-1}), however, this could be due to the increased amounts of bacteria present in sludge samples whereas in the more diluted conventional sewage measured by Leung (2001) there would be less opportunity for the bacteria to overwhelm the chemical as is likely to be the case in a sludge. No studies regarding the inhibitory effect of bronopol on wastewater could be found, however, as an effective antibacterial preservative (Bryce et al., 1978) it is likely to have an impact on the bacterial population and therefore IC_{50} values (100 mg L^{-1}) are relatively high which could again be accounted for by significant bacterial populations in the sludge overwhelming the addition of bronopol.

Table 3.3 Composition of faecal sludge from four different types of on-site sanitation facilities: pit latrines, unsewered public toilet holding tanks in *location x* and portable toilets and portable toilet holding tanks in Kumasi. Mean values are presented (with standard error).

	Location x			Kumasi, Ghana	
	Pit Latrines	Public Toilet Holding tank	Portable Toilets	Portable toilet IBC	Portable toilets (per cap/day)
n	*11*	*3*	*36*	*1*	*36*
pH	7.65 (0.031)	7.22 (0.025)	6.43 (0.27)	6.09 (0.16)	
Temp (^{o}C)	24.128 (0.713)	27.038 (0.813)	26.8 (1.5)	27.3 (0.2)	
ORP (mV)	-354.4 (7.7)	-280.7 (37.5)	-216.9 (147.5)	-282.7 (20.3)	
TS (% g/wet mass)	19.245 (1.638)	7.006 (2.634)	4.56 (2.17)	9.50 (5.82)	28.21 g (14.82)
VS (% g/TS)	47.767 (2.906)	70.581 (3.530)	78.87 (8.80)	85.45 (1.00)	22.38 g (12.68)
COD_{sol} (g/L)*	9.444 (2.219)	3.263 (2.498)	24.09 (9.33)	25.5 (6.73)	13.40 g (7.38)
Total N (mgL^{-1}	4561 (439)	2090 (733)	2590 (1190)	2080 (54.2)	1360 mg (560)
NH_4-N (mgL^{-1})	1853 (178)	846 (267)	520 (400)	948.8 (412.9)	250 mg (190)
NO_3 (mgL^{-1})*	10.83 (3.08)	2.99 (1.26)	68.8 (26.3)		55.6 mg (38.5)
Total P mgL^{-1}*	85.52 (14.6)	111.52 (45.65)	396.2 (149.0)	484.3 (120.9)	226.1 mg (156)
Total K mgL^{-1}*	1236 (306)	766 (647)	29100 (0.01)		13.4 mg (2.9)
Mg mgL^{-1}*	70.65 (33.29)	31.84 (4.43)	303.57 (102.16)	278 (68.94)	
%C	24.156 (1.567)	40.113 (2.248)	52.4994 (1.450)	49.821 (0.528)	
%H	3.738 (0.230)	5.997 (0.426)	7.998 (0.180)	7.576 (0.097)	
%N	2.122 (0.157)	3.682 (0.165)	7.324 (0.642)	4.781 (0.116)	
C: N ratio	11.579 (0.5757)	10.989 (1.058)	8.546 (0.966)	10.425 (0.142)	
P (mg kg^{-1})	26950 (1291)	12106 (913)	74588 (13152)	48248 (6372)	
Gross Energy (MJ/kg TS)	10.242 (2.861)	17.581 (2.848)	22.241 (0.436)	22.296 (0.416)	
Pb (mg kg^{-1})	135 (67)	121 (41)	140 (59)	337 (85)	
Cu (mg kg^{-1})	68 (11)	98 (18)	27251 (18971)	10330 (3452)	
Cr (mg kg^{-1})	<DL	<DL	83 (63)	58 (14)	
Zn (mg kg^{-1})	420 (281)	755 (298)	2538 (883)	1993 (509)	
Ni (mg kg^{-1})	84 (26)	75 (28)	177 (31)	196 (27)	
Cd (mg kg^{-1})	1.9 (0.3)	2.6 (0.4)	0.249 (0.098)	0.349 (0.018)	
Production rate (mL/cap/d)					244.0 (178.1)

*Determined on the soluble solid free fraction

3.3.4 Chemical composition and nutrient value of faecal sludge

The pH levels of pit latrine and public toilet FS were around neutral (7.22 – 7.65 Table 2) and were equivalent to Heinss et al. (1999) measurements of FS from septic tank collections in Ghana (pH 7.9). In contrast to this, the FS of individual portable toilets was more acidic (pH 6.43) and could be accounted for by the odour suppressant chemical additive or alternatively by the increased VFA formation in the IBC (26% increase in VFAs following storage).

As would be expected due to the addition of urine (urine comprises 68% of total N outputs in excreta (Rose et al., 2015)), the FS of pit latrines and public toilets had higher total nitrogen concentrations than that of portable toilets (Table 3.3). Within the solid fraction (Table 3.3), N made up 2.1% and 3.7% in pit latrines and public toilets respectively which is also within the range seen in primary (1.5-4%) and waste activated sewage sludge (2.4-5%) (Speece, 2008). However, values were significantly greater in portable toilet sludge (7% N) which subsequently reduced the C:N balance in the portable toilet waste stream (8:5).

3.3.5 Biological pathogen indicators

The mean value of faecal coliforms in pit latrine FS was 6.40×10^7 CFU/100 mL with a range of $4.28 \times 10^5 – 5.35 \times 10^8$ CFU/100 mL. These levels are similar to levels measured by Issah et al. (2012) who recorded faecal coliform values of 3.63×10^6 in FS. Similar levels of faecal coliforms have been found in sewage sludge with 10^6-10^7, 10^7-10^9 and 10^5-10^6 recorded in primary, secondary and mixed sludge respectively (Kiely, 1997) demonstrating that there is minimal removal of pathogen indicators despite percolation of liquids out of the pit latrine vault and significant retention time of FS in the latrine vault (generally >3 years).

3.3.6 Microbial composition

The proportions of the top 20 operational taxonomic units (OTU) that were present in pit latrines and public toilets are outlined in Figure 3.2. Pseudomonas was the most prevalent OTU present in the majority of samples, a genus containing a number of aerobic and facultative species, which are often

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described as infectious pathogens. The high proportion in all waste types is a reflection of its environmental adaptability and is common in soil, water as well as humans (Stover et al., 2000). However, where Pseudomonas is not in the greatest proportion, for instance in two pit latrines (S856 and S861) and one public toilet (S862), an unclassified Comamonadaceae, a similarly aerobic family, instead makes up the vast proportion of genome composition (Figure 3.2). Comamonadaceae is a beta proteobacteria that tends to use nutrients released from anaerobic digestion of organics (He et al., 2009). Another common OTU where high levels of abundance were detected included the Microvirgula genus, a denitrifier under aerobic conditions (Otani et al., 2004) which commonly occurs where there are fluctuating oxygen conditions and has been reported in aerobic and anoxic wastewater sludge (Patureau et al., 2000; Patureau et al., 2001). A high abundance of aerobic microbes was therefore present throughout all types of OSS system, which is interesting as it was expected that conditions within all OSS systems would be predominantly anaerobic. It is also evident that the OTU proportions of pit latrines are relatively similar between individual sites in comparison to public toilet samples which differ markedly between sites (Figure 3.2). This factor is also reflected by the high degree of clustering of pit latrines in comparison to public toilets (Figure 3.3) which indicates a stronger degree of OTU similarity between individual pit latrine sites.

Species diversity, as measured by the alpha diversity index, Shannon and Simpson diversity measures, was greatest in pit latrine samples in comparison to samples taken from public toilets (Figure 3.4). This is a reflection of the longer residence time under anaerobic conditions of pit latrines in comparison to public toilets. The difference in species diversity is important as it can have an effect on the stability of a subsequent AD treatment process if applied with Fernandez et al. (2000) reporting that the stability of reactors with greater diversity was less variable than those with reduced diversity during the start-up period.

In order to illustrate overall variation between different OSS facilities, the degree of similarity of microbial community structures is depicted in one single non-

metric multidimensional (NMDS) plot (Figure 3.3). Representative samples of the entire pit latrine contents had the greatest proportion of clustering in comparison to that of public toilets (Figure 3.3). This could be a reflection of residence time within the system or the similarity of pit latrines due to their close proximity (<5 km radius). It is also apparent, that as expected, the community structure of the sample taken from the top of a pit latrine is substantially different to that of all other types of OSS facility (Figure 3.3). This illustrates the undigested nature of this fraction and the more prevalent aerobic degradation that is taking place as opposed to the prolonged anaerobic conditions prevailing in all other OSS facilities measured resulting in the closer proximity of these OSS systems within the plot (Figure 3.3).
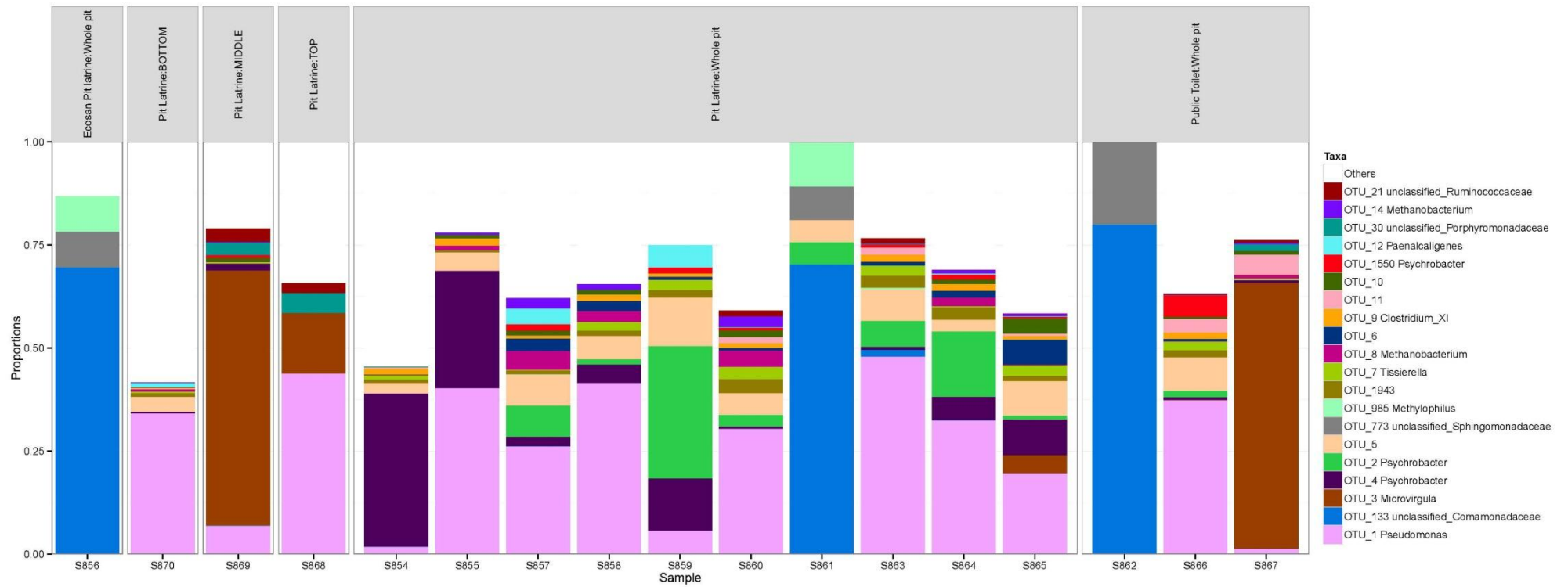
Figure 3.2 Comparison of the top 20 most abundant operational taxonomic units (OTU) in 11 pit latrines, 3 public toilets as well as the top, middle and bottom of one of the pit latrines sampled (S863).
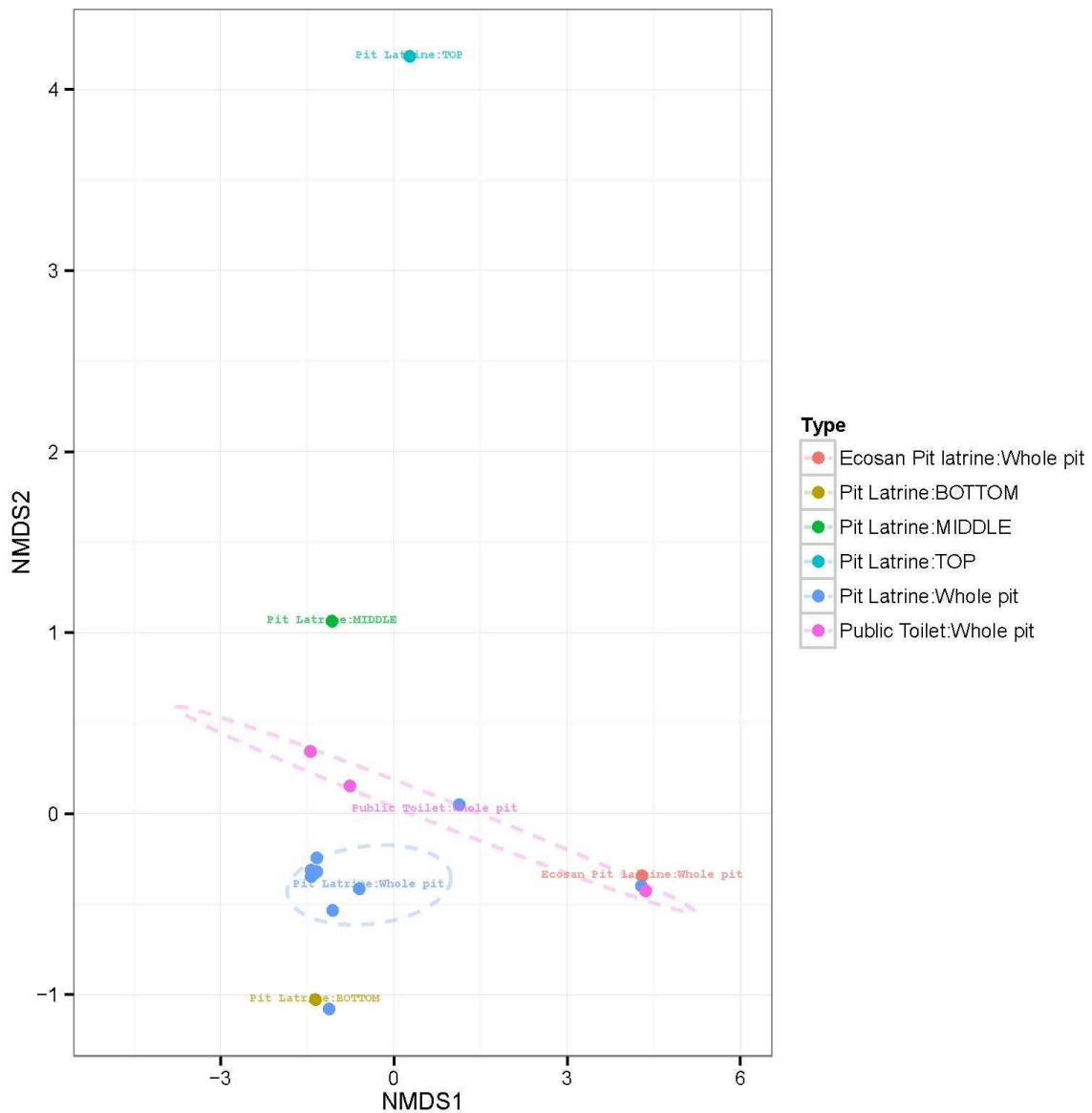
Figure 3.3 Weighted non-metric multidimensional scaling (NMDS) of representative samples from pit latrines and public toilets as well as the top, middle and bottom section of one of the pit latrines sampled. One pit latrine was of a different design and was labelled accordingly (ECOSAN). Points represent the original positions of communities in a multi-dimensional space and the relative sameness of OSS facilities. The greater the distance between samples the greater the difference in microbial community structure.
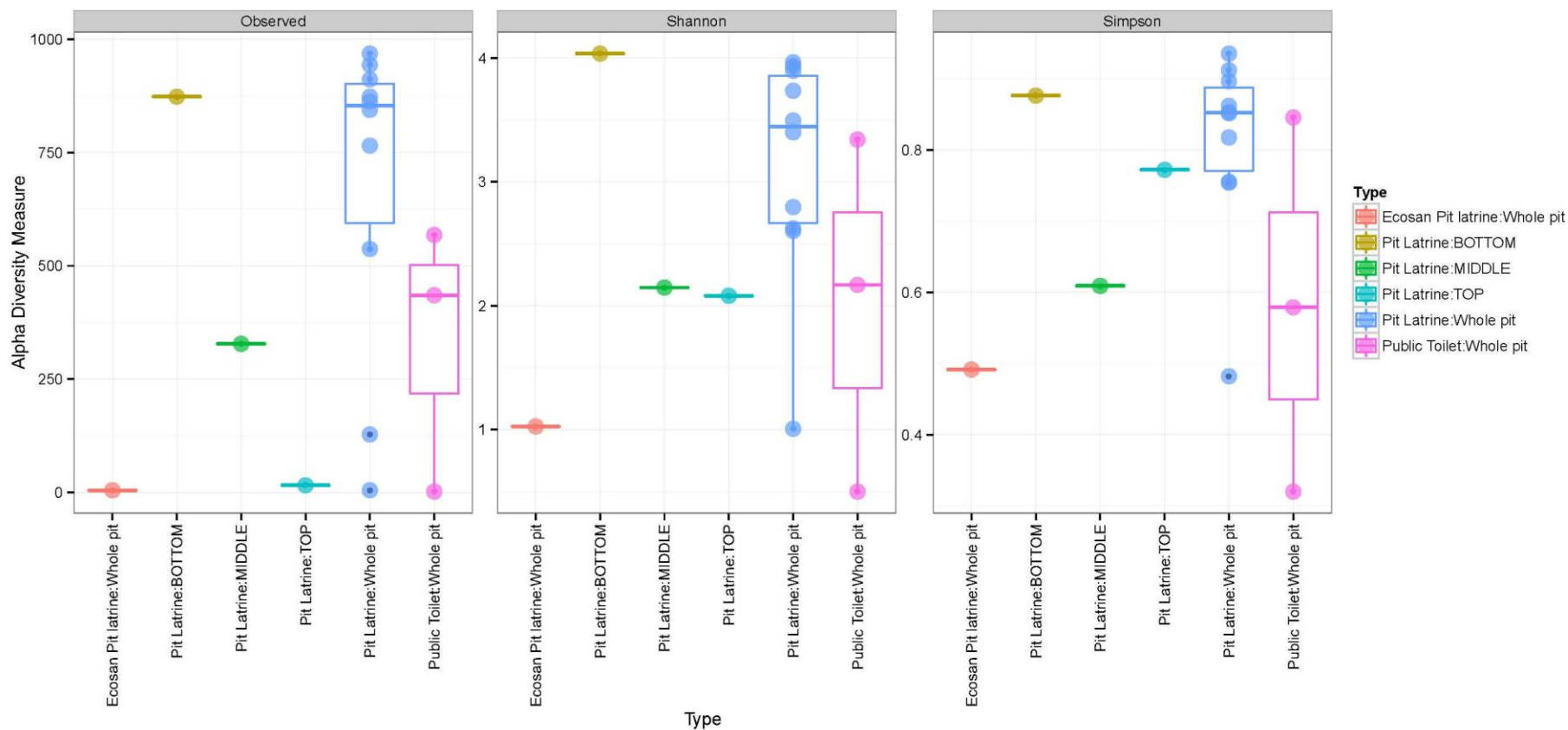
Figure 3.4 Alpha diversity measures illustrating the spread of OTUs within individual samples of pit latrines, public toilets and within the top, middle and bottom sections of one of the pit latrines sampled. One ECOSAN pit latrine is identified separately due to the difference in design and construction of this latrine type.

3.3.7 Collection and transportation of faecal sludge

Technology to enable the safe collection and transportation of FS from OSS facilities to treatment facilities is currently an extensive research area. Therefore, understanding how FS composition may impact collection technologies is important in addition to quantifying the volumes of FS being generated by populations in low income regions.

The high TS observed in pit latrine sludge (19.2% TS) is likely to impact collection technology of this FS type. Difficult access combined with large volumes of MSW (mean volume of MSW after screening of 5 pit latrines was 12% of the total sludge volume collected) will make the removal of FS using emptying devices that rely on suction, including conventional vacuum trucks and small scale vacuum pumps, challenging. This factor may promote the use of screw and auger systems (e.g. Rogers et al. (2014)) over suction systems for FS such as in *location x.*

The portable toilet accumulation rates and chemical composition were calculated on a per capita basis (Table 3.3). The volume of excreta collected in portable toilets (0.24 L/cap/day) was above mean literature values (0.14 L/cap/day) for faeces production (Meinzinger and Oldenburg, 2009). This could indicate that additional liquids (urine) are entering the toilets even though they are designed to be urine diversion toilets. Solids accumulation within portable toilets (28.21 g/cap/day) is consistent with literature values at between 12-81 g/cap/day (Rose et al., 2015), indicating that the sampling methodology and data collection methods were robust.

3.3.8 Treatability of faecal sludge in anaerobic processes

Anaerobic digestion has the potential to be used in low income countries to treat FS but a common constraint of assessing the suitability of AD in these localities is knowledge regarding the physical, biological and chemical characteristics of the feedstock.

The TS content of the feedstock impacts on the reactor volume and organic loading calculations. Total solids concentrations observed in pit latrine sludge (19% TS) are more suited to suspended growth reactors rather than up-flow or down-flow high rate processes (Hassan et al., 2013). This factor constitutes a major challenge to high-

rate AD treatment of FS and has led directly to research by Collins et al. (2013) into re-engineering AD biofilms and systems by using down-flow bioreactors employing combinations of trophic group-specific biofilms and materials to efficiently and rapidly digest high solids wastewater.

The TS of pit latrines is greater than the concentration that can be effectively mixed or pumped (Speece, 2008) which could make the use of higher rate systems more challenging and may make high solids AD (generally greater than 15% TS) more attractive and cost efficient. High solids AD (>15% TS) could bring advantages such as lower energy requirements, smaller reactor volumes, and less material handling (Guendouz et al., 2008) but with the disadvantages of lower CH_4 yields and a slower rate of treatment. On the other hand, portable toilet sludge (4.5% TS) and public toilet sludge (6.1% TS) had TS concentrations similar to that of sewage sludge from conventional treatment works such as primary sludge which is usually in the range of 2-8%TS (Tchobanoglous et al., 2003) and technology is already well adapted for these parameters.

Due to the manual methods of pit latrine emptying and the unlined nature of pit latrines there is also likely to be a large amount of sand/grit entering digesters which could cause hydraulic overloading of AD systems without an adequate grit screen. It could also contribute to a large sand/grit layer forming at the bottom of unmixed digesters that would have to be periodically removed in order to utilise the full digester volume. Due to the high MSW levels (12% of total volume of FS removed) found within pit latrines (and one public toilet holding tank) an extensive screening process prior to AD is essential.

The BMP of FS as measured by experimental BMP assays can also be compared to theoretical methane composition as determined by the stoichiometric equation based on the atomic composition of the FS by using the elements C, O, H and N (Table 3.3). Theoretical methane potential values, as calculated using Boyle's equation (Boyle, 1976), are greater than those recorded in experimental BMP assays in both pit latrines (199 and 49 mL CH_4/g VS_{added} respectively) and portable toilet samples (405 and 281 mL CH_4/g VS_{added} respectively). The higher theoretical values are likely to be due to substrate degradability not being accounted for which leads to higher

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theoretical methane production values (Labatut et al., 2011). In pit latrines the low BMP values demonstrate that there is a significant proportion of material that is not readily degradable and therefore designing systems using standard rates of VS destruction would lead to overestimation of methane yields. In pit latrines, as the bulk of methane production occurs with solids retention time (SRT) of less than 10 days (Figure 1), it would be difficult to justify a longer SRT for the purpose of increased CH_4 production. This is especially the case as the quantities of CH_4 are not sizeable in comparison to other FS types (49 mL CH_4/g VS_{added} and 281 mL CH_4/g VS_{added} in pit latrines and portable toilets respectively). A regular collection service such as portable toilets could provide an attractive opportunity for regular containment and substrate delivery to AD plants, however, small per capita methane production values calculated from BMP tests (0.00627 m^3 CH_4/person/day) illustrate that a large number of collections would be required.

Within pit latrines acetic acid (2057 mg L^{-1}) was the most significant VFA component (total 3736 mg VFA L^{-1}). High levels of acetate observed in FS are beneficial as acetate is the best substrate for methanogen bacteria, with the majority of methane produced in AD through the fermentation of acetate (acetoclastic cleavage) along with the reduction of carbon dioxide (Gerardi, 2003). The high acetic acid (55% of total VFA concentration in pit latrines) also indicates the hydrolysis stage of AD is complete, which is often the first and general rate limiting step in the digestion of particulate organic substrates (Zeeman and Sanders, 2001) and could resultantly reduce the need for separate hydrolysis/thermal hydrolysis steps before digestion.

3.3.8.1 Potential causes of toxicity to anaerobic digestion

Concentrations of NH_4-N in FS were observed to be very high in pit latrines (1853 mg NH_4-N L^{-1}) and public toilets (845 NH_4-N L^{-1}) and were still of significant concentrations in portable toilet sludge (396 NH_4-N L^{-1}). Ammonia (NH_3) can be a toxic component in the AD processes and can inhibit acetoclastic methanogens. Ammoniacal nitrogen ($NH_4 + NH_3$) is dependent on pH and temperature: at 30°C, NH_3 is approximately 5% of NH_4-N at pH 7.8 (Strauss et al., 2000). Using these assumptions NH_3 content of FS from pit latrines would give a concentration of 78 mg NH_3 L^{-1}. The toxicity threshold for ammonia has been reported to be 100 mg NH_3 L^{-1}

(McCarty and McKinney, 1961). Lay et al. (1998) also found a steady inhibition of methanogenic bacteria as the NH_4^+ concentration was increased from 50 to 500 mg NH_4^+ L^{-1}, with 500 mg NH_4-N L^{-1} the resultant toxicity level. However, all FS types exhibited relatively neutral pH values (pH 7.65, 7.21 and 6.43 in pit latrine, public toilet and portable toilet waste respectively) and consequently the majority of the ammoniacal nitrogen is in the ammonium-nitrogen form (NH_4^+). Therefore, the concentration of NH_3 in the FS of pit latrines is not likely to cause a reduction in enzyme activity or result in cell toxicity.

Heavy metal concentrations in pit latrines and public toilet sludge did not show high enough concentrations that could prove toxic to anaerobic bacteria (Table 3.3), although there could be the potential for the long term accumulation of metals as they are not biodegradable (Sterritt and Lester, 1980). Within portable toilet sludge high levels of Cu were found, likely due to the chemical toilet additive in use. Not only does this present potential toxicity to methanogen bacteria over long time periods but could also prove problematic if the digestate was applied to land following treatment. Concentrations of K, Ca and Mg in portable toilet waste (Table 3) were all below concentrations that could cause moderate inhibitory characteristics to AD.

The use of odour suppressant chemical toilet additives within portable toilets causes problems such as high Cu concentrations and the active ingredients glutaraldehyde and Bronopol were proven to cause CH_4 production inhibition (IC_{50} values ≥ 100 mg L^{-1} and ≥ 50 mg L^{-1} respectively). Although, it is likely reactors will become acclimatised to these chemicals to a certain extent and dilution will aid in reducing inhibitory concentrations. It nevertheless presents the question of toilet design and operation: if a toilet could be designed and operated in a way that uses less chemical odour suppressants this could lead to increased CH_4 yields and accelerated breakdown of organic material within digesters as well as prevent the build-up of heavy metals such as Cu.

3.4 CONCLUSIONS

This study undertook a detailed physical and chemical characterisation of four different types of FS and variation was found both within individual FS types and

across differing FS types. Public toilet and portable toilet FS has similar chemical characteristics to septage and primary sludge, which are regularly used as an AD feedstock. Pit latrine sludge could be more problematic to AD processes due to high TS and MSW content. However, if factors such as those discussed are taken into consideration in the design and operation of treatment plants, anaerobic digestion is an appropriate and viable treatment technology for all types of FS investigated.

3.5 REFERENCES

Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J., Guwy, A., Kalyuzhnyi, S., Jenicek, P. and Lier, J. v. (2009), "Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays", vol. 59, pp. 927-934 .

APHA (2005), *Standard Methods for the Examination of Water and Waste Water,* American Public Health Association, Baltimore, U.S.A.

Boyle, W. C. (1976), "Energy recovery from sanitary landfill, A review", in Schlegel, H. G. and Barnea, J. (eds.) *Microbial Energy Conversion,* 1st ed, Pergamon Press, Oxford, pp. 119-138.

Bryce, D., Croshaw, B., Hall, J., Holland, V. and Lessel, B. (1978), "The activity and safety of the antimicrobial agent bronopol (2-bromo-2-nitropropan-1, 3-diol)", *Journal of the Society of Cosmetic Chemists,* vol. 29, no. 1, pp. 3-24.

Buckley, C., Foxon, K. M., Broukaert, C. J., Rodda, N., Nwaneri, C., Balboni, E., Couderc, A. and Magagna, D. (2008), *Scientific support for the design and operation of Ventilated Improved Pit latrines (VIPs) and the efficacy of pit latrine additives,* WRC project no K5/1630, Water Research Commission, Durban, South Africa.

Chaggu, E. J. (2004), *Sustainable environmental protection using modified pit-latrines* (unpublished PhD thesis), Wageningen Universiteit, Wageningen, Holland.

Choi, E., Eum, Y., Gil, K. I. and Oa, S. W. (2004), "High strength nitrogen removal from nightsoil and piggery wastes", *Water Science and Technology,* vol. 49, no. 5-6, pp. 97-104.

Chynoweth, D. P., Turick, C. E., Owens, J. M., Jerger, D. E. and Peck, M. W. (1993), "Biochemical methane potential of biomass and waste feedstocks", *Biomass and Bioenergy,* vol. 5, no. 1, pp. 95-111.

Clean Team (2012)*, Clean Team Toilets*, available at: http://www.cleanteamtoilets.com/ (accessed August 2014).

Coetzee, M., Van der Merwe, M. and Badenhorst, J. (2011), "Effect of nitrogen loading rates on nitrogen removal by using a biological filter proposed for ventilated improved pit latrines", *International Journal of Environment Science and Technology,* vol. 8, no. 2, pp. 363-372.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd*

Water Research Conference, Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Czemiel, J. (2000), "Phosphorus and nitrogen in sanitary systems in Kalmar", *Urban Water,* vol. 2, no. 1, pp. 63-69.

Doku, I. A. (2003), *The potential for the use of upflow anaerobic sludge blanket (UASB) reactor for the treatment of faecal sludges in Ghana,* Assainissement A06, Ministère des Affaires Etrangères, Training Research and Networking for Development Group, Kumasi, Ghana.

Drechsel, P., Scott, C. A., Raschid-Sally, L., Redwood, M. and Bahri, A. (eds.) (2009), *Wastewater Irrigation and Health: Assessing and Mitigating Risk in Low Income Countries,* 1st ed, Earthscan/IWMI/IDRC, London.

Elbeshbishy, E., Nakhla, G. and Hafez, H. (2012), "Biochemical methane potential (BMP) of food waste and primary sludge: Influence of inoculum pre-incubation and inoculum source", *Bioresource technology,* vol. 110, no. 0, pp. 18-25.

Fenner, R. A., Guthrie, P. M. and Piano, E. (2007), "Process selection for sanitation systems and wastewater treatment in refugee camps during disaster relief situations", *Water and Environment Journal,* vol. 21, no. 4, pp. 252-264.

Fernandez, A. S., Hashsham, S. A., Dollhopf, S. L., Raskin, L., Glagoleva, O., Dazzo, F. B., Hickey, R. F., Criddle, C. S. and Tiedje, J. M. (2000), "Flexible community structure correlates with stable community function in methanogenic bioreactor communities perturbed by glucose", *Applied and Environmental Microbiology,* vol. 66, no. 9, pp. 4058-4067.

Foxon, K., Mkhize, S., Reddy, M. and Buckley, C. (2009), "Laboratory protocols for testing the efficiency of commercial pit latrine additives", *Water SA,* vol. 35, no. 2, pp. 228-235.

Gajurel, D. R., Li, Z. and Otterpohl, R. (2003), "Investigation of the effectiveness of source control sanitation concepts including pre-treatment with Rottebehaelter", *Water Science and Technology,* vol. 48, no. 1, pp. 111-118.

Gerardi, M. H. (2003), *The microbiology of anaerobic digesters,* 1st ed, John Wiley & Sons, Hoboken, USA.

Gonzalez Gil, G., Seghezzo, L., Lettinga, G. and Kleerebezem, R. (2001), "Kinetics and mass-transfer phenomena in anaerobic granular sludge", *Biotechnology and Bioengineering,* vol. 73, no. 2, pp. 125-134.

Graham, J. P. and Polizzotto, M. L. (2013), "Pit Latrines and Their Impacts on Groundwater Quality: A Systematic Review", *Environmental Health Perspectives,* vol. 121, no. 5, pp. 521.

Guendouz, J., Buffiere, P., Cacho, J., Carrere, M. and Delgenes, J. (2008), "High-solids anaerobic digestion: comparison of three pilot scales", *Water Science and Technology,* vol. 58, no. 9, pp. 1757-1763.

Hassan, S. R., Zwain, H. M. and Dahlan, I. (2013), "Development of Anaerobic Reactor for Industrial Wastewater Treatment: An Overview, Present Stage and Future Prospects.", *Journal of Advanced Scientific Research,* vol. 4, no. 1.

Hawkins, P. M. (1982), *Emptying on site excreta disposal systems in developing countries: An evaluation of the problems,* 17, IRCWD, Geneva, Switzerland.

He, Z., Kan, J., Wang, Y., Huang, Y., Mansfeld, F. and Nealson, K. H. (2009), "Electricity production coupled to ammonium in a microbial fuel cell", *Environmental science & technology,* vol. 43, no. 9, pp. 3391-3397.

Heinss, U., Larmie, S. A. and Strauss, M. (1998), *Solids separation and pond systems for the treatment of faecal sludges in the tropics: lessons learnt and recommendations for preliminary design,* 05/98, EAWAG/SANDEC, Dubendorf, Switzerland.

Heinss, U., Larmie, S. A. and Strauss, M. (1999), *Characteristics of faecal sludges and their solids-liquid separation,* 1, EAWAG/SANDEC, Zurich, Switzerland.

Henze, M., Harremoës, P., la Cour Jansen, J. and Arvin, E. (2001), *Wastewater treatment: biological and chemical processes,* 3rd ed, Springer, Berlin, Germany.

Issah, A. (2011), *Comparative study of the effluent from anerobic digesters for human and fruit waste* (unpublished MSc thesis), Kwame Nkrumah University of Science and Technology, College of Engineering.

Issah, A., Aklaku, E. D. and Salifu, T. (2012), "Comparative study of effluent for pollutionindicators and indicator pathogenic organisms from anerobic digesters from human and fruit wastes", *ARPN Journal of Agricultural and Biological Science,* vol. 6, no. 6, pp. 416-420.

Kiely, G. (1997), *Environmental engineering,* 2nd ed, McGraw-Hill, London.

Koné, D. and Strauss, M. (2004), "Low-cost options for treating faecal sludges (FS) in developing countries–Challenges and performance", *9th International IWA Specialist Group Conference on Wetlands Systems for Water Pollution Control and to the 6th International IWA Specialist Group Conference on Waste Stabilisation Ponds,* Vol. 27, 27th Sept. - 1st Oct.., Avignon, France, IWA, London, UK, .

Koottatep, T., Surinkul, N., Paochaiyangyuen, R., Suebsao, W., Sherpa, M., Liangwannaphorn, C., Panuwatvanich, A. (2012), *Assessment of Faecal Sludge Rheological Properties - Final report,* , Environmental Engineering Program, School of Environment, Resources and Development Asian Institute of Technology, Thailand.

Labatut, R. A., Angenent, L. T. and Scott, N. R. (2011), "Biochemical methane potential and biodegradability of complex organic substrates", *Bioresource technology,* vol. 102, no. 3, pp. 2255-2264.

Leung, H. (2001), "Ecotoxicology of glutaraldehyde: review of environmental fate and effects studies", *Ecotoxicology and Environmental Safety,* vol. 49, no. 1, pp. 26-39.

Lim, J. W. (2011), "Anaerobic Co-digestion of Brown Water and Food Waste for Energy Recovery", *11th edition of the World Wide Workshop for Young Environmental Scientists (WWW-YES-2011)-Urban Waters: resource or risks?* 5th-10th June 2011, Accueil, France, UPEC/NPEC, France, pp. 1.

Meinzinger, F. and Oldenburg, M. (2009), "Characteristics of source-separated household wastewater flows: a statistical assessment", *Water Science and Technology,* vol. 59, no. 9, pp. 1785-1791.

Muspratt, A. M., Nakato, T., Niwagaba, C., Dione, H., Kang, J., Stupin, L., Regulinski, J., Mbéguéré, M. and Strande, L. (2014), "Fuel potential of faecal sludge: calorific value results from Uganda, Ghana and Senegal", *Journal of Water, Sanitation and Hygiene for Development,* vol. 0.4.2, no. doi: 10.2166/washdev.2013.055, pp. 223-230.

Norris, G. A. (2000), *Sludge build-up in septic tanks, biological digesters and pit latrines in South Africa,* 544/1/00, Water Research Commision, South Africa.

Otani, Y., Hasegawa, K. and Hanaki, K. (2004), "Comparison of aerobic denitrifying activity among three cultural species with various carbon sources", *Water Science and Technology,* vol. 50, no. 8, pp. 15-22.

Owens, J. and Chynoweth, D. (1993), "Biochemical methane potential of municipal solid waste (MSW) components", *Water Science and Technology,* vol. 27, no. 2, pp. 1-14.

Panuvatvanich, A., Koottatep, T. and Kone, D. (2009), "Influence of sand layer depth and percolate impounding regime on nitrogen transformation in vertical-flow constructed wetlands treating faecal sludge", *Water Research,* vol. 43, no. 10, pp. 2623-2630.

Parawira, W., Murto, M., Read, J. S. and Mattiasson, B. (2004), "Volatile fatty acid production during anaerobic mesophilic digestion of solid potato waste", *Journal of Chemical Technology and Biotechnology,* vol. 79, no. 7, pp. 673-677.

Patureau, D., Helloin, E., Rustrian, E., Bouchez, T., Delgenes, J. and Moletta, R. (2001), "Combined phosphate and nitrogen removal in a sequencing batch reactor using the aerobic denitrifier, Microvirgula aerodenitrificans", *Water research,* vol. 35, no. 1, pp. 189-197.

Patureau, D., Zumstein, E., Delgenes, J. and Moletta, R. (2000), "Aerobic denitrifiers isolated from diverse natural and managed ecosystems", *Microbial ecology,* vol. 39, no. 2, pp. 145-152.

Pradt, L. A. (1971), "Some recent developments in night soil treatment", *Water Research,* vol. 5, no. 8, pp. 507-521.

Rajagopal, R., Lim, J. W., Mao, Y., Chen, C. and Wang, J. (2013), "Anaerobic co-digestion of source segregated brown water (feces-without-urine) and food waste: For Singapore context", *Science of the Total Environment,* vol. 443, pp. 877-886.

Rogers, T. W., Francis III, L., Beckwith, W. J. and Borden, R. C. (2014), "Power earth auger modification for waste extraction from pit latrines", *Journal of Water, Sanitation and Hygiene for Development,* vol. 4, no. 1, pp. 72-80.

Rose, C., Parker, A., Jefferson, B. and Cartmell, E. (2015), "The Characterisation of faeces and urine; a review of the literature to inform advanced treatment technology", *Critical Reviews in Environmental Science and Technology,* vol. 45, no. 17, pp. 1827-1879.

Salisbury, R. H., Lorentz, S., Buckley, C. A., Bhagwan, J. and Still, D. (2009), "Finding a sustainable solution to the overflowing pit problem: The role of SRF in the beneficiation of VIP sludge", *Water, sanitation and hygiene. Sustainable development and multisectoral approaches. Preceedings of the 34th WEDC International conference,* Vol. 1, 18th-22nd May 2009, Addis Ababa, Ethiopia, 34th WEDC International Conference, WEDC, Loughborough, UK, pp. 1.

Schouw, N. L., Danteravanich, S., Mosbaek, H. and Tjell, J. C. (2002), "Composition of human excreta — a case study from Southern Thailand", *Science of The Total Environment,* vol. 286, no. 1–3, pp. 155-166.

Speece, R. E. (2008), *Anaerobic biotechnology and odor/corrosion control for municipalities and industries,* 1st ed, Archae Press, Tennessee, USA.

Sterritt, R. and Lester, J. (1980), "Interactions of heavy metals with bacteria", *Science of the Total Environment,* vol. 14, no. 1, pp. 5-17.

Stover, C. K., Pham, X. Q., Erwin, A., Mizoguchi, S., Warrener, P., Hickey, M., Brinkman, F., Hufnagle, W., Kowalik, D. and Lagrou, M. (2000), "Complete genome sequence of Pseudomonas aeruginosa PAO1, an opportunistic pathogen", *Nature,* vol. 406, no. 6799, pp. 959-964.

Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) (2014), *Faecal Sludge Management: Systems Approach for Implementation and Operation,* 1st ed, IWA Publishing, London, UK.

Strauss, M. (1985), *Health aspects of nightsoil and sludge use in agriculture and aquaculture. Part II: pathogen survival,* 04/85, International Reference Centre of Waste Disposal, Duebendorf, Switzerland.

Strauss, M., Larmie, S. A., Heinss, U. and Montangero, A. (2000), "Treating faecal sludges in ponds", *Water Science and Technology,* vol. 42, no. 10, pp. 283-290.

Takahashi, Y., Wydeven, T. and Koo, C. (1989), "Subcritical and supercritical water oxidation of CELSS model wastes", *Advances in Space Research,* vol. 9, no. 8, pp. 99-110.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Zeeman, G. and Sanders, W. (2001), "Potential of anaerobic digestion of complex waste (water)", *Water Science & Technology,* vol. 44, no. 8, pp. 115-122.

CHAPTER 4

THE BIOCHEMICAL METHANE POTENTIAL OF FAECAL SLUDGE FROM DIFFERENT ON-SITE SANITATION FACILITIES

4 THE BIOCHEMICAL METHANE POTENTIAL OF FAECAL SLUDGE FROM DIFFERENT ON-SITE SANITATION FACILITIES

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ABSTRACT

On-site sanitation facilities comprise the majority of sanitation provisions in urban areas of low income countries. A common characteristic of on-site sanitation facilities is that residual sludge will eventually require further treatment before disposal or re-use. Anaerobic digestion is the most common form of sludge treatment in high income countries and is increasingly common in low income countries. In order to effectively design, plan and operate anaerobic digestion facilities it is beneficial to estimate the methane yields and biodegradation potential expected from differing substrates. The biochemical methane potential of faecal sludge from pit latrines, public toilets and portable toilets was determined from two peri-urban areas, in *location x* and Ghana. Sewage sludge from a wastewater treatment works in a high income country (UK) was also compared. The greatest methane potential was from portable toilet waste (276.0 ± 151.3 mL/g VS_{added}) with pit latrine sludge exhibiting

lower methane potential values (50.6 ± 19.4 mL/g VS$_{added}$) indicating that much of the easily biodegradable organics have already been digested. All faecal sludge types had methane potential values below that of primary sewage sludge (358.4 mL/g VS$_{added}$) and minimal difference between the top and bottom layer of pit latrine vaults (136 and 130 mL/g VS$_{added}$ respectively) was exhibited. The results indicate that the retention time of the on-site sanitation facility is the greatest influence of methane potential values and should be considered in the provision of on-site sanitation facilities and in the treatment of faecal sludge.

4.1 INTRODUCTION

It is estimated that 2.7 billion people worldwide are served by sanitation facilities that require faecal sludge management (Strande et al., 2014). In Sub-Saharan Africa, 80% of urban sanitation provision is met through on-site sanitation (OSS) facilities (Kone, 2010) in which residual solids, commonly referred to as "faecal sludge" (FS) are accumulated. Effective faecal sludge management can provide sustainable sanitation provision as long as there is safe storage, collection, treatment and disposal/reuse of FS by a means that prevents the spread of pathogens and parasites in the environment (Kone et al., 2007). However, in low income countries rapid urbanisation is causing substantial challenges for sanitation infrastructure such as the operation and maintenance of wastewater and faecal sludge treatment plants (Cofie et al., 2009; Rydin et al., 2012). In addition, the safe collection and treatment of FS is not guaranteed and is frequently discarded into water bodies or fields in close proximity to urban areas (Lalander et al., 2013). Resultantly, there is a strong driver for increased and improved FS treatment at both a centralised and decentralised level in peri-urban areas of low income countries.

Anaerobic digestion (AD) is one of the most common means of treatment for primary sewage sludge (following primary settlement in wastewater treatment works) in the

European Union (Fytili and Zabaniotou, 2008). The AD of high-solids wastewater, such as FS, has been identified as a research area that would allow innovative solutions to FS treatment in peri-urban localities (Collins et al., 2013); where water stress and energy constraints challenge conventional wastewater treatment systems. Anaerobic digestion is a multiple stage biological process that breaks down organic compounds and results in the production of methane and a semi-solid digestate that is rich in valuable nutrients. Both of these by-products can in turn help to offset the prior costs of collection and treatment and become a successful new value proposition for FS management (Diener et al., 2014). As a FS treatment mechanism, AD provides flexibility at a range of scales: with widespread household/community application in rural areas of India, Nepal and China where animal manure is frequently combined with FS (Gautam et al., 2009; Bond and Templeton, 2011; Chen et al., 2012; Song et al., 2014). However, great potential also exists for expansion of semi-centralised or centralised systems for FS treatment in peri-urban areas of large low income cities (Collins et al., 2013; Strande et al., 2014) where waste water treatment works (WwTW) are often either vastly overloaded or non-existent (Hounkpe et al., 2014).

The design and operation of anaerobic treatment facilities for FS has previously been reliant on limited chemical and physical FS characterisation data from similar OSS facilities. For instance, commonly reported FS characterisation data for pit latrines: such as COD (range of 10400-97000 mg COD L^{-1}) and BOD (range of 3800-1500 mg BOD L^{-1}) (Heinss et al., 1999; Doku, 2003; Coetzee et al., 2011). However, through the use of stoichiometric methods CH_4 production is directly related to degradation of organics (395 mL CH_4 equals 1 g COD reduction (Speece, 1996)),which will overestimate AD performance as the entire COD fraction will not be readily biodegradable. Similarly, BOD values will be misleading as they do not exclude aerobic non-biodegradable components that the BMP includes. Consequently, the direct measurement of the methane potential of faecal sludge is a requirement.

Anaerobic digestion is impacted by feedstock composition (Drosg et al., 2013). The direct comparison of biogas production from different feedstock is difficult as the performance of different digesters will be dependent on individual experimental

conditions such as temperature, mixing, pH, hydraulic retention time, solids and organic loading rates (Ward et al., 2008). Consequently, the comparison of feedstock is best determined through the use of biochemical methane potential (BMP) assays (Owens and Chynoweth, 1993), through which the ultimate yields of methane and the digestibility of differing feedstock can be determined. Biochemical methane potential assays determine the concentration of organics that can be anaerobically converted to CH_4 and allow comparison across other feedstock types, in addition, the rate at which CH_4 is produced with the proposed feedstock can also be assessed.

On-site sanitation facilities (OSS) within peri-urban areas of low income countries range vastly in regards to their design and operation (Tilley et al., 2008b), however, in regards to biogas production can be broadly grouped by the retention time of the systems. In order to obtain several different types of FS substrates from OSS facilities with a range in retention times; pit latrines, unsewered public toilets and portable toilets were investigated along with a primary sludge taken from a waste water treatment works (WwTW) in a high income country. Pit latrines are the most commonly used form of on-site sanitation (OSS) system in developing countries with an estimated 1.77 billion people around the world using some form of pit latrine (Graham and Polizzotto, 2013). Public toilet and community ablution blocks where toilet and grey-water waste is collected in a sealed holding tank and emptied on a regular basis (*ca.*3-6 months) are also commonly used in peri-urban areas (Drechsel et al., 2009). Finally, a new concept of OSS services is the provision of portable container based toilets to households which are then collected on a regular basis (Clean Team, 2012; Tilmans et al., 2015).

Differing retention times and storage conditions between OSS facilities is a factor that is hypothesised to cause variability in BMP values of FS. Pit latrine sludge is stored within pit latrine vaults for long periods of time (ranging from 1-10 years), with the gradual addition of faeces and urine to the surface of the vault. However, unsewered public toilets were typically emptied every 3-6 months. In contrast to these systems, portable toilets are operated with a high collection frequency (<4 days), after which the waste is emptied into an Intermediate Bulk Container (IBC) and stored (approximately 1 month) prior to collection and treatment. It was therefore

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also hypothesised that differences in BMP values could be apparent due to depth and position within the OSS system, with slower anaerobic processes operating in pit latrines and the likely settlement or flotation of particles within IBC tanks storing portable toilet waste.

This study presents a detailed assessment of the biochemical methane potential of FS and its potential as an AD feedstock in low income countries. An in depth study of BMP values of a range of FS types is explored and provides essential information in order to make the successful design and operation of AD systems treating FS possible in low income areas in need of effective faecal sludge management. The BMP of three types of faecal sludge from a range of OSS facilities used in peri-urban locations in low income countries are presented and compared to a baseline of sewage sludge from a WwTW in a high income country. Specifically, the objectives are to determine a) the ultimate methane potential of different substrates; b) the impact of sampling depth in pit latrine vaults on BMP values; and c) the impact of sampling by depth on BMP values within an intermediate storage tank for portable toilet waste.

4.2 METHODS

4.2.1 On site sanitation facilities and sampling location

In order to get a broad range of differing faecal sludge types, three different OSS facilities were selected to be investigated: FS from pit latrines, unsewered community ablution blocks and portable toilets. In order to acquire this range of FS types, two peri-urban field locations were selected: *Location x* and Kumasi, Ghana. In addition to the three different FS types selected; a primary sludge from a WwTW in the UK was investigated in order to act as a direct comparison and benchmark to conventional sewage sludge treatment in high income countries.

The peri-urban area of *location x* was selected as a site for the collection of faecal sludge samples from pit latrines and un-sewered public toilets. Pit latrines in the area were of a simple construction raised above ground level (0.5-2m) to prevent seasonal flooding, with no vent pipe present and the vaults of the latrines were partially lined. Manual collection methods were used to empty FS from pit latrine

vaults through the use of a range of modified equipment such as pitchforks and shovels; these methods allowed the collection of FS samples at varying depths. The disposal of Municipal Solid Waste (MSW) within the latrines was common practice in the area; large items (>100 mm diameter) were removed and not included in further analysis. Faecal sludge sampling from unsewered public toilets was carried out in the same peri-urban region of *location x*. Community ablution blocks consisted of between 4-12 flush toilets, urinals as well as wash-water from washing and showering. The collection tanks consisted of two fully lined concrete tanks connected in series, allowing partial settlement of solids and tanks were sunk below ground level. Manual collection methods were again used and both tanks were emptied simultaneously.

A pilot scheme in Kumasi, Ghana testing the implementation of portable toilets was selected as a site for portable toilet sludge sampling (Clean Team, 2012). Portable toilets consisted of household units comprising a detachable container in which faeces collected over time in a mixture of a commercially available chemical toilet additive (Active ingredients: Pentane 1,5 diol 5-10%, 2-Bromo-2-Nitropropane-1,3-diol <5%) diluted in between 5 and 7 L of water in order to prevent odour issues. Toilets were designed to be urine diverting and hence only the faeces were collected. Toilets were placed in customer households (average number of users 4.2) where they were regularly collected at set time intervals (<4 day intervals) and containers were subsequently emptied into an IBC for storage before final treatment and discharge.

A small WwTW (p.e. 3000 people) was selected in the UK for the collection of undigested sewage sludge following primary settlement; this was classified as primary sludge. This site was selected due to logistical ease and was assumed to be representative of primary sewage sludge from similar WwTW in the UK. Primary sludge was collected from the surface of the primary settlement tank at the head of the sewage works.

Institutional ethical approval (*Cranfield University 109-2013)* was obtained for the sampling and analysis of faecal sludge in both case study locations: *Location x* and Kumasi, Ghana.

4.2.2 Sampling description and methodology

Samples were collected from a total of 11 pit latrines, 2 unsewered public toilets and 10 portable toilets. Sampling according to depth was carried out at one pit latrine site and within the holding tank IBC for portable toilet sludge. Representative samples in pit latrines and unsewered public toilets were determined by taking a sub sample (0.5 L) from the centre of each 60 L barrel of sludge removed from each facility (range of 12-64 barrels removed). These sub samples were then combined to produce one composite sample (6-18 L), of which a sample of 0.5 L was taken. In order to assess the impact of sampling at different depths a sample from the top layer (0.15 m depth) was taken and a sample from the bottom layer (1 m depth) was taken. Representative samples for portable toilets were obtained by firstly manually mixing each individual toilet for 5 minutes to homogenise the contents, following this, three samples of 0.5 L were taken from the top, middle and bottom of the container by using a modified sample jar and drain rod configuration. These samples were combined (1.5 L) where the contents were once again manually mixed before a final sample was taken from this container. The holding tank, an Intermediate Bulk Container (IBC), containing FS waste from portable toilets was sampled with depth (0.3 m intervals) using the same methods allowing a vertical profile of the IBC to be constructed. Primary sludge was collected from the surface of a primary settlement tank. A sample of 5L was taken from the surface of the tank and homogenised for 5 minutes before a sub sample of 0.5 L was subsequently taken.

All samples were sealed in plastic sample bags and placed in a cool box (approximate journey time: 1 hour) prior to being frozen (-20°C). Frozen samples were subsequently transported back to a UK laboratory in a cool box packed with ice and samples were <4°C on arrival. All samples were subsequently stored at -20°C (< 3 months) before BMP assays were conducted in one laboratory utilising the same experimental set up and operational conditions. The freezing of samples was necessary in order to avoid microbial transformation and collate a large number of samples in order to ensure that BMP methodologies were conducted by consistent and robust methodologies. Triolo et al. (Triolo et al., 2014) illustrated that the freeze/thawing of samples before BMP assays was not too disruptive, with the

relative standard deviation between the BMP of controls and freeze-thawed samples in the range of 2.1-9%.

4.2.3 Analytical methods and calculations

Biochemical methane potential (BMP) assays were used to determine the potential methane yield of the sludge under anaerobic conditions, and were carried out according to Owens and Chynoweth (Chynoweth et al., 1993) and Angelidaki et al. (Angelidaki et al., 2009) (Figure 4.1). An anaerobic inoculum was taken from a mesophilic digester at a UK waste water treatment works (population equivalent of 288000) and a stock solution of micronutrients (according to Gonzalez-Gil et al. (Gonzalez Gil et al., 2001)) was added to ensure sufficient quantities of trace metals were available. An inoculum: substrate ratio of 2:1 on the basis of VS was used for all assays, exhibited to give maximum conversion rates by Chynoweth et al. (1993). A positive control containing inoculum and cellulose was used in order to provide an indication of the response of the inoculum to a standard material. All blanks, standards and feedstock assays were carried out in quadruplicates and the average value reported with standard deviation. Each assay was flushed with N_2/CO_2 (80:20% as volume) after transfer of inoculum and substrate before being anaerobically incubated for 32 days in a temperature controlled shaker (Excella E24, New Brunswick Scientific, Edison, USA) at 37.5°C±0.5: 150 rev. min^{-1}. In order to account for residual degradable matter in the inoculum, blanks containing only inoculum were run to account for gas production not attributed to the FS substrate being tested. The volume of biogas was determined using the water displacement method. A gas analyser (Servomex 1440, Zoetermere, Netherlands) was used to determine % methane concentration and was carried out at the same frequency as gas volume measurements. The total biogas production was calculated at standard temperature and atmospheric pressure (STP) was expressed in mL. Total solids (TS) and volatile solids (VS) were measured according to standard methods (APHA, 2005) and pH was measured using a portable YSI G3 60 (YSI Ltd., Hampshire, UK).
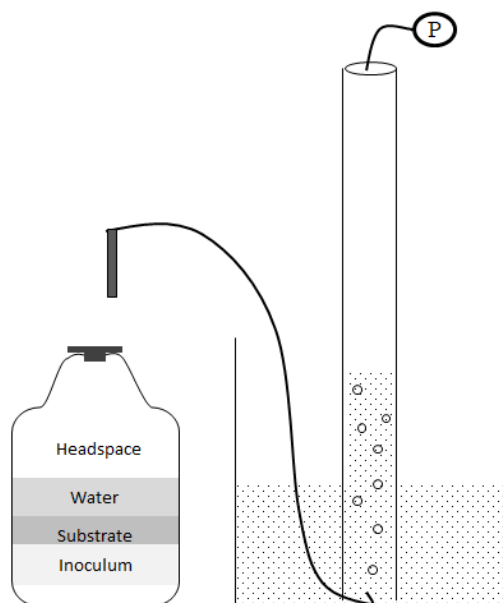
Figure 4.1 Biochemical methane potential assay containing an inoculum from a mesophilic digester treating sewage sludge, a nutrient stock solution, the faecal sludge substrate under analysis and deionised water for dilution. Biogas measurement was undertaken through water displacement after a assay bottles were de-gassed.

4.3 RESULTS AND DISCUSSION

4.3.1 Characterisation of substrates used in experiments

The BMP assay is a realistic way of measuring anaerobic biodegradability within substrates (Speece, 2008) and for each differing FS type shows both the ultimate CH_4 yield and provides an indication as to the amount of stabilisation occurring under differing storage conditions. The inoculum used had a pH of 8.52, a solids concentration of 44.15 g TS L^{-1} and 31.29 g VS L^{-1}. The characteristics of each faecal sludge type are outlined in Table 4.1. There was variation in organic composition across FS types with pit latrines showing on average the largest VS concentration (91.92 g VS L^{-1}), although this was predominantly due to the high TS content of pit latrines (19.2% TS). In contrast portable toilets had the lowest TS concentration (43.81 g TS L^{-1}) but with a comparatively high VS concentration in comparison to pit latrines (74.42% VS (8.60 Std Dev.) and 47.76% (Std Dev. 9.63) VS as a % of TS respectively). Primary sludge proved a sensible comparison to the

different FS types with TS and VS values within the range of the other sludge types (Figure 4.2).

Table 4.1 Characterisation of three types of faecal sludge used as substrate; pit latrines and public toilets in *location x* as well as from portable toilets in Kumasi, Ghana. In addition the composition of a primary sludge from a waste water treatment works in the UK. Average values of each classification presented (Standard Deviation in brackets).

	Pit latrine (location x)	Public toilets (location x)	Portable toilets (Ghana)	Primary sludge (UK)
TS (g L^{-1})	192.45 (54.31)	60.97 (20.81)	43.81 (12.71)	54.52 (0.54)
VS (g L^{-1})	91.92 (17.43)	36.97 (15.32)	33.04 (11.31)	48.96 (0.50)
pH	7.65	7.22	6.43	6.24
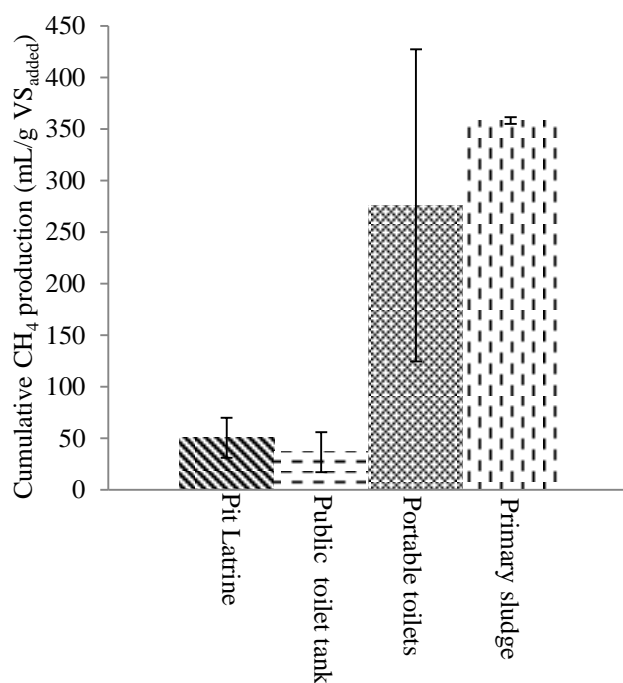
Figure 4.2 Average (with standard error) biochemical methane potential of primary sludge (n=1), portable toilet waste (n=10), unsewered public toilets (n=2) and pit latrines (n=11) after 32 days.Methane Production from Faecal Sludge Substrates

The BMP assays show the ultimate methane yield of sludge from pit latrines, unsewered public toilets and portable toilets (Figure 4.2). A wide variation both within and across FS types was observed in the BMP results. Sludge from portable toilets on average showed the highest methane production values (276.0 ± 151.3mL/g VS_{added}) and pit latrines the lowest methane potential (50.6 ± 19.4mL/g VS_{added}) at standard temperature and pressure. As would be expected, the range of values for pit latrines was much narrower (12.2 to 72.8 mL/g VS_{added}) than that of portable toilets (97.1 to 603.5 mL/g VS_{added}): this is likely to be due to the large amount of individual variation that would be expected between household portable toilet samples, this could be due to variations in the total number of users as well as their diet. The greater range in portable toilet waste could also be due to varying amounts of chemical additive in use, which at high concentrations could inhibit methane production through the suppression of bacteria (Leung, 2001; Bryce et al., 1978). Chemical toilet additives present at high concentrations were also hypothesised to have caused complete inhibition of methane production in three samples which were consequently excluded from experimental results. The toxicity of the active ingredients in the chemical toilet additive was consequently investigated in full in Chapter 3.

Biochemical methane potential tests carried out on a faeces only waste stream by Rajagopal (2013) gave similar CH_4 production values (260-300mL CH_4/g VS_{added}) and Lim et al. (2011) reported slightly higher cumulative CH_4 production values of 535-672 L/kg VS_{added} to that of cumulative CH_4 values seen in portable toilets in this study (280.5 mL/kg VS_{added}), although a wider range (97.2-603.5 mL/kg VS_{added}) was seen within this study. The low cumulative methane production values of pit latrines (Figure 4.3) show consistency with findings by Nwaneri *et al.* (2008) who found that methane production from pit latrine sludge was below detection levels in their study and in many cases caused inhibition to assays. Although no complete inhibition was

found within pit latrine FS in this study there were very low CH_4 production levels (50.6 mL/g VS_{added}) in comparison to both IBC storage of portable toilet sludge (211.6 mL/g VS_{added}) as well as to primary sludge from a WwTW as measured in this study (358.4 mL/g VS_{added}). The easily degradable fraction has already been lost within the pit latrines due to the much greater period of storage (between 2-10 years) in comparison to portable toilet waste which was collected at more regular intervals (< 4 days). The low methane production potential of pit latrine sludge could also be accounted for by other factors and not just a reflection of its greater age. High concentrations of nitrogen (N) were present in pit latrine sludge (average: 4561 mg N L^{-1}) with average values of ammonium nitrogen of 1853 mg NH_4-N L^{-1}. These high concentrations of N are similar to those reported by Coetzee *et al.* (2011) (3000-5000 mg N L^{-1}) in pit latrine FS. Due to the anaerobic nature of pit latrines the majority of N in FS of pit latrines will be in the inorganic form of ammonium-nitrogen (NH_4+NH_3). Therefore, due to these high ammonia (NH_3) concentrations there is a strong possibility of NH_3 toxicity occurring within the assays and could be a reason for the low BMP values seen in the FS of pit latrines in this study. It should be noted however that in practice the impact of NH_3 toxicity will reduce with time as bacterial communities within reactors become acclimatised to higher NH_3 concentrations (Speece, 1996).

Primary sludge from conventional wastewater treatment works was predicted to be the most comparable to that of FS from OSS facilities, due to its high solids (54.52 g TS L^{-1}) and undigested nature. All FS types exhibited lower cumulative CH_4 production values to that of primary sludge from a WwTW in the UK (358.4 mL CH_4/g VS_{added}), although only one WwTW was sampled in this study. Nevertheless, BMP values for primary sludge are numerous in the literature: values in this study are lower than that reported by Chenynoweth *et al.* (1993) who reported values of 590 mL CH_4/g VS_{added} but higher than the value of 230 mL CH_4/g VS_{added} reported by Elbeshbishy *et al.* (2012). The BMP values for primary sludge are therefore within a similar range to these studies indicating that the experimental methods were robust and further validating the results of FS substrates recorded. The BMP of primary sludge was the highest recorded of all FS types (Figure 4.2). This factor is not surprising as the skimming's from primary settlement tanks are generally high in

grease content (13-65% (Speece, 2008)), which results in significant CH_4 production due to the much greater extent to which grease components can be biodegraded. However, it is unlikely that OSS substrates contained great concentrations of fats and greases, as these predominantly come from being discharged to sewers from kitchens (Williams et al., 2012), which explains the lower BMP values observed in these substrates.
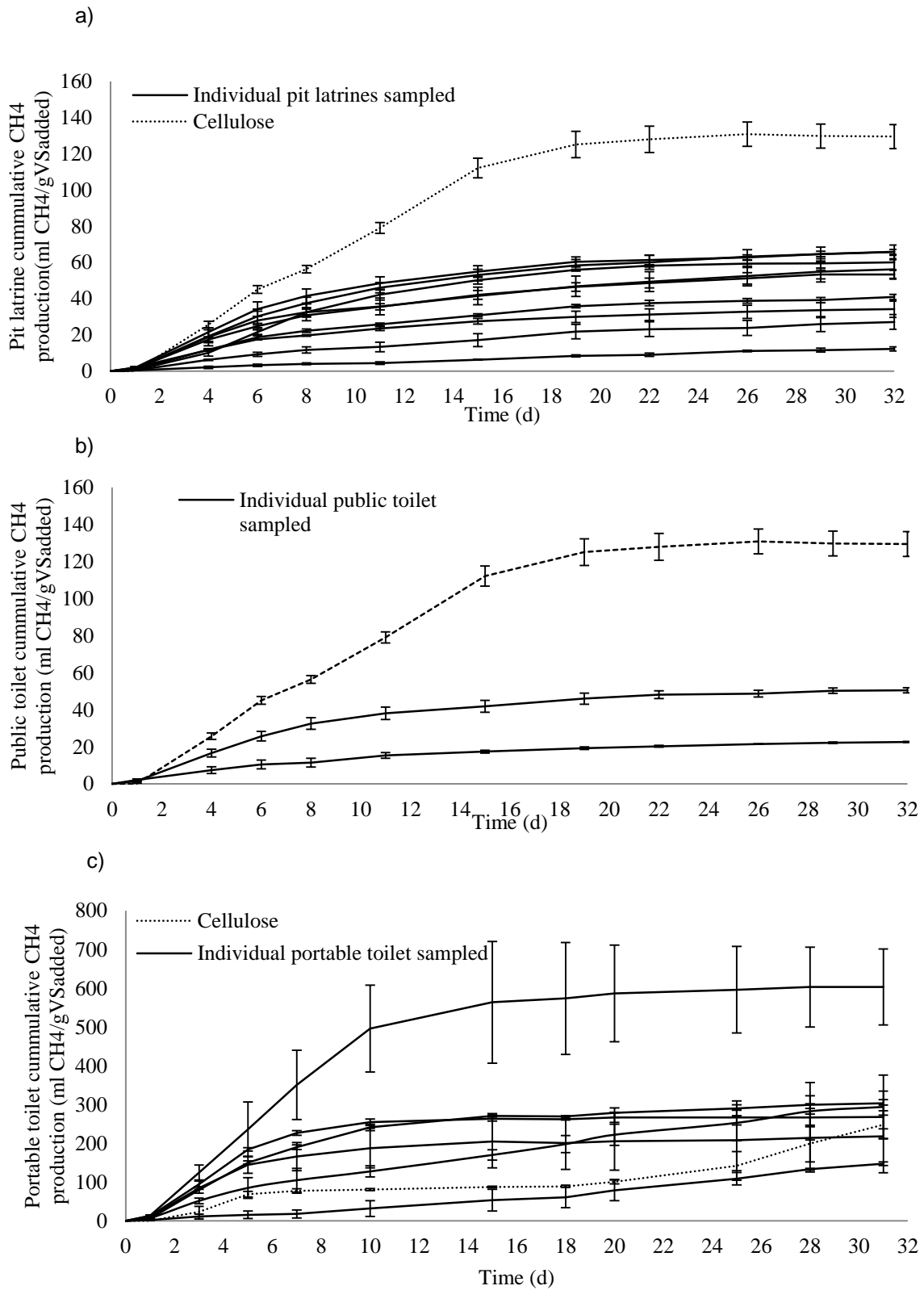
Figure 4.3 The biochemical methane potential of a) pit latrines (n=11) , b) pubic toilets (n=2) and c) portable toilet waste (n=10) in Kanayma, a peri-urban area of *location x*. Dashed line indicates cellulose standards.

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An additional reason for the greater biogas production of primary sludge in comparison to FS (Figure 4.2) could also be accounted for by the inoculum used in the BMP assays (from a mesophilic digester treating primary and waste activated sewage sludge). This inoculum source will be well acclimatised to primary sludge, however, it is possible that it may not have had the extensive trophic microbial composition required to ensure that the FS did not encounter any limitations when combined with FS, resulting in reduced CH_4 yields. As it is likely that FS from pit latrines also had additional materials other than human waste present in the waste stream (e.g. animal waste and vegetation) further verification of the results could be carried out through the mixing of several different inocula together (e.g. from mesophilic digesters treating sewage sludge and food waste) to make a combined inoculum with a wider microbial composition as suggested by Angelidaki et al. (2009). However, an inoculum from an active sewage sludge digester was selected due to it being cited as being a good broad spectrum inoculum suitable for a range of feedstocks (Chynoweth et al., 1993). As the tests were run for 33 days this should have ensured adequate time/acclimatisation for the inoculum to metabolise even the most toxic or unusual pollutants in the FS substrates (Speece, 2008). Additionally, as BMP assays of all FS types produced steady CH_4 production curves (Figure 4.3), without long start up times or inhibition phenomena, the relative impact of the inoculum is not likely to supersede the primary objective of assessing methane potential values.

4.3.2 The impact of sampling depth and storage

In pit latrines, minimal difference in BMP values between the top and bottom layers of the vault (Top: 136 mL/g VS_{added}, bottom: 130 mL/g VS_{added}) were observed. As there was minimal difference between VS levels (36.23% and 35.02% VS/TS in the top and bottom layers respectively), this indicates that predominantly most of the degradation and loss of organics happens rapidly on the surface of the pit latrine (faeces at point of release has a VS content of 84-93% (Rose et al., 2015; Bai and Wang, 2011; Feachem et al., 1978)). The non-readily biodegradable organics that are not rapidly digested on the surface of the pit are then slowly broken down

through anaerobic digestion in the subsequent lower layers. As the difference in BMP values by depth is minimal (Figure 4.4) this is likely to be a very slow process and does not contribute greatly to the reduction of organics. This is in line with findings by Buckley *et al.* (2008) that up to 50% of COD may be degraded under predominantly aerobic conditions on the pit surface. Therefore, if the maximisation of CH_4 yields was desired, the collection of pit latrine sludge at regular intervals (e.g. every 6 months) would not be beneficial, and the collection of the entire pit latrine contents would suffice.

Storage of portable toilet waste in IBC tanks meant that a cross section of the IBC depth could be created and this allowed the assessment of BMP values with depth (Figure 4.4). Within the IBC tank storing portable toilet sludge there was variation according to depth with the top producing BMP values more than 8 times that of the bottom layer (342.6 and 41.7 mL/g VS_{added} respectively). The variation in BMP values throughout the different depths of the IBC are expected due to the flotation and settling of particles. Higher BMP values at the top of the tank (342.6 mL/g VS_{added}) are likely to be due to the flotation of lighter fats, oil and grease particles that could cause a scum layer to form and will resultantly produce higher yields of methane. Lower BMP values from the bottom of the tank (41.7 mL/g VS_{added}) are likely to be due to the settlement of heavier inorganic solids.
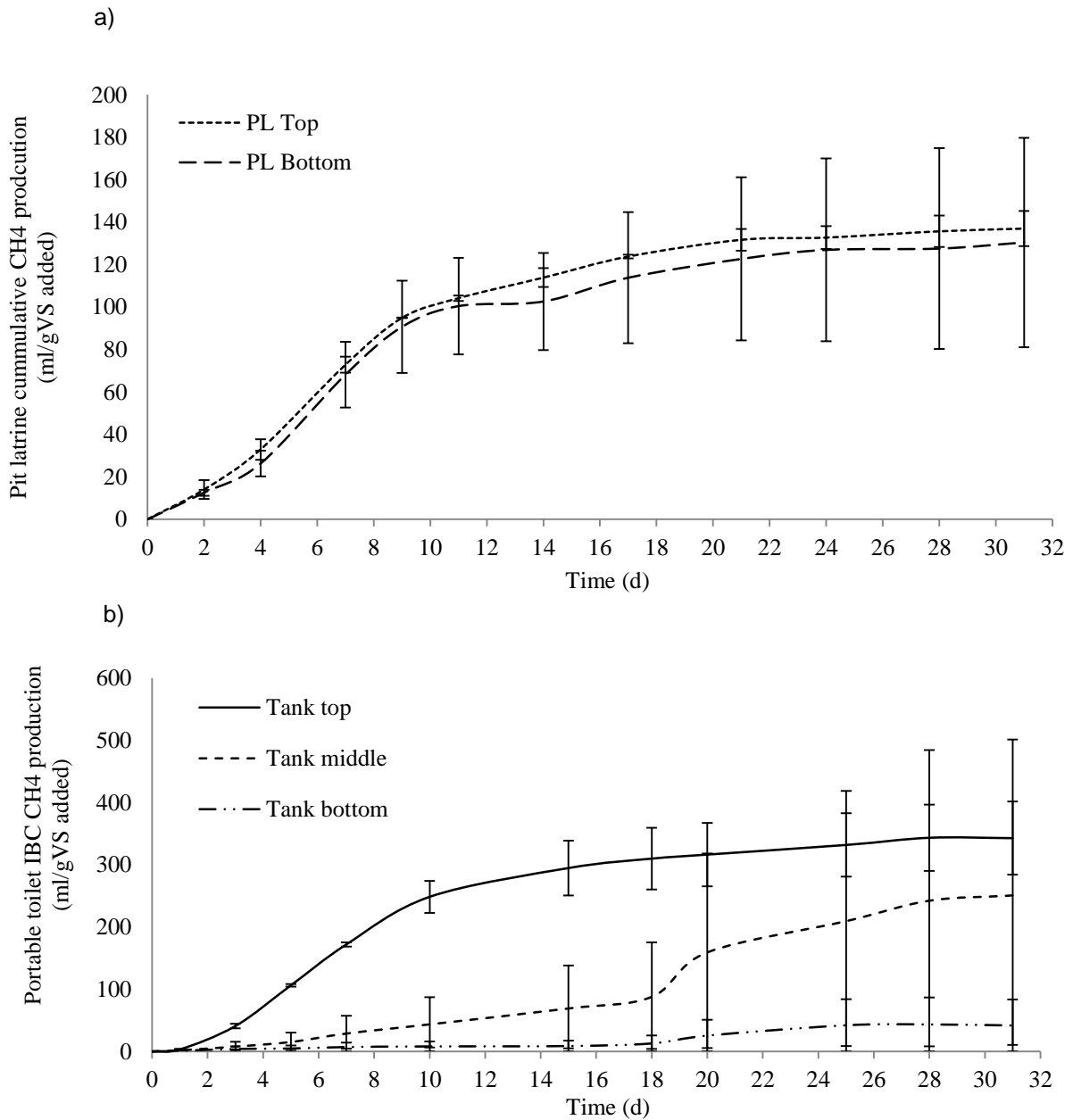
Figure 4.4 The biochemcial methane potential (BMP) of a) the top and bottom layers of pit latrines in *location x* and b) the BMP of a cross section of an intermediate bulk container with FS of portable toilets.

4.3.3 Implications for design and operation of treatment facilities

The BMP values combined with an estimate of organic loading over a given time period, can aid in determining the biogas storage volume for the reactor and calculate a rate of return from biogas production. As AD investment costs are high, careful planning is required to optimise performance and maximise return on investment in regards to methane production. Consequently, an accurate assessment of methane production is essential and BMP assays provide a much improved figure for methane production of different FS types in comparison to stoichiometric predictions of CH_4 production. In addition, the high repeatability of the tests both within (illustrated by the low standard deviation in experiments: Figure 4.3) and across (BMP value of primary sludge, 358.4 mL CH_4/g VS_{added}, within literature values of 230-590 mL CH_4/g VS_{added} (Chynoweth et al., 1993; Elbeshbishy et al., 2012)) studies allows relative comparisons across OSS facility types as well as to more commonly used feedstocks.

However, it is evident that CH_4 yields (Figure 4.3) are under optimal conditions (e.g. temperature and mixing) and actual yields will be reduced in full scale operation, for example, average biogas and methane production was over predicted in BMP trials by 51.4% and 1.2% respectively in comparison to full scale operation when treating dairy manure (Bishop et al., 2009). Additional reasons for CH_4 yield overestimation could be due to the BMP methodology utilised: as dilution is required, it is therefore possible that potential causes of toxicity by FS types are masked and additional continuous bench scale tests may be required to assess potential toxicity due to compound build up or conversely any acclimatisation that could occur.

Nevertheless, despite potential inaccuracies, the range of OSS systems assessed will assist in site specific design criteria for similar OSS facility types as well as AD design for other FS types. Although feedstock BMP assays in isolation cannot be relied upon for digester design, they allow an improved understanding of how digesters may operate. For instance, in all FS types the majority of methane production was complete within 10 days (Figure 4.3) indicating that a sludge retention time of any greater than this would not be beneficial in regards to CH_4 yields.

The characteristics of FS are highly variable and dependent on factors such as OSS system type, usage, collection method, infiltration and storage duration (Bassan et al., 2013; Strande et al., 2014) . However, the primary factor influencing BMP values can be attributed to storage duration: with pit latrine (storage time 1-10 years) values (50.6 ± 19.4mL/g VS_{added}) significantly below that of relatively fresh (≤4 days) portable toilet waste (276.0 ± 151.3mL/g VS_{added}). This factor has significance as it indicates FS should not be compared to primary sewage sludge from WwTW for design purposes, as it may overestimate CH_4 production potential which could in turn influence expected cost recovery expected through CH_4 generation.

4.3.4 Implications for practitioners and the sector

Although OSS facility types in this study were limited to the three types across two geographical locations, the BMP values stated provide a realistic estimate of the anaerobic digestibility of FS that are likely to be significantly more accurate than design values for wastewater sludge in high income countries or stoichiometric calculations from similarly sparse FS characterisation data. Furthermore it has been established that the physical and chemical composition of faeces is not controlled by any overriding factor (Rose et al., 2015), therefore there is no reason to suspect these locations were not representative and applicable to other geographical regions. Consequently, the most important factor in the interpretation of these results is the storage time and conditions within the OSS facility. These factors bear significance as individual site specific feasibility studies are often not financially viable or practical in a low income context.

Existing OSS facilities in peri-urban areas of Sub-Saharan Africa largely consist of pit latrines and public toilets. However, minimal CH_4 production values (Figure 4.2) make the use of AD uninspiring if CH_4 production is the primary motive. However, the production of CH_4 is not always the principal driving force for the implementation of AD systems and can instead be approached from a faecal sludge management perspective: providing benefits such as sludge stabilisation, odour reduction and pathogen destruction. In this context, any CH_4 production is therefore an additional benefit. However, it may be a practical consideration in the design and planning stage to supplement aged FS with additional substrates through co-digestion in order

to reach desired levels of CH_4 production. Alternatively a system could incorporate regular collection of FS, for example through OSS facility types such as portable toilets, that have a shorter retention times and resultantly greater methane yields.

4.4 CONCLUSIONS

In conclusion, this study presents BMP values from three different types of sanitation facilities. Faecal sludge from portable toilets has the potential to produce high volumes of CH_4 (276.0 mL/g VS_{added}), and is comparable to BMP values seen in sewage sludge of a WwTW (358.4 mL CH_4/g VS_{added}). Methane production from the AD of this feedstock could potentially help to recover costs and promote the use of AD as a treatment facility when combined with regular collection. However, the ultimate yield of CH_4 from pit latrine FS is minor (50.6 mL/g VS_{added}) in comparison, partly due to the rapid degradation of organics on the pit surface leaving the non-readily biodegradable organics to be broken down in the remainder of the pit vault. This is a slow process, with minimal difference in BMP values between the top (136 mL/g VS_{added}) and bottom (130 mL/g VS_{added}) of pit latrines. The retention time of the OSS facility type is likely to be the best indicator in assessing potential methane yields of FS substrates from different OSS facilities and this should be used for guideline values, as opposed to BMP values from WwTW, when planning the design, construction and optimisation of AD systems treating FS.

4.5 REFERENCES

Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J., Guwy, A., Kalyuzhnyi, S., Jenicek, P. and Lier, J. v. (2009), "Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays", vol. 59, pp. 927-934.

APHA (2005), *Standard Methods for the Examination of Water and Waste Water,* American Public Health Association, Baltimore, U.S.A.

Bai, F. and Wang, X. (2011), "Study on dynamics model of Biodegration of organic matter in aerobic mesophilic composting reactor for sanitary disposal of human feces", *Environmental, Biomedical and Biotechnology,* vol. 3, pp. 11-16.

Bassan, M., Tchonda, T., Yiougo, L., Zoellig, H., Mahamane, I., Mbeguere, M. and Strande, L. (2013), "Characterisation of faecal sludge during dry and rainy seasons in Ouagadougou, Burkino Faso", *36th WEDEC International Conference, Nakuru, Kenya, 2013. Delivering water, sanitation and hygiene services in an uncertain environment,* 1st-5th July 2013, Nakuru, Kenya, WEDEC, Loughborough University, UK, pp. 1814.

Bishop, G., Burns, R., Shepherd, T., Moody, L., Gooch, C., Spajić, R. and Pronto, J. (2009), "Evaluation of laboratory biochemical methane potentials as a predictor of anaerobic dairy manure digester biogas and methane production", *ASABE Paper,* no. 096769.

Bond, T. and Templeton, M. R. (2011), "History and future of domestic biogas plants in the developing world", *Energy for Sustainable Development,* vol. 15, no. 4, pp. 347-354.

Bryce, D., Croshaw, B., Hall, J., Holland, V. and Lessel, B. (1978), "The activity and safety of the antimicrobial agent bronopol (2-bromo-2-nitropropan-1, 3-diol)", *Journal of the Society of Cosmetic Chemists,* vol. 29, no. 1, pp. 3-24.

Buckley, C., Foxon, K. M., Broukaert, C. J., Rodda, N., Nwaneri, C., Balboni, E., Couderc, A. and Magagna, D. (2008), *Scientific support for the design and operation of Ventilated Improved Pit latrines (VIPs) and the efficacy of pit latrine additives,* WRC project no K5/1630, Water Research Commission, Durban, South Africa.

Chen, L., Zhao, L., Ren, C. and Wang, F. (2012), "The progress and prospects of rural biogas production in China", *Energy Policy,* vol. 51, no. 0, pp. 58-63.

Chynoweth, D. P., Turick, C. E., Owens, J. M., Jerger, D. E. and Peck, M. W. (1993), "Biochemical methane potential of biomass and waste feedstocks", *Biomass and Bioenergy,* vol. 5, no. 1, pp. 95-111.

Clean Team (2012)*, Clean Team Toilets*, available at: http://www.cleanteamtoilets.com/ (accessed August 2014).

Coetzee, M., Van der Merwe, M. and Badenhorst, J. (2011), "Effect of nitrogen loading rates on nitrogen removal by using a biological filter proposed for ventilated improved pit latrines", *International Journal of Environment Science and Technology,* vol. 8, no. 2, pp. 363-372.

Cofie, O., Kone, D., Rothenberger, S., Moser, D. and Zubruegg, C. (2009), "Co-composting of faecal sludge and organic solid waste for agriculture: Process dynamics", *Water Research,* vol. 43, no. 18, pp. 4665-4675.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd Water Research Conference,* Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Diener, S., Semiyaga, S., Niwagaba, C. B., Muspratt, A. M., Gning, J. B., Mbéguéré, M., Ennin, J. E., Zurbrugg, C. and Strande, L. (2014), "A value proposition: Resource recovery from faecal sludge—Can it be the driver for improved sanitation?", *Resources, Conservation and Recycling,* vol. 88, no. 0, pp. 32-38.

Doku, I. A. (2003), *The potential for the use of upflow anaerobic sludge blanket (UASB) reactor for the treatment of faecal sludges in Ghana,* Assainissement A06, Ministère des Affaires Etrangères, Training Research and Networking for Development Group, Kumasi, Ghana.

Drechsel, P., Scott, C. A., Raschid-Sally, L., Redwood, M. and Bahri, A. (eds.) (2009), *Wastewater Irrigation and Health: Assessing and Mitigating Risk in Low Income Countries,* 1st ed, Earthscan/IWMI/IDRC, London.

Drosg, B., Braun, R., Bochmann, G. and Al Saedi, T. (2013), "Analysis and characterisation of biogas feedstocks", *The biogas handbook: Science, production and applications*, Woodhead Publishing Ltd., Cambridge, UK.

Elbeshbishy, E., Nakhla, G. and Hafez, H. (2012), "Biochemical methane potential (BMP) of food waste and primary sludge: Influence of inoculum pre-incubation and inoculum source", *Bioresource Technology,* vol. 110, no. 1, pp. 18-25.

Feachem, D. J., Bradley, H. :., H. and Mara, D. D. (1978), *Health Aspects of Excreta and Wastewater Management,* 1st ed, The World Bank, Washington, DC. U.S.A.

Fytili, D. and Zabaniotou, A. (2008), "Utilization of sewage sludge in EU application of old and new methods-A review", *Renewable and Sustainable Energy Reviews,* vol. 12, no. 1, pp. 116-140.

Gautam, R., Baral, S. and Herat, S. (2009), "Biogas as a sustainable energy source in Nepal: Present status and future challenges", *Renewable and Sustainable Energy Reviews,* vol. 13, no. 1, pp. 248-252.

Gonzalez Gil, G., Seghezzo, L., Lettinga, G. and Kleerebezem, R. (2001), "Kinetics and mass-transfer phenomena in anaerobic granular sludge", *Biotechnology and Bioengineering,* vol. 73, no. 2, pp. 125-134.

Graham, J. P. and Polizzotto, M. L. (2013), "Pit Latrines and Their Impacts on Groundwater Quality: A Systematic Review", *Environmental Health Perspectives,* vol. 121, no. 5, pp. 521.

Heinss, U., Larmie, S. A. and Strauss, M. (1999), *Characteristics of faecal sludges and their solids-liquid separation,* 1, EAWAG/SANDEC, Zurich, Switzerland.

Hounkpe, S. P., Adjovi, E. C., Crapper, M. and Awuah, E. (2014), "Wastewater Management in Third World Cities: Case Study of Cotonou, Benin", *Journal of Environmental Protection,* vol. 5, no. 05, pp. 387.

Kone, D. (2010), "Making urban excreta and wastewater management contribute to cities' economic development: a paradigm shift", *Water Policy,* vol. 12, no. 4, pp. 602-610.

Kone, D., Cofie, O., Zurbrügg, C., Gallizzi, K., Moser, D., Drescher, S. and Strauss, M. (2007), "Helminth eggs inactivation efficiency by faecal sludge dewatering and co-composting in tropical climates", *Water Research,* vol. 41, no. 19, pp. 4397-4402.

Lalander, C., Diener, S., Magri, M. E., Zurbrügg, C., Lindström, A. and Vinnerås, B. (2013), "Faecal sludge management with the larvae of the black soldier fly (Hermetia illucens) — From a hygiene aspect", *Science of The Total Environment,* vol. 458–460, no. 0, pp. 312-318.

Leung, H. (2001), "Ecotoxicology of glutaraldehyde: review of environmental fate and effects studies", *Ecotoxicology and Environmental Safety,* vol. 49, no. 1, pp. 26-39.

Lim, J. W. (2011), "Anaerobic Co-digestion of Brown Water and Food Waste for Energy Recovery", *11th edition of the World Wide Workshop for Young Environmental Scientists (WWW-YES-2011)-Urban Waters: resource or risks?* 5th-10th June 2011, Accueil, France, UPEC/NPEC, France, pp. 1.

Nwaneri, C., Foxon, K., Bakare, B. and Buckley, C. (2008), "Biological degradation processes within a pit latrine", *WISA,* Vol. 1, 18-22 May 2008, Sun CIty, WISA, South Africa, pp. 1.

Owens, J. and Chynoweth, D. (1993), "Biochemical methane potential of municipal solid waste (MSW) components", *Water Science and Technology,* vol. 27, no. 2, pp. 1-14.

Rajagopal, R., Lim, J. W., Mao, Y., Chen, C. and Wang, J. (2013), "Anaerobic co-digestion of source segregated brown water (feces-without-urine) and food waste: For Singapore context", *Science of the Total Environment,* vol. 443, pp. 877-886.

Rose, C., Parker, A., Jefferson, B. and Cartmell, E. (2015), "The Characterisation of faeces and urine; a review of the literature to inform advanced treatment

technology", *Critical Reviews in Environmental Science and Technology,* vol. 45, no. 17, pp. 1827-1879.

Rydin, Y., Bleahu, A., Davies, M., Dávila, J. D., Friel, S., De Grandis, G., Groce, N., Hallal, P. C., Hamilton, I. and Howden-Chapman, P. (2012), "Shaping cities for health: complexity and the planning of urban environments in the 21st century", *The Lancet,* vol. 379, no. 9831, pp. 2079-2108.

Song, Z., Zhang, C., Yang, G., Feng, Y., Ren, G. and Han, X. (2014), "Comparison of biogas development from households and medium and large-scale biogas plants in rural China", *Renewable and Sustainable Energy Reviews,* vol. 33, no. 1, pp. 204-213.

Speece, R. (1996), *Anaerobic Biotechnology for Industrial Wastewaters,* Archae Press, Tennessee, USA.

Speece, R. E. (2008), *Anaerobic biotechnology and odor/corrosion control for municipalities and industries,* 1st ed, Archae Press, Tennessee, USA.

Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) (2014), *Faecal Sludge Management: Systems Approach for Implementation and Operation,* 1st ed, IWA Publishing, London, UK.

Tilley, E., Supply, W. and Council, S. C. (2008), *Compendium of sanitation systems and technologies,* Eawag Dübendorf, Switzerland.

Tilmans, S., Russel, K., Sklar, R., Page, L., Kramer, S. and Davis, J. (2015), "Container-based sanitation: assessing costs and effectiveness of excreta management in Cap Haitien, Haiti", *Environment and Urbanization,* vol. 27, no. 1, pp. 89-104.

Triolo, J. M., Sommer, S. G. and Pedersen, L. (2014), "Influence of freezing/thawing and drying/milling on Biochemical Methane Potential", *Environmental Engineering and Management Journal,* .

Ward, A. J., Hobbs, P. J., Holliman, P. J. and Jones, D. L. (2008), "Optimisation of the anaerobic digestion of agricultural resources", *Bioresource Technology,* vol. 99, no. 17, pp. 7928-7940.

Williams, J. B., Clarkson, C., Mant, C., Drinkwater, A. and May, E. (2012), "Fat, oil and grease deposits in sewers: Characterisation of deposits and formation mechanisms", *Water Research,* vol. 46, no. 19, pp. 6319-6328.

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CHAPTER 5

THE RECOVERY OF NUTRIENTS FROM FAECAL SLUDGE: APPROPRIATE SELECTION AND ADVANCEMENT OF TECHNOLOGY

5 THE RECOVERY OF NUTRIENTS FROM FAECAL SLUDGE: APPROPRIATE SELECTION AND ADVANCEMENT OF TECHNOLOGY

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ABSTRACT

This study reviews the potential for nutrient recovery following the anaerobic digestion of faecal sludge. Faecal sludge characterisation data from pit latrines in *location x* is utilised to make predictions of digestate composition. The high likelihood of pathogens in digestate makes direct application to land unsafe, therefore secondary treatment processes are explored alongside the optimal recovery of nutrients. Nitrogen was found to have the greatest potential for recovery, with significant total nitrogen (1440 mg N L^{-1}) concentrations present in the digestate with current secondary treatment technologies failing to recover this resource in a simple and cost efficient way. The high NH_4-N concentrations (864 mg NH_4-N L^{-1}) as well as the high solids (2.1 g TS L^{-1}) nature of digestate prove problematic to existing NH_4-N recovery technologies and the requirement for a technology that simultaneously dewaters waste and recovers nutrients is apparent.

KEYWORDS

Nutrient recovery, anaerobic digestion, ammonium recovery, faecal sludge management

5.1 INTRODUCTION

On-site sanitation systems can effectively form part of a sustainable system of Faecal Sludge Management (FSM) in peri-urban cities providing that residual sludge from these systems is collected, transported and treated before final reuse or safe disposal (Strande et al., 2014). As part of the FSM chain, anaerobic digestion (AD) is a viable means of treatment for OSS facility waste (Kerstens et al., 2012) and can be operated at varying scales from small rural digesters to larger centralised or semi-centralised systems (Gautam et al., 2009; Bond and Templeton, 2011; Song et al., 2014). Higher rate AD systems, such as up-flow anaerobic sludge blanket (UASB), exhibit great potential providing increased efficiency through the digestion of high solids faecal sludge (FS), with biogas production and stabilisation at a higher rate than unmixed plug-flow digesters (Collins et al., 2013; Parker et al., 2013). The suspended sludge bed in the UASB reactor filters and treats wastewater as the wastewater moves vertically upwards through it. Microbes in the sludge particles act upon the wastewater and break down the organic matter and the up-flow nature, along with gas bubbles, allow efficient mixing without the need for mechanical agitation. Reactors based upon UASB design are attractive due to their compact nature, low sludge production rates and low operational costs with significant cost recovery potential through methane production (Chong et al., 2012). The use of expanded granular sludge bed (EGSB) reactors for high solids waste digestion is also a growing research area (Collins et al., 2013). An EGSB reactor is a variation on the UASB reactor design; which utilises effluent recirculation combined with taller reactors (with a greater height to diameter ratio) and an increased velocity of wastewater applied in order to cause the sludge bed to expand and resultantly increase the contact between sludge and wastewater leading to improved treatment.

However, AD is not a complete treatment tool in itself and should be combined with additional treatment processes in order to protect the environment, through the prevention of diffuse nitrogen (N) and phosphorus (P) pollution (Lu et al.,

2012), and public health through preventing the spread of pathogens and disease vectors (Issah et al., 2012; Katukiza et al., 2012). Resultantly, AD of faecal sludge cannot operate in isolation; instead, an entire treatment flow sheet needs to be developed prior to its implementation in order to achieve comprehensive treatment of AD effluents and ensure the safe recovery and reuse of valuable nutrients.

Human waste comprises a wide range of valuable nutrients (Rose et al., 2015), in particular N and P which along with potassium (K) are the most critical nutrient elements for agriculture and horticulture worldwide (Heinonen-Tanski and van Wijk-Sijbesma, 2005). During the AD process the total mass of nutrients is not reduced and due to the mineralisation of the organic speciation, the inorganic speciation will increase (for instance, the conversion of organic N to ammonium N). In order to assess nutrient recovery potential of waste streams the primary prerequisite is to know the composition of the product that is to be re-used/disposed. However, this information is often not available at the early stages of treatment process flow planning and resultantly there is little emphasis on secondary treatment, resource recovery and the recovery of nutrients from digestate. However, it is possible to predict what the physical and chemical characteristics of AD will be by assessing the influent sludge composition and utilising performance data of similar anaerobic digesters. This information will in turn allow an assessment to be made as to the most appropriate secondary treatment processes and help determine feasible options available when considering technologies for nutrient recovery after AD.

This study will utilise faecal sludge characterisation data from *location x* in order to project nutrient flows around anaerobic digestion. Utilising this data, nutrient recovery processes will be reviewed alongside secondary treatment mechanisms and opportunities for research and development will be explored according to the needs and requirements of the *location x* study site. Specifically, the objectives of this study are to predict the nutrient composition of anaerobically digested faecal sludge using results from a pilot expanded

granular sludge bed (EGSB) reactor (Aguilera et al., 2012); and identify feasible technologies to recover these nutrients in a low income context.

5.2 METHODS

5.2.1 Study site and faecal sludge management system in operation

Location x was used as a study site with data collection taking place in March and December 2014. A peri-urban locality of *location x* was used as an example of a newly implemented Faecal Sludge Management (FSM) scheme which was launched in 2013. This FSM project was carried out in collaboration with WSUP (Water and Sanitation for the Urban Poor) and BORDA (Bremen Overseas Development Association), by the community based water service provider in *location x* who initiated a formalised pit latrine emptying service combined with semi-centralised primary and secondary treatment (WSUP, 2014). Primary treatment is by a batch-fed un-mixed anaerobic digester, with the liquid fraction of the digester discharged to leach fields and the residual solids transported by vacuum tanker to a secondary treatment site where sludge drying beds are used (Figure 5.1). The dried solids are finally utilised as an agricultural soil amendment (WSUP, 2014).
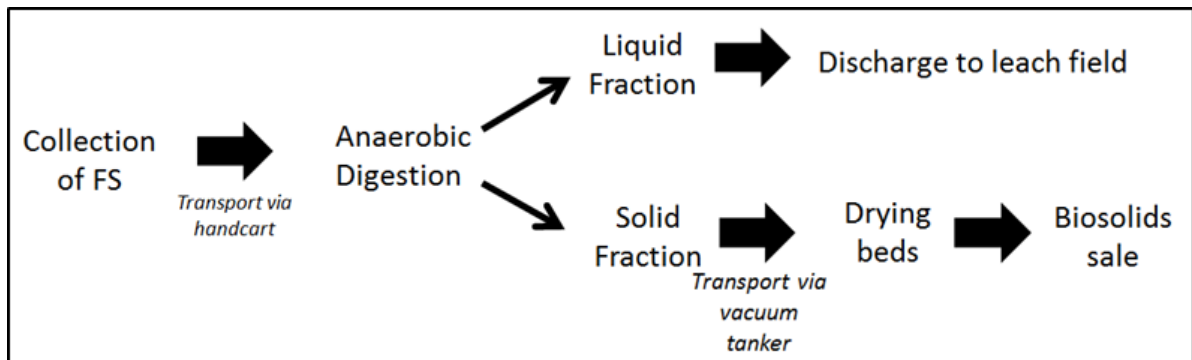
Figure 5.1 Simplified faecal sludge management in *location x.*

5.2.2 On-site sanitation facilities and faecal sludge characterisation

An extensive faecal sludge characterisation programme was carried out in March 2013 by accompanying manual pit latrine emptiers in the peri-urban district of *location x* with data collected from a total of 11 pit latrines. The sampled pit latrines were of a simple design with the drop hole and superstructure raised above ground level by between 1-2 m with no vent pipe present. Pit latrines were partially lined with the exception of one latrine that had a completely sealed tank. Manual methods using an array of modified equipment such as pitchforks and shovels were used to remove sludge from pit latrine chambers and these emptying methods allowed representative samples of the entire contents of pit latrines to be taken. The results of the physical and chemical characterisation (Figure 5.2) of pit latrine sludge are used as the basis for projecting nutrient flows post anaerobic digestion.

Institutional ethical approval (*Cranfield University 109-2013)* was obtained for the sampling and analysis of faecal sludge in both case study locations: *location x* and Kumasi, Ghana.

5.2.3 Predicting anaerobically digested faecal sludge composition

Utilising data regarding the physical and chemical characterisation of pit latrine sludge along with performance data of a pilot-scale mesophilic UASB reactor (Aguilera et al., 2012) a set of projections were constructed in order to establish the composition of anaerobically digested faecal sludge. There are numerous methods of treatment for FS that can be applied to a low income country context. Anaerobic technologies present significant advantages over aerobic technologies due to lesser reliance on costly energy inputs. Primary settlement ponds combined with secondary treatment through Anaerobic Baffled Reactors (ABR), present a simple, effective means of treatment, however, have a large operational footprint in a context where land constraints often prevail. Therefore, the benefit of using anaerobic technologies that operate at a higher rate is paramount and has great potential and was subsequently selected to be incorporated in the analysis An UASB reactor was used for the projections, due

to its aspirational nature and great potential for the treatment of faecal sludge at a higher rate than commonly used plug flow reactors. Due to the design and operation of an UASB digester there is no separation between the solid/liquid fraction, with one combined digestate produced. Assumptions of removal efficiency were determined with a reactor retention time of 7 days and are outlined in Figure 2. As a result of the high solids nature of pit latrine sludge (190 g TS L^{-1}) all influent sludge was diluted to 6 g TS L^{-1} as this was deemed an achievable level in which digestion in a UASB reactor could take place (Collins et al., 2013).

5.3 RESULTS AND DISCUSSION

5.3.1 Digestate output projections

Projections on the composition of the effluent are shown in Figure 5.2. Within these projections it is presumed that microbial pathogens are still present in the AD effluent and require further pathogen destruction before re-use or discharge. This is because two of the principal components casing pathogen decay or loss of viability in AD are temperature and hydraulic retention time (Smith et al., 2005), which are not favourable with higher rate AD processes operating within an EGSB. As a result secondary treatment is essential from a public health perspective.

5.3.2 Secondary treatment of digestate

The pathogenic content of AD effluents combined with the high water content (97.9 % water content) make the use of secondary treatment processes essential but also require the preservation of the valuable resources such as organic matter (4472 mg COD L^{-1}), trace elements and macronutrients (1440 mg N L^{-1}, 1617 mg P L^{-1}, 390 mg K L^{-1}) enabling nutrient recovery. There are many low-cost secondary treatment methods to treat AD effluents: for example, solids and organics reduction can be achieved through waste stabilisation ponds, Imhoff tanks, planted/unplanted drying beds and, following this, the liquid fraction can be treated by aerobic and anaerobic filters or constructed

wetlands which can further reduce organics and nutrients present. Removal of P occurs through a combination of microbial growth, precipitation and adsorption; with nitrogen removal through nitrification/denitrification processes. However, the majority of these processes will not be recovering all of the valuable nutrients present, and through the use of systems such as constructed wetlands, nutrients will be used through plant growth. There is therefore a need for nutrient recovery options that combine secondary treatment and resource recovery, in turn creating a valuable product that is safe for land application, feasible to transport as well as desirable to use.

**Pit latrine
FS influent
(mg L^{-1})***
Diluted to 6% TS

TS: 60000
VS: 28201
COD$_{tot}$ 8943
COD$_{sol}$: 2981

pH: 7.65
Total N: 1440
NH$_4$-N: 585
P$_{sol}$:   27
P$_{tot}$ 1617
K: 390

**Effluent digestate
(mg L^{-1})***

TS: 21001
VS: 14100
COD$_{tot}$: 4472
COD$_{sol}$: 1043

pH: 7.42
Total N: 1440
NH$_4$-N: 864
P: 1617
K: 390

**Gas
collection**

**Recycled
liquid**

Baffle   *Baffle*

Sludge blanket

**Recycled
liquid**

Influent faecal sludge
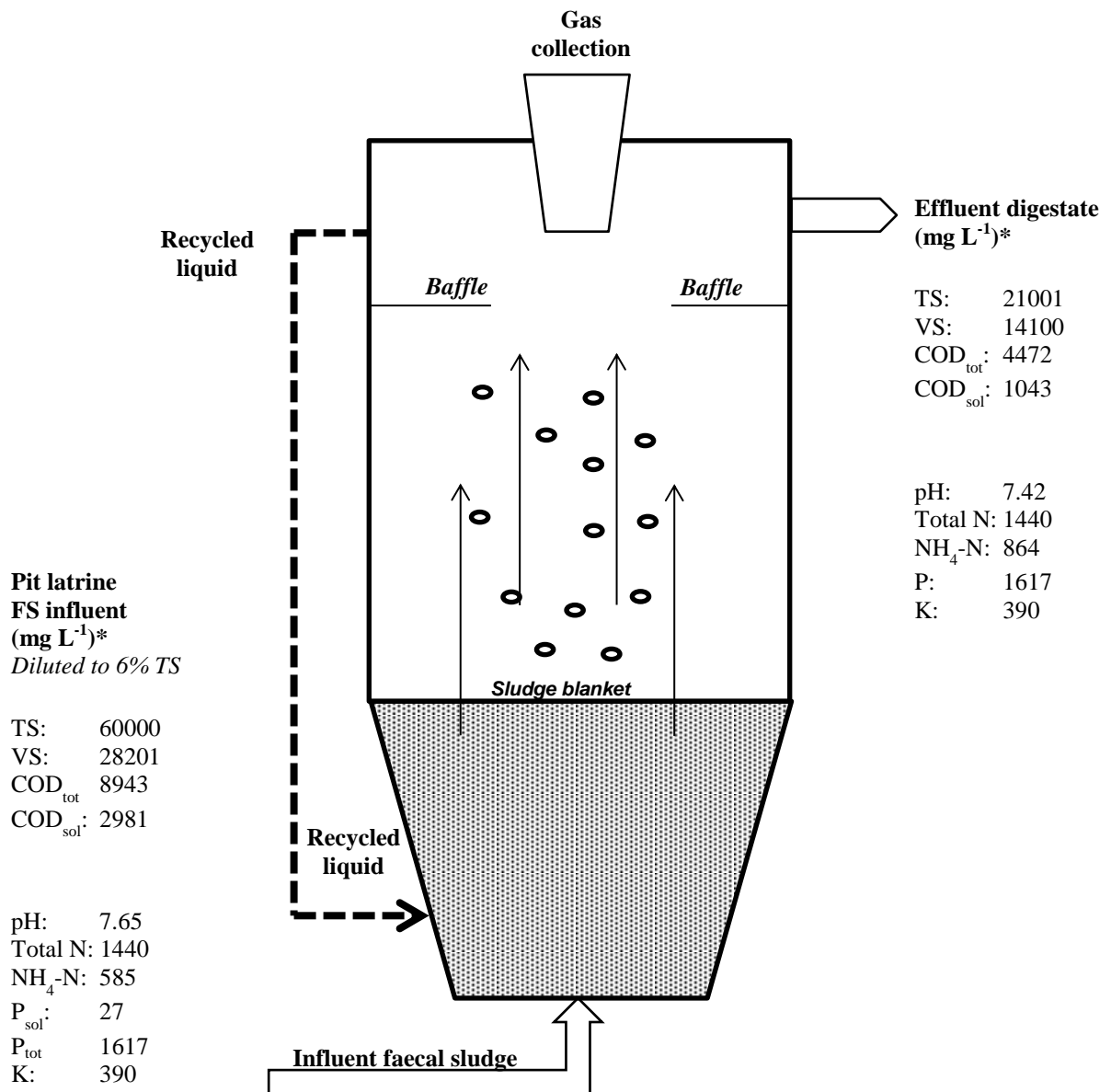
***Assumptions of UASB projections according to Aguilera et al. (2012)**

COD$_{tot}$:   50% removal

COD$_{sol}$:   35% removal

TS: 35% removal

VS: 50% removal

pH: slight reduction

Total N: remain constant

NH$_4$-N: 60% total N to NH4-N

Other Nutrients: remain constant

**Figure 5.2 Mass flow projections of an up flow anaerobic sludge bed (UASB)
reactor fed with pit latrine sludge (diluted to 6 % TS) as a feedstock. Influent
faecal sludge characterisation (left) from location x was used. Solids removal,
organics reduction and nutrient speciation transformation (centre) are based on**

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mass flow projections from the performance data of a pilot scale UASB reactor with a hydraulic retention time of (10 days) and a liquid recycle in an up flow configuration. .

5.3.3 Opportunities for nutrient recovery

The recovery of nutrients for use in agriculture is a recognised resource for potential cost-recovery in many FSM projects. However, in the design stages of treatment process flows, in order to determine nutrient recovery components there is a need to prioritise the most important nutrients to target for recovery.

The residual sludge solids of anaerobically digested FS also contain significant P concentrations (26950 ± 1291 mg P/kg TS) and are comparable to digested sewage sludge biosolids from WwTW in high income countries (19500-29917 mg P/kg TS (Mantovi et al., 2005; He et al., 2010)). It can be estimated that 38% of P present will be bound to the solid matter (Martin, 2005) making the residual digested solids a valuable source of P that can be directly utilised in agricultural soils. In addition, P through the AD and subsequent dewatering steps, will be concentrated in the solid matter providing a valuable source of P to agricultural soils. The reduced solubility of P in dewatered solids also make the direct use attractive due to the reduced likelihood of leaching in soils providing there is no direct run-off of solids or the soil. The remaining 62% of the P fraction will be in the liquor stream after dewatering, making the precipitation of P, for example through struvite precipitation, a nutrient recovery possibility (Miles and Ellis, 2001; Nelson et al., 2003). Resultantly, through the combination of P precipitation in the liquor fraction and the use of dewatered solid matter as a direct source of P in soils, the recovery of P can be fully utilised.

In contrast, the nitrogen fraction is more problematic due to the potential volatilisation and loss of nitrogen when stored or applied direct to the soil. The outputs of AD are predicted for the case study site (Figure 5.2) have significant quantities of NH_4-N (840 mg NH_4-N L^{-1}). If there is a solid/liquid separation

process, the majority of NH_4-N (89%) will be in the liquor fraction (Martin, 2005) and if there is no solid/liquid separation there is the high likelihood of ammonia (NH_3) volatilisation post in digestate. The significant quantities of NH_4-N in digestate, result in the need for careful N management, due to the damaging impact of N when leached to the surrounding environment. This often results in limiting the application of digestate during seasons of high rainfall. However, agricultural food production is heavily reliant on N fertiliser inputs in order to achieve successful yields worldwide (Xu et al., 2012). Ammonium nitrate is the most common nitrogen fertiliser product in Europe (Fertilizers Europe, 2013), with production through the Haber-Bosch process consuming more than 1% of the world's power production (Kitano et al., 2012). This factor resultantly makes the cost of N fertilisers reliant on global gas prices, which frequently has the negative impact of out-pricing farmers in Sub-Saharan Africa. Therefore, if N recovery from wastewater could be achieved, this could help to decrease demand for artificial fertiliser production and offset the ecological impact of energy usage through N production (McCarty et al., 2011). In addition, low income countries that are previously reliant on international imports and global energy prices, could become partially self-sufficient in regards to their domestic N demand making them less vulnerable to global market fluctuations. This factor could in turn make N fertilisers which are frequently too expensive due to energy prices and import taxation for low income countries increasingly more available.

Resultantly, in situations such as the case study site, there are significant drivers for designing NH_4-N recovery systems as the principle target for nutrient recovery as opposed to P recovery systems. The use of dewatered solids provides a sustainable source of P to agricultural soils with minimal management measures required. Whereas, direct application of digestate provides localised environmental issues as well as having wider financial and ecological impacts. It is therefore evident that if N could be recovered and released in a slow release format, this would provide significant benefit to

farmers and the surrounding environments in low income countries such as *location x*.

5.3.4 Direct application of anaerobic digestate

After the disinfection or pasteurisation of anaerobic digestate, direct application to land can be an appropriate means of nutrient recovery and is widely practised in high (WRAP, 2012) and low income countries (Vögeli et al., 2014). Projections of the AD composition in *location x* indicate significant nutrient potential in the digestate with 2.12 kg nitrogen/tonne, 0.26 kg phosphorus/tonne and 0.39 kg potassium/tonne. In addition to this, valuable micro nutrients/trace elements are present (68 mg/kg CU, 84 mg/kg Ni and 420 mg/kg Zn) which further benefits crop production. In *location x*, sludge from waste stabilisation ponds at the municipal wastewater treatment works is regularly purchased and utilised for landscaping purposes, a factor that illustrates demand and acceptance for human derived by-products at large scale farming operations. In contrast, at the household level, extensive interviews conducted in *location x* established that 60.7% of respondents believed it was not safe to use human derived fertiliser after treatment (Kennedy-Walker et al., 2015). Demand for using digestate as a complete product is therefore likely to be from large scale farmers from the fringes of *location x* who have the equipment and capital to purchase and use digestate as a fertiliser product instead of the small-scale peri-urban farmer.

Digestate can be transported from the treatment site to farmland by tanker and distributed via an irrigation network or mechanical spreader. However, digestate storage facilities must be sufficient in order to enable substantial storage until the desired time of year for application, and sludge must be directly injected or incorporated into the ground to avoid nitrogen loss through NH_3 volatilisation. The volatilisation and loss of NH_3 following application of digested sludge is a key concern with Smith et al. (2007) reporting losses of 22-34% of NH_4-N applied via trailing hose application. These high costs of storage, transportation and application cause a barrier to the use of digestate. The separation of the

liquid and fibre fractions of the digestate would reduce transportation costs significantly due to volume reduction; however, the liquid fraction would still require either substantial irrigation networks or transportation to farmland.

It is resultantly evident that more valuable products could be produced if nutrients could be removed, recovered and concentrated on-site: creating high value products that can be marketed without the transportation and application difficulties that applying digestate brings.

Table 5.1 Ammonium recovery options from anaerobic digestate

Ammonium Recovery Process	Process Description	Pre-treatment Required	Limitations and applicability to low income context	References
Algae ponds	NH_4-N taken up by algae for biomass growth	Significant TS reduction required and dilution	Sensitive to high concentrations and fluctuations of NH_4-N	(Hoffmann, 1998; Wilkie and Mulbry, 2002; Mulder, 2003)
Reverse Osmosis	NH_4-N concentrated in semi-permeable membrane	TS reduction required	Solid/liquid separation required to reduce membrane fouling and clogging, high energy use	(Mondor et al., 2008; Ledda et al., 2013)
Ammonia Stripping	Raised pH to convert to NH_3, air passed through solution into concentrated liquid $(NH_4)2SO_4$	Chemical additions for pH adjustment, solid/liquid separation	High energy usage of compressed air, high temperature required high cost of chemical requirements to raise pH. Air stripping towers are large and can become clogged by solids. Acid required to convert NH_3 to marketable fertiliser.	(Guštin and Marinšek-Logar, 2011; Li et al., 2014; Serna-Maza et al., 2014)
Struvite Precipitation	$NH_4MgPO_4 \cdot 6H_2O$ mineral precipitation when ions are released	Mg and P addition and pH adjustment	High cost of chemical additions, extra processing costs of Ca ions , process difficulties with high solids effluents.	(Miles and Ellis, 2001; Nelson et al., 2003)
Ion Exchange	NH_4-N ions present in wastewater are exchanged with ions of media resin	Solid/liquid separation to prevent media clogging	Media costs and/or regeneration cost of chemicals	(Wang and Wu, 2006; Hankins et al., 2005; Cooney et al., 1999)
Adsorption	NH_4-N adsorbed to surface of media which was strong adsorption capacity	Solids/liquid separation likely to be required	Solids inhibit the adsorption capacity of media, cost of media and/or regeneration costs including chemicals and infrastructure.	(Vassileva et al., 2009; Sarkhot et al., 2013)

5.3.5 Impact of predictions on existing NH_4-N recovery technologies

There are numerous means of recovering ammonium from digestate which are described in Table 5.1, however, many of the processes (reverse osmosis, NH_3 stripping and struvite precipitation) present significant challenges to application in a low income context as they have significant energy needs, and require chemicals and consumables that are both costly and have a difficult-to-manage supply chain. Algae ponds, although having a low capital and operational costs are sensitive to the high NH_4-N levels in digestate (1440 mg NH_4-N L^{-1}), and high TS in the effluent will also be problematic as it will impact light availability of the algae. The two technologies with the most potential for NH_4-N recovery in this context are adsorption and ion exchange processes, with the primary challenge in the recovery of nutrients being the high solids nature of the digestate (2.1 g TS L^{-1}), high organic matter (4472 mg COD L^{-1}) and the cost or regeneration of adsorption or ion exchange medias.

5.3.6 Separation technologies and opportunities for ammonium recovery

The majority of ammonium recovery processes therefore require separation of the solid and liquid fractions in order for nutrient recovery processes to take place. Dewatering can be achieved through centrifugation, filtration and evaporation or through a combination of these processes. The majority of mechanical means of dewatering (e.g. centrifugation and belt presses) are not suitable for low income countries due to the high levels of capital and operational expenditure. Nevertheless, dewatering can take place through simple processes of filtration and evaporation; with unplanted sludge drying beds one of the most numerous forms of digested sludge dewatering in developing countries (Koné and Strauss, 2004).

However, simplified process flow sheets in faecal sludge treatment are important. Therefore, if a dewatering system could be combined with the recovery of ammonium this would present a significant benefit. Due to the simplicity and wide application of sludge drying beds in developing countries

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adapting a hybrid between sludge drying bed and an adsorption or ion exchange system would be a feasible and realistic target for the recovery of NH_4-N.

5.3.7 Application of low cost media adsorbents

Numerous types of adsorbents for NH_4-N removal from wastewater have been investigated; for example activated carbon, limestone, clay and zeolites (Horan et al., 1997; Rožić et al., 2000; Aziz et al., 2004; Wang et al., 2006). One of the greatest limitations to the application of adsorbents and ion exchange media for nutrient recovery in wastewater treatment is the cost of regenerating exhausted media (Wang and Wu, 2006). Regeneration of media causes operational complications, increased infrastructure and uncertainties that prevent simple treatment flows for application in a low income country context. Consequently, the selection of media should not only account for NH_4-N uptake potential, but also for potential as a soil amendment once saturated in NH_4-N. This approach could form two approaches: firstly a relatively inert material could be selected with a view to adding significant value (e.g. through the use of natural zeolites) which are relatively inert and have been used as filler products in fertilisers (Pawełczyk and Popowicz, 2006), secondly a material already established for its benefits as a soil amendment product could be used (e.g. biochar (Jeffery et al., 2015)) and further value added to it through the capture of nutrients.

5.4 CONCLUSIONS

In conclusion this study has utilised the performance data of an existing EGSB reactor (Aguilera et al. 2012) to make predictions of the composition of the AD effluent based on faecal sludge characterisation data collected. Significant opportunities to recover nutrients were highlighted, especially NH_4-N, from the digestate of AD treating faecal sludge. Despite many methods of secondary treatment, all fail to capture nutrients such as NH_4-N in a manner that is both cost-efficient and appropriate for a low income context. Adsorption and ion exchange have been identified as feasible methods for NH_4-N recovery in this context; however, high costs as well as complications involved in the

regeneration process promote the use of ion exchange/adsorbent media that can be directly applied to agricultural land as an enhanced fertiliser product. Furthermore, if the ion exchange/adsorption processes could be combined within a sludge drying bed configuration, enabling digestate dewatering and simultaneous capture of NH_4-N a significant benefit could be gained. Through this, process flows would be simplified and a valuable product created that will be more concentrated in regards to nutrient values, easier to transport, store and apply to agricultural land, as well as creating a product that is more detached from human waste appearance that currently limits social acceptance in peri-urban agriculture.

5.5 REFERENCES

Abbasi, T. and Abbasi, S. A. (2012), "Formation and impact of granules in fostering clean energy production and wastewater treatment in upflow anaerobic sludge blanket (UASB) reactors", *Renewable and Sustainable Energy Reviews,* vol. 16, no. 3, pp. 1696-1708.

Aguilera, Y., Cartmell, E., McAdam, E. J. and Lester, J. N. (2012), "Understanding the criticality of granule integrity to resilience of anaerobic wastewater treatment", *IWA World Congress on Water Climate & Energy,* Vol. 1, 13-18 May 2012, Dublin, Ireland, IWA, London, pp. 1.

Aziz, H. A., Adlan, M. N., Zahari, M. S. and Alias, S. (2004), "Removal of ammoniacal nitrogen (N-NH3) from municipal solid waste leachate by using activated carbon and limestone", *Waste management & research : the journal of the International Solid Wastes and Public Cleansing Association, ISWA,* vol. 22, no. 5, pp. 371-375.

Bond, T. and Templeton, M. R. (2011), "History and future of domestic biogas plants in the developing world", *Energy for Sustainable Development,* vol. 15, no. 4, pp. 347-354.

Chong, S., Sen, T. K., Kayaalp, A. and Ang, H. M. (2012), "The performance enhancements of upflow anaerobic sludge blanket (UASB) reactors for domestic sludge treatment - A State-of-the-art review", *Water Research,* vol. 46, no. 11, pp. 3434-3470.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd Water Research Conference,* Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Cooney, E. L., Booker, N. A., Shallcross, D. C. and Stevens, G. W. (1999), "Ammonia removal from wastewaters using natural Australian zeolite. I. Characterization of the zeolite", *Separation Science and Technology,* vol. 34, no. 12, pp. 2307-2327.

Gautam, R., Baral, S. and Herat, S. (2009), "Biogas as a sustainable energy source in Nepal: Present status and future challenges", *Renewable and Sustainable Energy Reviews,* vol. 13, no. 1, pp. 248-252.

Guštin, S. and Marinšek-Logar, R. (2011), "Effect of pH, temperature and air flow rate on the continuous ammonia stripping of the anaerobic digestion effluent", *Process Safety and Environmental Protection,* vol. 89, no. 1, pp. 61-66.

Hankins, N., Pliankarom, S. and Hilal, N. (2005), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

He, Z., Zhang, H., Toor, G. S., Dou, Z., Honeycutt, C. W., Haggard, B. E. and Reiter, M. S. (2010), "Phosphorus distribution in sequentially extracted fractions of biosolids, poultry litter, and granulated products", *Soil Science,* vol. 175, no. 4, pp. 154-161.

Heinonen-Tanski, H. and van Wijk-Sijbesma, C. (2005), "Human excreta for plant production", *Bioresource Technology,* vol. 96, no. 4, pp. 403-411.

Hoffmann, J. P. (1998), "Wastewater treatment with suspended and nonsuspended algae", *Journal of Phycology,* vol. 34, no. 5, pp. 757-763.

Horan, N. J., Gohar, H. and Hill, B. (1997), "Application of a granular activated carbon-biological fluidised bed for the treatment of landfill leachates containing high concentrations of ammonia", *Water Science and Technology,* vol. 36, no. 2–3, pp. 369-375.

Issah, A., Aklaku, E. D. and Salifu, T. (2012), "Comparative study of effluent for pollutionindicators and indicator pathogenic organisms from anerobic digesters from human and fruit wastes", *ARPN Journal of Agricultural and Biological Science,* vol. 6, no. 6, pp. 416-420.

Jeffery, S., Bezemer, T. M., Cornelissen, G., Kuyper, T. W., Lehmann, J., Mommer, L., Sohi, S. P., Voorde, T. F., Wardle, D. A. and Groenigen, J. W. (2015), "The way forward in biochar research: targeting trade-offs between the potential wins", *GCB Bioenergy,* vol. 7, no. 1, pp. 1-13.

Katukiza, A. Y., Ronteltap, M., Niwagaba, C. B., Foppen, J. W. A., Kansiime, F. and Lens, P. N. L. (2012), "Sustainable sanitation technology options for urban slums", *Biotechnology Advances,* vol. 30, no. 5, pp. 964-978.

Kennedy-Walker, R., Amezaga, J. and Patterson, C. (2015), "Planning for improved faecal sludge management for informal settlements in x", *The 3rd International Faecal Sludge Management Conference,* 18th-21st January, Hanoi, Vietnam, FSM3, EAWAG, pp. 1.

Kerstens, S., Legowo, H. and Gupta, I. H. (2012), "Evaluation of DEWATS in Java, Indonesia", *Journal of Water, Sanitation and Hygiene for Development,* vol. 2, no. 4, pp. 254-265.

Koné, D. and Strauss, M. (2004), "Low-cost options for treating faecal sludges (FS) in developing countries–Challenges and performance", *9th International IWA Specialist Group Conference on Wetlands Systems for Water Pollution Control and to the 6th International IWA Specialist Group Conference on Waste Stabilisation Ponds,* Vol. 27, 27 Sept. – 1 Oct., 2004, Avignon, France, IWA, London, UK, .

Ledda, C., Schievano, A., Salati, S. and Adani, F. (2013), "Nitrogen and water recovery from animal slurries by a new integrated ultrafiltration, reverse osmosis and cold stripping process: A case study", *Water Research,* vol. 47, no. 16, pp. 6157-6166.

Li, A. F., Ning, Y. Y., Xu, W. J. and Pan, T. (2014), "Air Stripping of Ammoniacal Nitrogen from Fecal Sewage Using a Fluidized Bed", *Advanced Materials Research,* Vol. 926, Trans Tech Publ, pp. 4209.

Lu, Q., He, Z. L. and Stoffella, P. J. (2012), "Land application of biosolids in the USA: A review", *Applied and Environmental Soil Science,* vol. 2012, no. Article ID 201462, pp. 1-11.

Mantovi, P., Baldoni, G. and Toderi, G. (2005), "Reuse of liquid, dewatered, and composted sewage sludge on agricultural land: effects of long-term application on soil and crop", *Water Research,* vol. 39, no. 2–3, pp. 289-296.

Martin, J. H. (2005), *An evaluation of a Mesophilic, modified plug flow anaerobic digester for dairy cattle manure,* EPA Contract No. GS 10F-0036K Work Assignment/Task Order 9, Eastern Research Group, Inc, Lexington, USA.

Miles, A. and Ellis, T. G. (2001), "Struvite precipitation potential for nutrient recovery from anaerobically treated wastes", *Water Science and Technology,* vol. 43, no. 11, pp. 259-266.

Mondor, M., Masse, L., Ippersiel, D., Lamarche, F. and Massé, D. I. (2008), "Use of electrodialysis and reverse osmosis for the recovery and concentration of ammonia from swine manure", *Bioresource Technology,* vol. 99, no. 15, pp. 7363-7368.

Mulder, A. (2003), "The quest for sustainable nitrogen removal technologies", *Water Science and Technology,* vol. 48, no. 1, pp. 67-75.

Nelson, N. O., Mikkelsen, R. L. and Hesterberg, D. L. (2003), "Struvite precipitation in anaerobic swine lagoon liquid: effect of pH and Mg: P ratio and determination of rate constant", *Bioresource technology,* vol. 89, no. 3, pp. 229-236.

Parker, A., Kennedy-Walker, R., Rose, C., Cartmell, E. and Collins, G. (2013), "A novel, high-rate, anaerobic digester to treat high solids waste ensuring re-use and good sanitation planning", *Delivering water, sanitation and hygiene services in an uncertain environment,* July 1st-5th, Nakkuru, Water Engineering and Development Center (WEDC)), Loughborough University, pp. 1.

Pawełczyk, A. and Popowicz, A. (2006), "Application of natural zeolites in the production of controlled release fertilizers", *Book of Abstracts,* pp. 203.

Rose, C., Parker, A., Collins, G. and Cartmell, E. (2015a), "Nutrient recovery form high solids anaerobic digestion effluents in *Location x*", *3rd International Faecal Sludge Management Conference,* 18-21 January, Hanoi, Vietnam.

Rose, C., Parker, A., Jefferson, B. and Cartmell, E. (2015b), "The Characterisation of faeces and urine; a review of the literature to inform advanced treatment technology", *Critical Reviews in Environmental Science and Technology* 45, 1-53

Rožić, M., Cerjan-Stefanović, Š, Kurajica, S., Vančina, V. and Hodžić, E. (2000), "Ammoniacal nitrogen removal from water by treatment with clays and zeolites", *Water Research,* vol. 34, no. 14, pp. 3675-3681.

Sarkhot, D., Ghezzehei, T. and Berhe, A. (2013), "Effectiveness of biochar for sorption of ammonium and phosphate from dairy effluent", *Journal of Environmental Quality,* vol. 42, no. 5, pp. 1545-1554.

Serna-Maza, A., Heaven, S. and Banks, C. J. (2014), "Ammonia removal in food waste anaerobic digestion using a side-stream stripping process", *Bioresource Technology,* vol. 152, no. 0, pp. 307-315.

Smith, K., Metcalfe, P., Grylls, J., Jeffrey, W. and Sinclair, A. (2007), *Nutrient value of digestate from farm-based biogas plants in Scotland. Report for Scottish Executive Environment and Rural Affairs Department-ADA/009/06.*

Smith, S., Lang, N., Cheung, K. and Spanoudaki, K. (2005), "Factors controlling pathogen destruction during anaerobic digestion of biowastes", *Waste Management,* vol. 25, no. 4, pp. 417-425.

Song, Z., Zhang, C., Yang, G., Feng, Y., Ren, G. and Han, X. (2014), "Comparison of biogas development from households and medium and large-scale biogas plants in rural China", *Renewable and Sustainable Energy Reviews,* vol. 33, no. 0, pp. 204-213.

Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) (2014), *Faecal Sludge Management: Systems Approach for Implementation and Operation,* 1st ed, IWA Publishing, London, UK.

Vassileva, P., Tzvetkova, P. and Nickolov, R. (2009), "Removal of ammonium ions from aqueous solutions with coal-based activated carbons modified by oxidation", *Fuel,* vol. 88, no. 2, pp. 387-390.

Vögeli, Y., Riu, C., Amalia, L., Diener, S. and Zurbrügg, C. (2014), *Anaerobic digestion of biowaste in developing countries,* , Eawag – Swiss Federal Institute of Aquatic Science and Technology, Dübendorf, Switzerland.

Wang, S. and Wu, H. (2006), "Environmental-benign utilisation of fly ash as low-cost adsorbents", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 482-501.

Wang, Y., Liu, S., Xu, Z., Han, T., Chuan, S. and Zhu, T. (2006), "Ammonia removal from leachate solution using natural Chinese clinoptilolite", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 735-740.

Wilkie, A. C. and Mulbry, W. W. (2002), "Recovery of dairy manure nutrients by benthic freshwater algae", *Bioresource Technology,* vol. 84, no. 1, pp. 81-91.

WRAP (2012), *Using quality anaerobic digestate to benefit crops,* 1, Waste and resource action plan, UK.

WSUP (2014), *FSM services in x: moving up the excreta management ladder,* pn#017, Water and Sanitation for the Urban Poor, London, UK.

CHAPTER 6

PASSIVE AMMONIUM RECOVERY THROUGH THE USE OF LOW-COST MEDIA WITHOUT REGENERATION

6 PASSIVE AMMONIUM RECOVERY THROUGH THE USE OF LOW COST MEDIA WITHOUT REGENERATION

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ABSTRACT

This study investigates the potential for the use of non-regenerative media as part of a passive treatment system for the recovery of ammonium from high strength (>200 mg L^{-1}) wastewater. Clinoptilolite and biochar were identified as suitable non-regenerative media. Batch adsorption and kinetic experiments were carried out along with dynamic column experiments to determine the media capacity, the most efficient operation and configuration in order to establish scale-up design values. Clinoptilolite was established as the more effective media with a superior capacity to biochar (12.2 and 5.0 g NH_4-N/kg respectively) despite a 23.8% reduction in the uptake capacity due to the presence of competing cations in high strength wastewater. Empty bed contact time (20-354 minutes) was the most important factor influencing clinoptilolite performance with the most efficient operation (4.45-8.73 kg/$m^3_{treated}$) at empty bed contact times of ≥ 226 minutes. Increasing influent NH_4-N concentrations caused quicker exhaustion with bed volumes treated to exhaustion decreasing from 635 to 301 with an increase from 60 to 200 mg NH_4-N L^{-1}. A passive nutrient recovery system (with a feasible footprint of 67-190 L/m^2/day) for the recovery of ammonium is a viable alternative treatment and recovery option, utilising clinoptilolite

as a non-regenerative media that can subsequently be used directly as an enriched agricultural fertiliser product.

KEY WORDS

Ammonium recovery, ion exchange, nutrient recovery, clinoptilolite, biochar.

HIGHLIGHTS

- The non-regenerative use of clinoptilolite is proposed for ammonium recovery.

- Clinoptilolite had a greater NH_4-N capacity to biochar (9.3 and 4.8 mg NH_4-N/g).

- Operation at long empty bed contact times (>226 mins) provides the most efficient uptake of NH_4-N.

- A passive system can remove NH_4-N and the direct use of saturated media can achieve nutrient recovery.

6.1 INTRODUCTION

Ammonia is one of the major pollutants introduced into receiving natural waterways by industrial, domestic and agricultural wastewater discharges (Hasanoğlu et al., 2010). This has significance due to the toxic effect of ammonia on most fish species (Tetreault et al., 2013) as well as the biological nitrification of ammonia to nitrite and nitrates which are undesirable to human health (Cockburn et al., 2013). Wastewaters with high concentrations of ammonium-nitrogen (>200 mg NH_4-N L^{-1}) are produced due to a wide range of human activities: such as landfill leachates, sewage treatment works, agricultural slurries as well as numerous chemical and industrial processes. Many of these wastewater streams are treated through sludge digestion, and effluents from subsequent dewatering processes frequently contain high NH_4-N concentrations, with typically 15-30% of the total nitrogen (N) load in a sewage treatment works found as NH_4-N in centrate or filtrate streams (US EPA, 2007).

Not only is there an environmental driver for reducing NH_4-N discharges to waterways but there is also an increasing ecological driver to recover rather than remove key macronutrients (Guest et al., 2009), such as N, required in plant production. Agricultural productivity relies heavily on N fertilisers in order to achieve maximum crop production (Xu et al., 2012). Nitrogen fertiliser production amounted

to 113 million tonnes in 2014, with forecasted growth of 1.4% annually until 2018 (FAO, 2015). Ammonium nitrate is the most commonly used compound for chemical nitrogen fertiliser production in Europe (Fertilizers Europe, 2013), with production through the Haber-Bosch process consuming more than 1% of the world's power production (Kitano et al., 2012). Consequently, if the sustained recovery of N from wastewater could be achieved, this could help to decrease demand for artificial fertiliser production and offset the ecological impact of energy usage through N production (McCarty et al., 2011).

Existing technologies for NH_4-N recovery, such as membrane separation and air stripping, have a high energy demand (Park and Kim, 2015) and as a result are mainly confined to large wastewater treatment works (Liu et al., 2014). There is however, a need for passive treatment technologies, e.g. constructed wetlands and sand filtration systems, where conventional wastewater treatment works are uneconomical to construct, operate and maintain (Speer et al., 2012). These passive systems have significant benefits to situations where it is less cost efficient to operate high intensity large scale processes and fit in line with technology development recommendations in low income countries (Parkinson and Tayler, 2003). There is, consequently, a strong driver for the development of passive solutions to recover NH_4-N from wastewater.

Ion exchange and adsorption systems have the potential to be applied in a passive way to recover NH_4-N from high strength waste streams, such as anaerobic liquors, with concentrations reported at 200-700 and 520-1853 mg NH_4-N L^{-1} in faecal sludge and sewage sludge respectively (Thornton et al., 2007; Rose et al., 2015). However, conventional ion exchange and adsorption processes require the regeneration of exhausted media to take place when reaching uptake capacity (Hedström, 2001). This factor creates one of the greatest complications and costs involved in the process: requiring increased infrastructure, purchase of chemical regenerants and disposal costs for regenerants (Wang and Wu, 2006). Consequently, this promotes the potential of low cost single-use non-regenerative media in passive adsorption and ion exchange processes. This media, following exhaustion, can then be applied directly to land as an enhanced fertiliser product, achieving effective low cost nutrient recovery. This concept has been applied to

phosphorus recovery in wastewater treatment with non-regenerative media promoted as a calcium phosphate fertiliser product (Johansson and Gustafsson, 2000; Berg et al., 2006; Hylander et al., 2006), however, its application in the recovery of NH_4-N is limited.

In order to effectively design a fixed bed absorber for the recovery of NH_4-N in a passive system there are two major considerations. Firstly, an appropriate media must be selected and the uptake capacity determined using the expected wastewater composition; secondly, the most efficient operational configuration of the media bed must be determined. Media capacity is primarily determined through the use of batch adsorption isotherms and kinetic studies (Bulut et al., 2008; Guo et al., 2008; Vassileva et al., 2009). The design of a fixed bed absorber can also be calculated through batch adsorption isotherm and kinetic data, using theoretical models utilising mass transfer coefficients (Bulut et al., 2008; Barros et al., 2013). However, an empirical approach is preferable with pilot scale columns being the most reliable means of determining the effect of key operational parameters such as empty bed contact time (EBCT), bed depth and influent NH_4-N concentrations (Cooney et al., 1999). Through the operation of pilot scale columns at anticipated flow rates, bed depths and influent NH_4-N concentrations expected in the final application, the most reliable design criteria for scale-up can be determined.

6.1.1 The selection of non-regenerative media

The selection of non-regenerative media that could be used in a passive NH_4-N recovery application is dependent on three primary factors: firstly the media must have a low enough purchase price so it can be reimbursed in the final sale of the enhanced product, its physico-chemical properties must be beneficial to soil application and, finally, the media must capture a significant quantity of NH_4-N to enrich the product. A preliminary review identified biochar and clinoptilolite as meeting these criteria.

Biochar is solid carbon residue that is produced through the pyrolysis of biomass and is generally distinguished from the term charcoal due to its intended final use as a soil amendment (Sohi et al., 2009). Biochar has been promoted recently as a soil amendment due to its potential for amelioration of soil degradation as well as

increasing soil fertility (Jeffery et al., 2015). As a water treatment mechanism, activated carbon filtration is used in wastewater treatment (Tchobanoglous et al., 2003); however, charcoal that has not been activated has also been used for adsorbing NH_4-N in wastewater (Vassileva et al., 2009) as well as in animal manure effluents (Sarkhot et al., 2013).

Clinoptilolite is a natural zeolite, an aluminosilicate mineral with a porous structure, and can be used as filler material in artificial fertiliser production (Pawełczyk and Popowicz, 2006) as well as directly as a soil amendment with an ability to act as a slow release fertiliser, improve soil performance and increase crop yields (Malekian et al., 2011; Lija et al., 2012; Aainaa et al., 2014). Clinoptilolite has a highly porous structure and high affinity for the ammonium ion (Demir et al., 2002). However, using clinoptilolite to recover ammonium from high strength liquor streams may be challenging due to high concentration of organic matter and competing cations which impact the exchange of the ammonium ion (Carley and Mavinic, 1991; Wang et al., 2007; Huang et al., 2010). Most researchers have investigated the potential use of clinoptilolite to remove NH_4-N from low strength wastewaters such as the final effluent of wastewater treatment works (Metropoulos et al., 1993; Baykal and Guven, 1997; Cooney et al., 1999; Wang and Peng, 2010). These wastewaters have low NH_4-N, total solids, organic matter and cationic ion concentrations, that are all likely to interrupt the ion exchange process; however, the use of clinoptilolite to directly capture NH_4-N from high strength wastewaters with this interference, such as in high NH_4-N concentration liquors, has not previously been investigated.

The aim of this study is to assess the feasibility of using a passive system for the recovery of NH_4-N from anaerobic digestate liquors (that have high NH_4-N concentrations) utilising non-regenerative media (biochar and clinoptilolite) that can be used directly as an enhanced fertiliser product once exhaustion has been reached. The design values for scale-up procedures will be established with regards to the main operational parameters for passive bed operation; particle size, empty bed contact time (EBCT), bed depth and influent NH_4-N concentration. Specifically, the objectives are to a) select an appropriate media for the recovery of NH_4-N from anaerobic digestate liquors through the use of batch adsorption and kinetic studies; b) quantify the impact of high concentrations of organic matter and competing

cationic ions present in sludge liquors; c) establish, in continual operational mode, the optimum operational capacity of a down-flow fixed media bed with regards to empty bed contact time, influent NH_4-N concentration, particle size and bed depth when treating digestate liquors.

6.2 MATERIALS AND METHODS

6.2.1 Media and solutions used in batch and dynamic experimental work

6.2.1.1 Non-regenerative media used

Silica sand (Garside Sands, Leighton Buzzard, UK) was utilised as a baseline comparator in the study with a small (0.63-0.85 mm) and large (2-2.7 mm) particle size selected. Clinoptilolite (supplied by RS Minerals Ltd., Cleveland, UK) was screened into two fractions of 0.7-1.6 mm and 5-9 mm. The clinoptilolite used had 92% purity with other clay minerals (5% smectite and 3% biotite) present. Clinoptilolite had a pH of 6.5-7.5, bulk density of 1.23 g/cm^3, real density of 2.25 g/cm^3, porosity of 84.5% and a specific surface area of 41 m^2/g. A deciduous mixed wood biochar (Biochar Foundation, Loanhead, UK) was used with production carried out according to Ulyett et al. (2014). The biochar had a pH of 10.2, carbon: nitrogen ratio of 117 and a cation exchange capacity of 66 $cmol^+$ kg^{-1} (Ulyett et al., 2014). The char was sieved to fractions of 5.4-6 mm and 1-2 mm. Before use in experiments, all media was rinsed with deionised water to remove impurities, before drying at 103°C.

Batch and kinetic experimental work was carried out on sand, biochar and clinoptilolite and a small and large particle size of each medium was selected for comparative purposes. Dynamic experimental work was carried out on the clinoptilolite medium only.

6.2.1.2 Competing cationic ions and organic matter solutions

A wastewater solution containing a high concentration of competing cations was deemed appropriate for a wide range of studies as significant concentrations are normally present when NH_4-N concentrations are high: for example, Karadag et al. (2008) reported concentrations of 3328 Na^+, 1785 K^+, 220 Mg^{2+} and 36 Ca^{2+} in landfill leachate and similarly high concentrations are found in digestate liquor streams (Thornton et al., 2007). In order to replicate the complex nature of high

strength liquors a solution of competing cationic ions was created to replicate concentrations as measured in effluent digestate (Table 6.1). Digestate was from a mesophilic anaerobic digester treating a mixture of primary and waste activated sewage sludge (population equivalent 288000). The synthetic solution was constructed using calcium chloride dehydrate, magnesium sulphate heptahydrate, sodium chloride and potassium chloride acquired from Fischer Scientific (Loughborough, UK). Organic matter (7675 mg COD L^{-1}) was simulated using D-Glucose monohydrate (VWR International, Lutterworth, UK) and the concentration of NH_4-N was adjusted using a concentrated stock solution of ammonium chloride (NH_4Cl) (Fischer Scientific, Loughborough, UK).

Table 6.1 Composition of digested sewage sludge from a mesophillic anaerobic digester and the synthetic solution of competing cations and organic matter created to replicate digestate leachate without the interference of solid matter.

	Anaerobic Digestate (mg TS L^{-1})	1/10 dilution Sludge (mg TS L^{-1})	Synthetic AD leachate (mg TS L^{-1})
Total Solids (TS)	51000	5000	
Ammonium (NH_4-N)	2060	206	200
Calcium (Ca)	533	53.3	53.4
Magnesium (Mg)	450	45.0	44.9
Sodium (Na)	858	85.8	86.5
Potassium (K)	2775	277.5	277
Chemical oxygen demand (COD)	80000	8000	7995

6.2.2 Experimental setup and operation

6.2.2.1 Batch adsorption isotherms and kinetic studies

In all batch experiments 1 g of media was placed in 250 mL Erlenmeyer flasks and equilibrated with 100 mL of NH_4-N solution of varying concentration (10-400 mg NH_4-N L^{-1}) on an orbital shaker at 125 revolutions min^{-1} (Stuart Orbital Shaker, Bibby Scientific Ltd., Stafford). All experiments were carried out at room temperature (20 ± 1°C) with a contact time of 24 hours, deemed suitable after preliminary tests. In order to determine the maximum NH_4-N capacity of the media NH_4-N solutions were made

up of laboratory grade NH_4Cl (Fischer Scientific, Loughborough, UK) and deionized water. In order to determine the NH_4-N adsorption kinetics of the media, samples (0.1 mL) were taken at regular time intervals throughout the 24 h period (0.5, 1, 1.5, 2, 4, 6, 0, 14, 20, 22, 24 h). The NH_4-Ncapacity and removal efficiency were calculated according to equation 1 and 2:

$$Qe = \frac{(C_0 - C_e)\,V}{m} \tag{1}$$

$$RE = \frac{(C_0 - C_e)}{C_0} \cdot 100 \tag{2}$$

where Q_e is the ammonium adsorption capacity at equilibrium (mg/g), RE the removal efficiency (%), C_0 and C_e the initial and equilibrium concentrations (mg L^{-1}), respectively, V the volume of the solution (L) and m the mass of media (g).

6.2.2.2 Dynamic column studies

In order to obtain the maximum fertiliser value from the media all operational column experiments were operated until exhaustion, where column effluent concentration was ≥ 95% of column influent NH_4-N concentration (C_e NH_4-N ≥ 95% of C_0 NH_4-N). The operational capacity of clinoptilolite was determined through investigations into empty bed contact time (EBCT), variations of influent NH_4-N concentration and media bed depth. A column (inside diameter: 0.05 m, length: 0.7 m) was used with clinoptilolite (particle size: 0.7-1.3 mm) and a synthetic solution replicating digestate without the interference of solid matter (Table 6.1).

6.2.2.3 Effect of empty bed contact time

The effect of EBCT (bed volume/volumetric flow) on the ability of clinoptilolite to capture NH_4-N was investigated using 500 g of clinoptilolite, giving a fixed bed height of 0.33 m (bed volume of 590 cm^3). A synthetic digestate liquor recipe was used with a 200 mg NH_4-N L^{-1} concentration (Table 6.1). Each experimental run was ended following exhaustion of the media (C_e NH_4-N ≥ 95% of C_i NH_4-N). Columns were gravity fed using a peristaltic pump (Watson Marlow, Falmouth, UK) at 13 different EBCT giving a range of 20 – 354 mins with individual experimental periods ranging from 1-65 days before exhaustion of the media was complete. The risk of

channelling within the profile of the column was minimised in all column experiments due to the ratio of column diameter: media particle size being greater than 20 according to Martin et al. (2013). A mesh (0.5 mm) was used to prevent media loss from the base of the column.

6.2.2.4 Continuous and batch fed operation of clinoptilolite beds

The performance of clinoptilolite was investigated in two modes of operation: continuous and batch-fed. Two EBCT of 40 and 80 mins were selected with continuous operation (as of section 2.3.2) with batch-fed operation carried out by complete loading of the allotted volume and restricting the drainage to give two comparative EBCT.

6.2.2.5 Operational capacity of media at different NH_4-N concentrations

Utilising one single bed depth (0.33 m) and EBCT (80 mins) the effect of NH_4-N uptake by clinoptilolite at varying influent concentrations was assessed. Six different NH_4-Nconcentrations (20, 60, 80, 100, 150, 200 mg NH_4-N L^{-1}) were used in order to establish the effect of NH_4-N concentration on media operational capacity. These NH_4-N concentrations were selected in order to represent a range in values ranging from zero to a maximum of 200 mg NH_4-N L^{-1} commonly seen in low strength or diluted anaerobic liquors (Rose et al. 2015). Nonlinear regression analysis with groups was used to compare data that fitted an asymptotic curve (or exponential curve). The analysis had a corrected r^2 (percentage variation accounted for) value of 95.8% with a pooled standard error of observations = 0.0745 (C_i/C_o units). For each different NH_4-N concentration a rate constant was calculated (the rate that the slope reaches the asymptote). Exponential rate constants are presented with standard error and are presented for each NH_4-N concentration.

6.2.2.6 Operational capacity of media at different bed depths

In order to establish the effect of bed depth on NH_4-N uptake capacities, a total of 6 different bed heights (0.11 - 0.66 m) were assessed (media volumes 230 – 1400 cm^3). The same EBCT (80 mins) was maintained for each different bed height and resultant media volume. The media utilisation rate, the mass of media required in order to remove a defined mass of NH_4-N from m^3 of liquor with a concentration of

200 mg/L NH$_4$-N was subsequently calculated to the point of exhaustion (kg media/m$^3_{treated}$).

6.2.2.7 Determining operational column capacity

Effluent of the columns was collected at regular time intervals until the media reached exhaustion (C$_e$ NH$_4$-N ≥ 95% of C$_0$ NH$_4$-N). The NH$_4$-N adsorption capacity and removal efficiency of clinoptilolite was determined according to equations 3 and 4:

$$C_{total} = \int_{V=0}^{V=V_{total}} (C_0 - C_e)$$
$$\cdot \, dV \tag{3}$$

$$RE = \frac{C_{total}}{C_{fed} \cdot V_{total}} \cdot 100 \tag{4}$$

where C$_{total}$ is the total NH$_4$-N adsorbed in the column (mg), Q$_{fed}$ the total NH$_4$-N fed (mg L^{-1}), RE the removal capacity (%), C_0 and C_e are the ammonium concentrations of the influent and effluent (mg L^{-1}), respectively, V$_{total}$ the solution volume fed (L).

The fraction of unused bed length (LUB) is a measure of the unused capacity of the bed if the adsorption process was to be stopped at the point of breakthrough (0.05% C$_0$) (Cooney et al., 1999). This was determined according to equation 5:

$$LUB = 1 - \frac{tb}{te} \tag{5}$$

Where t$_b$ is the time until breakthrough (5% of influent concentration) and t$_e$ is the time until exhaustion (95% of influent concentration).

In order to establish whether different column configurations had an impact on the flow dynamics through the fixed media bed, the Reynolds number was calculated according to the method used by Martin et al. (2013) as described in equation 6:

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$$Re = \frac{\rho V D}{\mu(1 - \varepsilon)}$$ (6)

Where ρ is the density of the water, V is the superficial velocity of the feed solution through the bed, D is the particle size of the media, μ is the dynamic viscosity of the water (0.001 Pa s) and ε is the void fraction (0.11 as determined in the used media through the methodology of Kleinübing and Da Silva (2008)).

6.2.3 Analytical procedures

In the batch adsorption and kinetic testing, NH_4-N concentrations were determined after filtration through 0.45 μm microporous membrane filters (Sartorius, Epsom, UK) before analysis by a Burkard SFA-2000 auto-analyser (Burkard Scientific Ltd., Uxbridge, UK) according to the Automated Phenate Method (APHA, 2005). In all dynamic column experiments analysis was carried out in duplicate: ammonium-nitrogen (NH_4-N), magnesium (Mg), potassium (K) and phosphorus (P) were all determined photometrically using Spectroquant Nova 60 (Merck-Millipore, Darnstradt, Germany). The soluble solid free fraction of the digestate was also used for the analysis of calcium (Ca) and sodium (Na) by Atomic Absorption Spectrophotometry (Shimadzu 6300, Shimadzu, Japan). The pH of samples and solutions was measured directly by pH meter (model 3540, Jenway, Dunmow, UK).

6.3 RESULTS AND DISCUSSION

6.3.1 Media Selection

6.3.1.1 Batch adsorption Isotherms and Kinetics

Three adsorption isotherm models (Langmuir, Freundlich and Tempkin) are applied to the data (Table 6.2) with the Langmuir model proving the best fit (r^2=0.945) for clinoptilolite (0.7-1.6mm), which suggests that NH_4-N adsorption onto the clinoptilolite is more like a monolayer adsorption process and that the clinoptilolite presents homogenous sites for ammonium adsorption and a uniform distribution of energetic adsorption sites (Widiastuti et al., 2011). The Psuedo-first-order and Psuedo-second-order models were used to model the kinetics of the NH_4-N uptake of the clinoptilolite with the Psuedo-second-order model providing the best fit

(r^2=0.997) over the entire kinetic period (Table 6.2). This indicated that a pseudo-second-order reaction was predominant and that chemisorption controlled the adsorption process (Kučić et al., 2012). The ideal maximum adsorption capacity (Q_L) of clinoptilolite was 14.1 mg/g, which was higher than those reported by Zhang (2012) (10.5 mg/g) and Sarioglu (2005) (12.5 mg/g). This factor is likely to be due to differences in the chemical composition of the media, for instance impurities in the clinoptilolite, with a high (92%) purity used in this study.

At a concentration of 200 mg NH_4-N L^{-1}, Clinoptilolite with the smaller particle size (0.7-1.6 mm) had both a greater capacity (12.2 mg/g) as well as a faster kinetic rate (1.013 h^{-1}) in comparison to biochar with a similar particle size (1-2 mm), exhibiting comparative kinetic uptake rates of 5 mg/g and a reduced kinetic rate of 0.557 h^{-1} (Table 6.3). The greater performance of clinoptilolite was anticipated due to its high selectivity for the ammonium ion (Demir et al., 2002) and hydrophilic surface, nevertheless, biochar did capture NH_4-N with adsorption over double that of a silica sand which represented a control in the study (2 mg/g). The adsorption capacity of clinoptilolite was influenced by particle size of the media with a 25% reduction in NH_4-N adsorption capacity when particle size is increased from 0.7-1.6 mm to 7-9 mm. Similarly, Sprynskyy et al. (2005) reported a 14% reduction in the uptake capacity of clinoptilolite when particle size fractions were increased from 0.5-0.7 to 1.4-2 mm. The reduction in capacity is frequently accounted for by increases in the external surface area of clinoptilolite particles (for instance in this study external surface area increased from 0.029 m^2 g^{-1} to 7.45 m^2 g^{-1} media when particle size fractions decreased from 8- 1.2 mm). However, when particle size is reduced this does not reduce internal surface area which is not considerably impacted except for minor changes due to the opening of clogged pores in the clinoptilolite's structure which could account for the increases in capacity observed (Erdoğan and Ülkü, 2011). The faster kinetic uptake rate of the smaller particle size (0.725 h^{-1}) indicates an increased initial uptake which is likely to be due to the larger external surface area exposed to cations in the solute. However, over time the saturation of external sites results in the predominant mechanism becoming internal mass transfer, as expected from experimental isotherms (Table 6.1). This indicates that the clinoptilolite has a high degree of internal porosity, a factor similarly observed by

Hankins et al. (2005a). Therefore, it is not the external surface area that is the principle driver but the diffusion of water molecules and cations through the clinoptilolite framework. Therefore, the effect of particle size is more apparent when establishing the kinetic rate of NH_4-N uptake, with the quicker rate of uptake in the smaller particle size in comparison to the larger clinoptilolite particle size (0.323 h^{-1} and 1.013 h^{-1} respectively).

6.3.1.2 Influence of anaerobic liquors on uptake capacity

The presence of competing cations in the solution caused a 23.8% reduction in the uptake capacity of clinoptilolite (0.7-1.6 mm) due to increased competition for exchange sites on the media (Table 6.3). This reduction was greater with a 34.7% reduction in uptake capacity when a larger clinoptilolite particle size (7-9 mm) was used. This is a reflection of the reduced external surface area of the larger particle size (7.45 m^2/g and 0.029 m^2/g media in the small and large particle size respectively), resulting in fewer sorption sites available for exchange, meaning that there is greater competition for these sorption sites between competing cations. In a similar study detailing the effect of competing cations on clinoptilolite, Wang et al. (2006) reported a reduction in capacity of 10-20%. The reduction in capacity of clinoptilolite due to the presence of competing cations is also consistent with the results of others (Nguyen and Tanner, 1998; Wang et al., 2007; Huang et al., 2010). This is a factor of paramount importance when applying batch adsorption data to an applied context, such as passive removal in high NH_4-N concentration waste streams in which equally high competing cations will be present. The addition of competing cations on biochar's uptake capacity was greatest when the particle size was greatest (Table 6.3); similarly, this is likely a result of decreased external surface area meaning increased competition for sorption sites by other cations attaching to the biochar surface.

Table 6.2 Batch adsorption isotherms and kinetic modelling for clinoptilolite, biochar and sand using a solution of NH₄Cl and a solution of NH₄Cl mixed with competing cations (equal to anaerobic digestate) and organic matter (8000 mg/L COD).

Media	Zeolite						Char						Sand	
Particle size	*Large (7-9 mm)*			*Small (0.7-1.6 mm)*			*Large (5.4-6 mm)*			*Small (1-2 mm)*			*Large 1.2-2.4mm*	*Small 2.4-4 mm*
Solution	NH_4-N	NH_4-N cations[a]	NH_4-N organic[b]	NH_4-N	NH_4-N cation[a]	NH_4-N organic[b]	NH_4-N	NH_4-N cations[a]	NH_4-N organic[b]	NH_4-N	NH_4-N cations[a]	NH_4-N organic[b]	NH_4-N	NH_4-N
Freundlich model														
Kf	0.772	0.195	0.779	1.187	0.158	0.856	0.189	0.038	0.144	0.189	0.033	0.150	0.038	0.048
1/n	0.469	0.598	0.459	0.432	0.704	0.510	0.527	0.649	0.574	0.571	0.855	0.614	0.719	0.676
R2	0.928	0.981	0.962	0.956	0.964	0.971	0.920	0.908	0.947	0.972	0.996	0.959	0.706	0.708
HYBRID	22.403	4.459	9.616	16.977	11.629	13.723	13.51	5.496	11.877	7.984	0.945	10.013	24.086	25.421
Langmuir model														
Qm	10.195	7.307	9.801	11.444	16.532	14.107	3.949	2.609	4.329	5.700	14.326	6.086	3.590	3.048
aL	0.029	0.009	0.026	0.043	0.004	0.022	0.016	0.004	0.012	0.011	0.001	0.009	0.005	0.008
R2	0.980	0.930	0.952	0.957	0.948	0.953	0.875	0.863	0.913	0.947	0.994	0.950	0.855	0.850
HYBRID	3.660	7.851	9.095	8.985	17.100	13.115	10.68	7.055	8.961	6.659	0.740	6.190	14.340	14.411
Tempkin model														
b	1.286	2.297	1.621	1.274	1.900	1.180	3.048	7.955	3.001	2.498	3.346	2.369	4.241	4.089
K	0.426	0.212	0.656	0.870	0.225	0.596	0.201	0.153	0.173	0.193	0.118	0.162	0.103	0.116
R2	0.989	0.877	0.936	0.971	0.765	0.923	0.907	0.751	0.921	0.933	0.826	0.943	0.942	0.939
HYBRID	2.442	11.664	20.784	10.600	44.921	28.110	6.940	7.961	5.572	5.376	22.257	4.550	12.317	2.865

KINETICS

First order model

k1	0.305	0.239	0.585	0.633	0.927	1.386	1.589	0.593	0.527	0.701	0.383	0.632	0.608	0.502
R^2	0.951	0.962	0.854	0.848	0.781	0.749	0.917	0.781	0.843	0.634	0.956	0.623	0.724	0.943
HYBRID	12.675	6.355	16.991	17.212	8.167	9.852	0.897	3.956	7.755	13.89	3.086	15.866	7.055	2.152

Second order model

k2	0.400	0.323	0.609	0.725	1.013	1.524	3.497	0.518	0.483	0.464	0.557	0.377	0.453	0.680
$\beta 2$	1.148	1.099	1.496	1.496	1.723	2.062	0.861	1.623	1.460	1.948	1.090	1.924	1.601	1.198
R^2	0.978	0.972	0.977	0.962	0.961	0.953	0.984	0.954	0.959	0.937	0.942	0.928	0.875	0.976
HYBRID	3.969	2.436	1.629	3.182	0.793	0.714	0.062	0.381	0.587	1.162	3.654	1.253	1.155	0.662

[a] Competing cation solution constructed to give a concentration of 53.4 mg/L Ca, 44.9 mg/L Mg, 86.5 mg/L Na, 277 mg/L K as measured in anaerobic digestate.
[b] Concentration of organics constructed using glucose monohydrate to give a COD concentration of 8000 mg/L.

High organic loading (8000 mg COD L^{-1}) is also present in high strength liquor streams (Table 6.1) and caused a slight increase in uptake capacity in biochar (Table 6.3). A more profound increase of 13.9% was observed under high COD concentrations in clinoptilolite (0.7-1.6 mm). An increase in NH_4-N uptake capacity was also observed with the addition of glucose by Semmens et al. (1981). This could be accounted for by a reduction in surface tension caused by the organics and/or the adsorption of NH_4-N to organic matter in the solution or attached to the media surface, providing alternative exchangeable sites, as illustrated by Muherei and Junin (2009). However, the clinoptilolite with a larger particle size (7-9 mm) was not impacted by high COD loading (1% reduction in capacity) which indicates that when the surface area of the media is reduced then there is more competition for sorption sites and this is the predominant mechanism. An additional factor that may have caused an increase in NH_4-N uptake across all media types is the use of glucose as a method of increasing COD concentrations in the experiments. The presence of glucose in the solution gives increased availability of carbon, providing surfaces within the solution in which nitrifying bacteria can grow (Carley and Mavinic, 1991). This resultantly reduces the NH_4-N concentrations remaining in the solution following the completion of batch experiments.

Table 6.3 Summary table reporting the theoretical capacity of media through batch adsorption isotherms and kinetics studies of NH_4-N uptake of sand, char and clinoptilolite at a concentration of 200 mg NH_4-N L^{-1}.

Media	Particle size (mm)	Capacity at 200 mg NH_4-N L^{-1} Q_e (mg/g)			Impact on uptake capacity (%)		Kinetic rate of adsorption (k_2) (h^{-1})		
		NH_4-N only	NH_4-N and cations[a]	NH_4-N and organics[b]	Addition of competing cations[a]	Addition of Organic loading[b]	NH_4-N only	NH_4-N and cations[a]	NH_4-N and organics[b]
Clinoptilolite	0.7-1.6	12.2	9.3	13.9	23.8 (-)	13.9 (+)	0.725	1.013	1.524
	7-9	9.8	6.4	9.9	34.7 (-)	1.0 (+)	0.400	0.323	0.609
Biochar	1-2	5.0	4.8	5.1	4.0 (-)	2.0 (+)	0.464	0.557	0.377
	5.4-6	4.0	1.9	4.0	52.5 (-)	0.0 (+)	3.497	0.518	0.483
Silica Sand	1.2-2.4	2.0	-	-	-	-	0.453	-	-
	2.4-4	2.0	-	-	-	-	0.680	-	-

[a] Competing cation solution (Ca, Na, K, Mg) created to replicate the high concentrations of competing cations found in anaerobic digestion effluents.
[b] High organic loading (8000 mg COD L^{-1}) in anaerobic digestion effluents were replicated by the addition of glucose.

6.3.2 Operational capacity in dynamic experiments

Clinoptilolite had a 2.4 fold superior capacity in comparison to biochar under batch experimental conditions and was more suited to the recovery of NH_4-N under challenging conditions presented in digestate liquors (200 mg NH_4-N L^{-1} , 8000 mg COD l^{-1} and competing cations). For this reason clinoptilolite was selected for further experimental work into establishing design parameters.

6.3.2.1 Empty Bed Contact Time

Breakthrough curves at all EBCT had an idealised (S) shape (Figure 6.1), indicating that bed packing and hydraulic flow rates were sufficient and that channelling was not likely to be occurring. It is evident that as EBCT is increased the exhaustion curve becomes more drawn-out indicating greater sorption capacity by the biochar (Figure 6.1). Both the total bed volumes treated to breakthrough (5% C_0) and exhaustion (95% C_0) had a weak linear relationship to EBCT (r = 0.703, p = 0.011 and r = 0.836, p = <0.001 respectively) (Figure 6.2). The fewer number of bed volumes until exhaustion at low EBCT (Figure 6.1) is a result of NH_4-N ions flowing through the column before the complete exchange of ions with clinoptilolite is complete and was similarly observed by Demir et al. (2002) and Karadag et al. (2008). As a result, the total uptake of NH_4-N by clinoptilolite was also positively correlated to EBCT (r = 0.899, p = < 0.001) with the capacity ranging from 5-60 g/kg dependent on the EBCT (Figure 6.3). The dramatic increase in media capacity was a result of increasing EBCT and was similarly observed by Sarioglu et al. (2005a) who reported an increase in clinoptilolite capacity by 248% when EBCT was increased from 9.8 to 38 mins. This was again attributed to contact time.
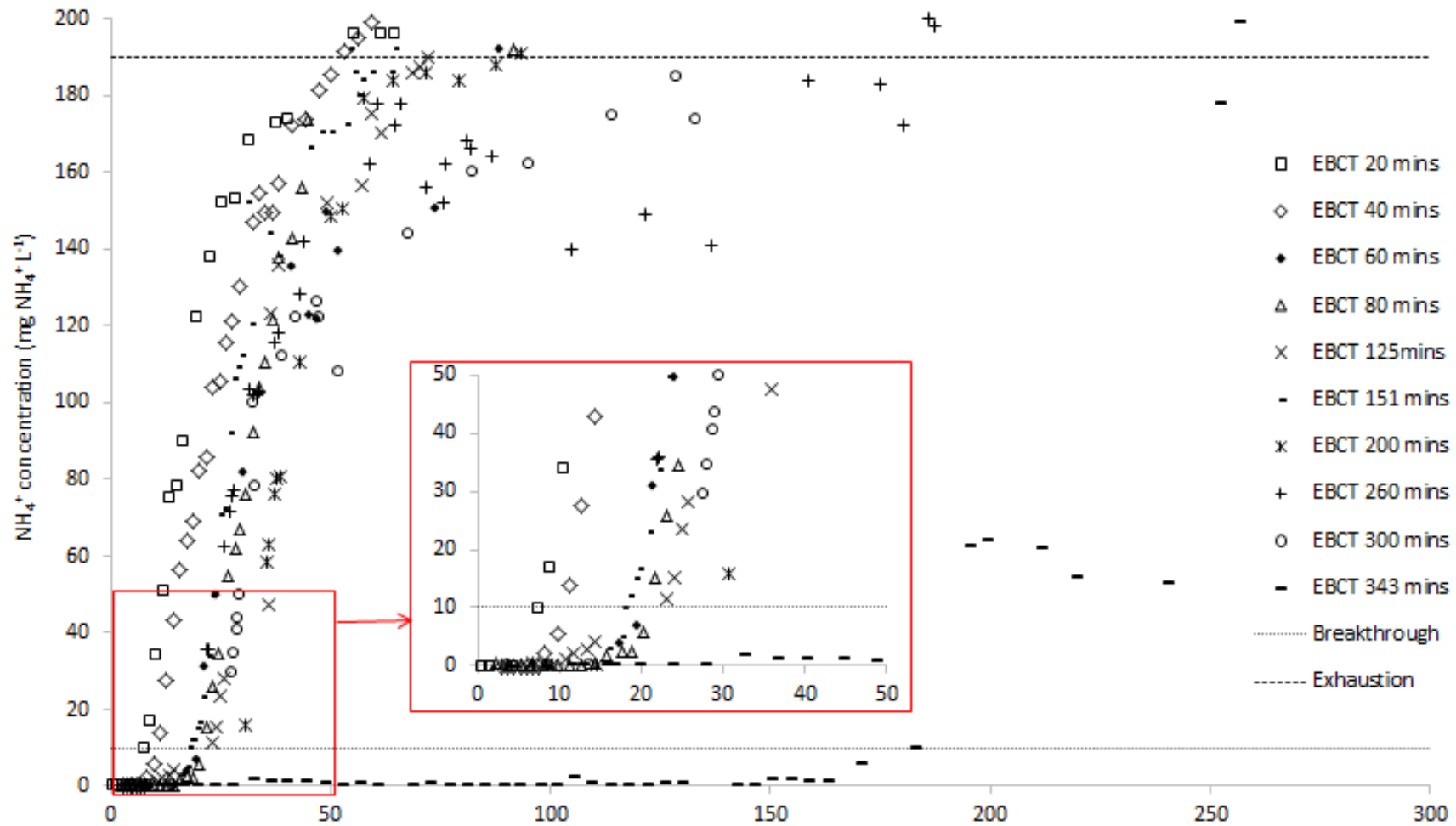
Figure 6.1 Exhaustion curves for clinoptilolite (0.7-1.6 mm) with a bed depth of 0.33m and a synthetic AD liquor with an influent concentration of 200 mg NH$_4$-N L^{-1}.
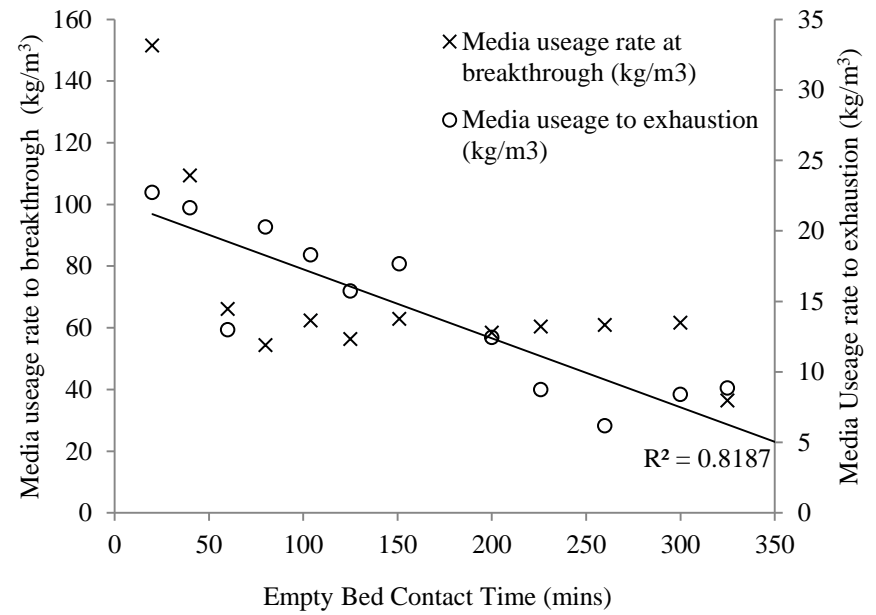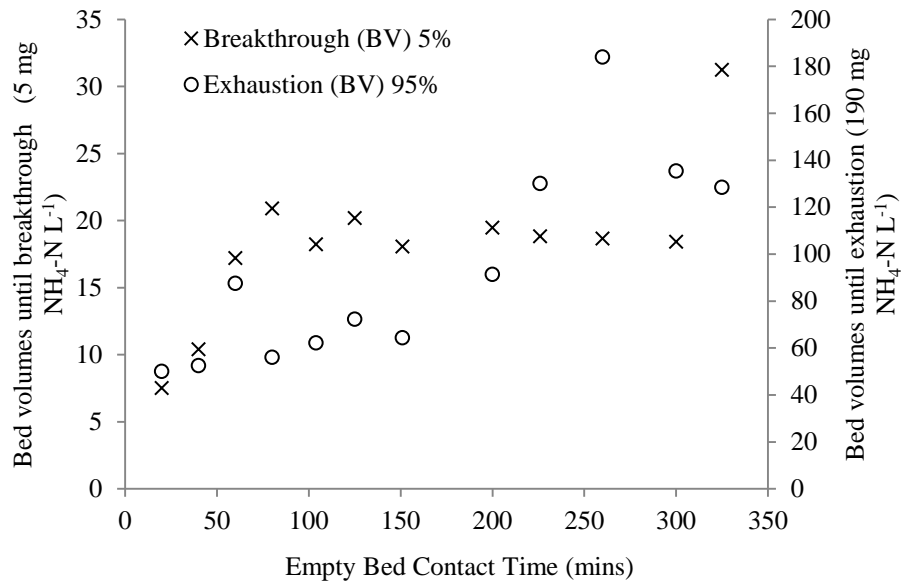
Figure 6.2 a) The effect of changing EBCT on breakthrough (C$_e$ 5% C$_0$) and exhaustion (C$_e$ 95% of C$_0$) of the media at 200 mg NH$_4$-N L^{-1} synthetic no solids solution.

This subsequently meant that the media usage rate was reduced when operating at longer EBCT (Figure 6.2); for instance at the shortest EBCT (20 mins) the media usage rate at exhaustion was 23 kg/m^3 wastewater treated, however, at the longest EBCT (354 mins) the media utilisation rate was vastly reduced to 4 kg/m$^3_{treated}$. Therefore operation at an extended EBCT would be beneficial in terms of extending service time as well as ensuring maximum efficiency (60 g NH$_4$-N /kg clinoptilolite). However, the media utilisation rate in relation to EBCT (Figure 6.2) indicates that at an EBCT of ≥226 mins, the media utilisation rate flattens and the marginal benefit of operating at a longer EBCT is minimal in regards to maximising media efficiency. It was similarly observed by Jorgensen et al. (1976) that capacity increased with EBCT up to 120 mins, however this study was carried out at a reduced NH$_4$-N concentration. There are limited studies that use long EBCT, as used in this study, most likely due to the intended application in high intensity configurations utilising regeneration, consequently there is a paucity of comparative literature values at these extended contact times.
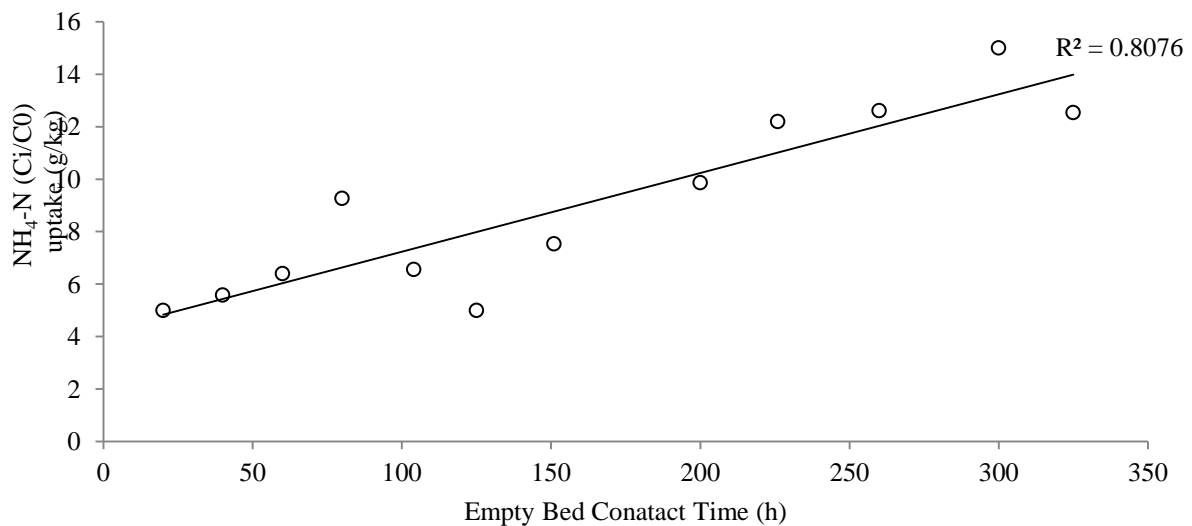
Figure 6.3 The effect of increasing EBCT on the NH$_4$-N uptake of clinoptilolite when operated with a bed depth of 0.33m and a particle size range of 0.7-1.6 mm

6.3.2.2 Continuous and batch fed operation of clinoptilolite bed

Minimal difference was observed in regards to the operational feed mode. Batch fed operation yielded slightly increased NH_4-N uptake at both a 40 min EBCT (5.59 g NH_4-N/kg vs 5.69 g NH_4-N/kg) and 80 min EBCT (9.27 NH_4-N/kg vs 9.62 NH_4-N/kg). It is therefore likely that the effect of the feed regime is minimal at the range of EBCT operated at. The slight increase in NH_4-N uptake by the media when operated in batch mode, can be attributed to greater turbulence and mixing as a result of batch fed loading, leading to improved mixing and contact between the solution and the media.

6.3.2.3 Bed Depth

The most efficient bed depth in regards to media utilisation rate is 0.11 m (Table 6.4), this is primarily due to the reduced mass of media used (10.387 kg/m^2) in comparison to that of deeper bed depths of 0.55-0.66 m (44.539 and 52.880 kg/m^2 respectively). Increasing bed height by 6 fold, increased the total uptake of the bed by 28% (Table 6.4), this was similarly the case in a study by Karadag et al. (2008) who reported an increase in 5% exchange capacity as a result of a 2 fold increase in bed height. This was unexpected as an increase in bed depth would provide an increase in the number of exchange sites, which should increase ammonium uptake. Others have investigated the influence of increasing bed depth without maintaining a consistent EBCT. For instance, Mashal et al. (2014) found that increasing bed depth from 0.1 to 0.4 m caused an increase in exhaustion times from 90 – 340 mins, however, hydraulic flow rates were not adjusted to maintain constant EBCT in this study and as EBCT is one of the key variants in capacity, comparison to these results is not appropriate.

The reason for a limited increase in exchange capacity in this study could be accounted for by the increase in flow rate to maintain a consistent EBCT throughout all experiments. This would have caused irregularities in flow pattern at different bed depths and may not have allowed constant pattern behaviour to occur. However, a ratio of bed depth to column diameter of at least 2:1 was maintained and a Reynolds's number of <10 was not reached at any bed depth (calculated Reynold's number range of 0.030 to 0.178) which limits the effect of these factors on

experimental results (Martin et al., 2013). Nevertheless, the results indicate that bed depth does impact media uptake with a range of 5.52-22.25 g NH_4-N/kg media (Table 6.4). However, the desired service time as well as the pressure drop within the bed is likely to predominate over the efficiency of the media at different bed depths (4.05-11.89 kg/m^{3}treated) (Table 6.4). Only a true economic analysis of the media cost, service cost and final market value of the fertiliser product will be able to determine the most efficient bed depth.

Table 6.4 The effect of bed depth (0.11-0.66 m) on clinoptilolite performance when hydraulic flow rate is adjusted in order to maintain a consistent EBCT (80 mins). The media utilisation rate, the mass of media required in order to remove NH_4-N mass per m^3 of influent with a concentration of 200 mg/L NH_4-N was subsequently calculated to the point of exhaustion (kg media/m^{3}treated).

Bed depth	BVs to breakthrough	BVs to exhaustion	NH_4-N captured (mg)	Media utilisation rate (kg NH_4-N/m^3)
0.11	16.93	67.00	4537	4.05
0.22	14.32	60.71	3616	7.89
0.33	20.90	56.04	3282	11.89
0.44	28.96	102.56	3702	8.71
0.55	12.62	164.93	5424	7.06
0.66	12.59	188.12	5821	7.35

6.3.2.4 Influent ammonium concentration

The exhaustion curves with different influent NH_4-N concentrations are illustrated in Figure 6.4. When the influent NH_4-N concentration is decreased, the exhaustion curve is shifted to the right, indicating an increased sorption capacity (Figure 6.4). It is also evident that exhaustion becomes less steep and vertical and becomes increasingly horizontal as the influent NH_4-N concentration is decreased (Figure 6.4). This is also evident that as NH_4-N concentration is increased, the rate of reaching the asymptote gets faster, that is, the rate constant gets smaller (reduction from 1.00133 (standard error.0.000474) at 60 mg NH_4-N L^{-1} to 0.98537 (standard error 0.00237) at 200 mg NH_4-N L^{-1}: Figure 6.4).This indicates that NH_4-N adsorption at high concentrations is faster and exhaustion is likely to be reached quicker. This

finding corresponds to theory, as it would be expected that the higher the concentration of NH_4-N in the influent solution, the higher the solute gradient will be, meaning that it will provide the necessary driving force for replacement of cations by NH_4-N ions within the same given contact time (Du et al., 2005). For instance, NH_4-N uptake in a dynamic study by Dryden and Weatherley (1989) increased linearly with influent concentration, although this was carried out at lower concentrations and at a reduced range (1-5 mg NH_4-N L^{-1}) in comparison to this study (20-200 mg NH_4-N L^{-1}). Similarly, Mashal et al. (2014) observed an exhaustion curve that shifted to the right with increasing NH_4-N concentrations (ranging from 15-50 NH_4-N L^{-1}). However, in dynamic experiments carried out by Karadag et al. (2008) only a slight drop in performance was measured when influent concentration was increased from 100-200 mg NH_4-N L^{-1}, but, when concentrations were increased to 400 mg NH_4-N L^{-1}, a sharp decrease in bed volumes to exhaustion was recorded.

An increase in influent NH_4-N concentrations led to an increase in the LUB fraction from 0.38 at 20 mg NH_4-N L^{-1} to 0.94 with an influent concentration of 200 mg NH_4-N L^{-1}. This demonstrates that a change in the concentration gradient affects the saturation rate of the clinoptilolite meaning that the diffusion process is concentration dependent. Therefore, as influent concentration increases, so does the driving force for mass transfer, causing the LUB fraction to increase (Barros et al., 2013).
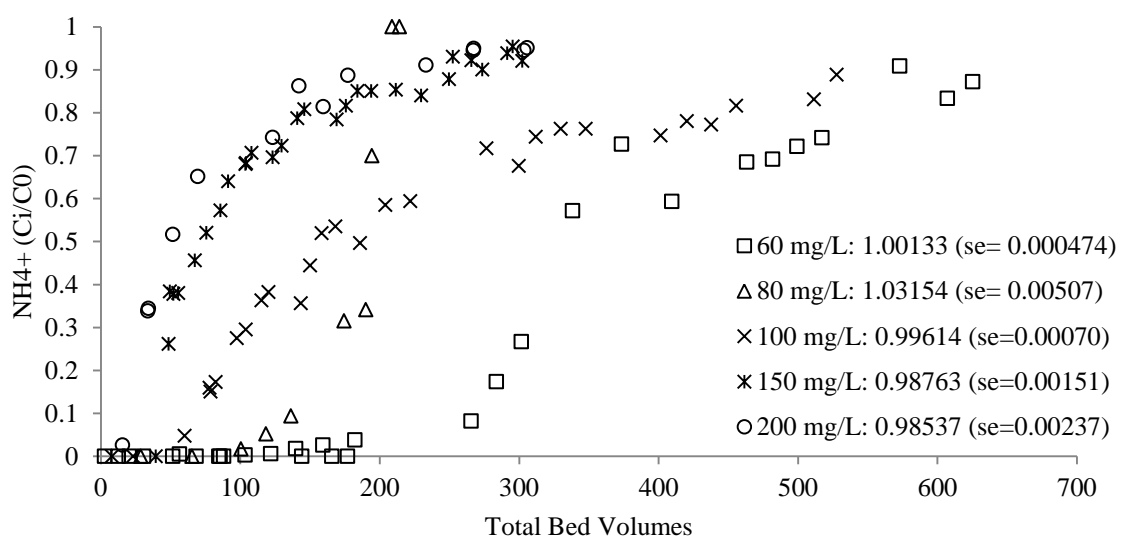
Figure 6.4 The effect of increasing influent ammonium concentration (60-200 mg NH$_4$-N L^{-1}) on exhaustion curve shape in a dynamic continuous flow operation with a bed depth of 0.33 m and an EBCT of 80 minutes. Legend details exponential rate constant and standard error for each different NH$_4$-N L^{-1} concentration group.

6.3.3 Structural integrity of media

Clinoptilolite experienced structural disintegration during dynamic experiments, with exhausted media displaying greater fracturing and signs of erosion in comparison to unused washed clinoptilolite (Figure 6.5). The greater pore depth and favourable adsorption characteristics depicted in the saturated clinoptilolite (Figure 6.5) can most likely be attributed to the presence of dust and fines on the unused media which is subsequently removed following saturation. The susceptibility to disintegration of clinoptilolite's structural aggregates after wetting has also been reported by Nguyen and Tanner (1998). Disintegration of clinoptilolite during continuous flow operation causes increased amounts of pores to appear in the media (Figure 6.5), which has the benefit of increasing surface area for exchange sites which could be a contributing reason for increased capacity at greater EBCT (Figure 6.2). This factor was observed by Huo et al. (2012) as a result of pre-treatment of clinoptilolite leading to increased NH$_4$-N uptake. However, media disintegration also has the detrimental effect of causing clogging as particle dust and fines fill media voids and an inhibitory pressure drop within the media bed could be created. This factor is likely to be increasingly problematic when media is being regenerated and used over multiple cycles, and further promotes the use of clinoptilolite as a non-regenerative single use media.

Before clinoptilolite exhaustion   *After clinoptilolite exhaustion*
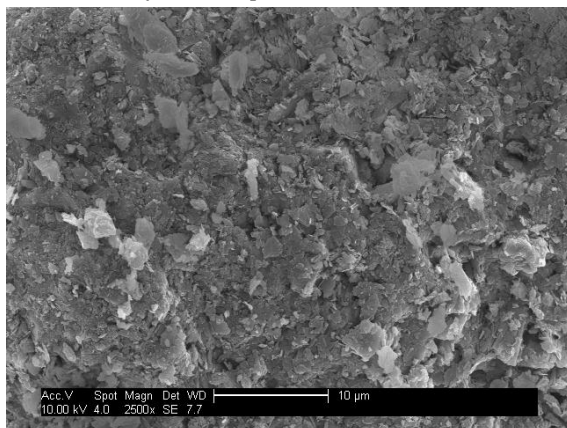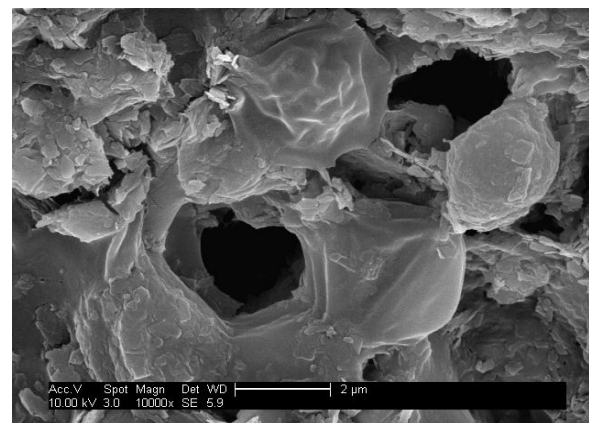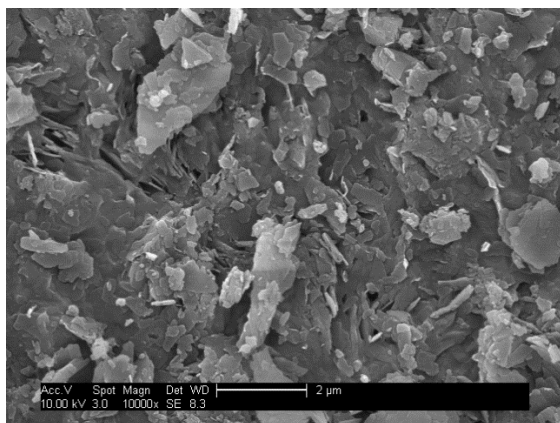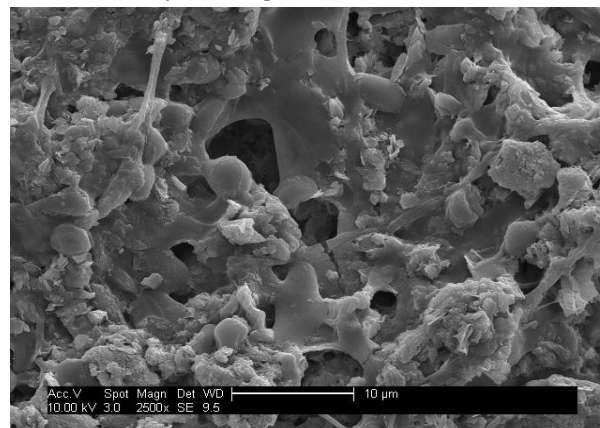
Figure 6.5 Scanning electron microscopy (SEM) images of clinoptilolite (0.7-1.6 mm) before and after saturation in the media bed with a synthetic anaerobic digestion leachate solution (200 mg/L NH$_4$-N, COD loading of 8000 mg L^{-1} with the addition of competing cations) with a bed depth of 0.33 m and an EBCT of 80 mins.

6.3.4 Design and scale up considerations

6.3.4.1 Media selection and performance

Clinoptilolite was established as the most appropriate media with a superior capacity to biochar (12.2 and 5.0 g NH_4-N/kg respectively) through batch adsorption isotherms and kinetic studies. However, empirical data through column experiments is preferable to establish operational capacity and bed design parameters (Cooney et al., 1999). Clinoptilolite has the greatest NH_4-N uptake capacity at long EBCT (Figure 6.2b), which is consistent with the literature (Jorgensen et al., 1976; Demir et al., 2002; Sarioglu, 2005b). However, no other studies have utilised such long EBCT. This is because in conventional ion exchange systems in wastewater treatment (utilising regeneration of the media on-site) it is rare to have such a protracted EBCT because the marginal gain of increasing contact time (20-354 mins) in order to reduce the media usage rate (23-4 $kg/m^3_{treated}$) would not be considered worthwhile to merit the increased reactor volumes that would be required. In addition, as NH_4-N removal treatment systems using ion exchange predominantly rely on media regeneration (Gupta et al., 2015), there is not usually the requirement to utilise the full capacity of the media and consequently when the effluent reaches a defined breakthrough concentration the process is stopped and the media regenerated. However, in a passive NH_4-N recovery system utilising non-regenerative media, it is essential to have a completely saturated media in order to maximise its potential sale price as a fertiliser product. Consequently, operation at long EBCT (>226 mins) would be worthwhile in a passive recovery system utilising clinoptilolite as a non-regenerative media.

Increasing the clinoptilolite particle size from 0.7-1.6 mm to 7-9 mm caused a 25% reduction in NH_4-N adsorption capacity. The uptake of NH_4-N by clinoptilolite has also been observed to decrease by others with the increase in clinoptilolite particle size (Sprynskyy et al., 2005; Wang et al., 2006), and it is apparent that a smaller particle size has higher kinetic uptake rates (Table 6.1). However, there is a trade-off in regards to performance and practicality as the media bed is likely to be more prone to clogging, especially if the digestate liquor has solid matter present in the influent with 150-384 mg TSS L^{-1} reported in digestate liquor in sewage treatment

works (Fux et al., 2002; Mosquera-Corral et al., 2005) and 13-5000 mg TSS L^{-1} in landfill leachate (Renou et al., 2008)). The TSS present in waste streams may resultantly clog void spaces and result in an inhibitory pressure drop build up in the bed before the point of exhaustion has been reached. This means that finer grained material is more susceptible to clogging by solid matter than coarser grained material (Brune et al., 1994). Therefore, use of a larger particle size may be preferential despite reduced uptake capacity (9.3 and 6.4 mg NH_4-N/kg in 0.7-1.6 mm and 7-9 mm clinoptilolite particle sizes respectively). In order to reduce potential clogging problems further it could be beneficial to increase the media particle size to greater than the 7-9 mm tested, such as in gravel filters investigated by Peeling et al. (1999) and Paksy et al. (1998). However, this is not recommended and may be problematic as the majority of fertiliser products have a particle size of between 1.1-5.5 mm (Antille et al., 2013). It is therefore important to remain as close as possible to this range in order to be comparative to commercial fertiliser products and allow existing spreading equipment to be utilised.

6.3.4.2 Passive media bed configuration

If media beds were operated to the point of breakthrough (95% C_0), the LUB fraction ranged from 0.32 to 0.90 dependent upon EBCT. Consequently, this means that at shorter EBCT the media is not being used optimally, which further promotes the use of clinoptilolite as a non-regenerative media and the operation of beds in series. Furthermore, in order to maximise the clinoptilolite's NH_4-N uptake capacity, the operation of media beds in series is necessary and will result in fluctuating NH_4-N concentrations entering subsequent beds. Increasing influent NH_4-N concentrations caused quicker rates of exhaustion (Figure 6.4): with bed volumes to exhaustion decreasing from 635 to 301 with an increase from 60 to 200 mg NH_4-N L^{-1}. In addition it was reported by Hankins et al. (2005b) (in up-flow dynamic column studies) that although clinoptilolite was able to tolerate NH_4-N concentration disturbances, the system was likely to reach exhaustion faster than that of a non-disturbance system. It was also observed by Nguyen and Tanner (1998) that NH_4-N removed from wastewater by clinoptilolite was tightly held by the media and was not released when extraction was attempted with deionised water. This is beneficial as it means that media beds can operate in series and the subsequent flow through each

media bed will not cause desorption of NH_4-N from the media to occur. Despite the use of multiple beds in series, periodic monitoring would also be required to establish when a bed within the series has reached full exhaustion and provide subsequent maintenance and flow redirection. This allows substantial flexibility in the design, for instance in regards to service time, however, it results in the system not being entirely passive in regards to servicing and monitoring.

The EBCT was established as the most important factor influencing the design and operation of a clinoptilolite bed. This has been similarly observed by others when assessing clinoptilolite performance (Du et al., 2005; Karadag et al., 2008; Saeed and Sun, 2012). However, the extended EBCT investigated in this study have not previously been examined, which is important in the creation of a passive NH_4-N recovery system. A bed depth of ≥0.33 m is recommended in the design of a passive NH_4-N recovery system, which means extensive land areas would not be required. Whereas, if a shallow bed depth was used, such as 0.11 m, a 3 fold greater land requirement would be necessary in order to maintain the most efficient EBCT (>226 mins). In addition the need for operation in series would further add to the system's footprint with at least 2 beds in series, dependent on service time required.

The most efficient operation (8.73 kg/m$^3_{treated}$) was at an EBCT ≥ 226 mins which would give a hydraulic flow rate of 33.51 L/m^2/day (with a bed depth of 0.33 m, with two beds in series). However, if an increased rate of flow was desired, an EBCT of 80 mins could be used (efficiency rate of 20.28 kg/m$^3_{treated}$), giving a daily flow rate of 94.82 L/m^2/day. These flow rates are low in comparison to conventional filtration treatment systems for final effluents from sewage treatment works: with reported values for slow sand filtration, rapid sand filtration and deep bed filtration at 720-1140, 1920-4800 and 1920-7680 L/m^2/day respectively (Tchobanoglous et al., 2003). However, the values presented in this study, although lower in regards to system footprint, are feasible in comparison to other passive treatment systems, such as constructed wetlands with loading rates of 18-160 L/m^2/day (Nivala et al., 2013), so could consequently present a viable alternative for NH_4-N recovery.

Therefore, a non-regenerative bed of clinoptilolite for the recovery of NH_4-N has potential for a broad range of applications. For instance, wastewater high in NH_4-N,

that is produced in remote locations, at low hydraulic flow rates such as landfill leachate (0.2 m^3/m^2/year according to Rowe et al. (2000)) and the percolate fraction of sewage sludge dewatering processes such as sludge drying beds (2.4 m^3/m^2/year (Tchobanoglous et al. 2003)), could benefit from a passive nutrient recovery system that requires minimal construction, operation and maintenance costs and, through nutrient recovery, produces a high value fertiliser product. A clinoptilolite bed could also be incorporated into other existing passive systems. For instance, clinoptilolite has been combined with sand filters to retain low concentrations of NH_4-N and effluent spikes that occur in domestic wastewaters (Ferguson and Pepper, 1987; Baykal and Guven, 1997). However, none of these studies aimed to utilise exhausted clinoptilolite, therefore missing the nutrient recovery dimension that can easily be achieved.

Clinoptilolite is an abundant, low cost media, available extensively in countries such as China, Turkey and India at potentially viable prices (approximately 50-100 $USD/tonne) (Shokrian et al., 2015). Through using passive NH_4-N recovery systems the value of clinoptilolite could therefore be raised substantially due to the media now containing increased amounts of NH_4-N (60g NH_4-N/kg clinoptilolite). Through this, NH_4-N can be recovered and concentrated on the clinoptilolite allowing subsequent storage before application at beneficial periods of the growing season, providing greater agronomic benefits as well as reducing leaching into watercourses. The high concentration of NH_4-N on clinoptilolite resultantly reduces transportation costs of liquid digestate. The transport of liquid sludge is extremely expensive and a reduction in transportation costs in excess of 80% can be achieved if the sludge is dewatered and only the dewatered cake has to be removed (Thornton, 2007). In addition, saturated clinoptilolite contains 60 g NH_4-N/kg clinoptilolite in comparison to the same NH_4-N concentration found in the same mass of liquid digestate which contains only 0.02 g NH_4-N/L. Consequently, the recovery of N through saturated clinoptilolite also has the essential element of concentrating N, allowing both storage as well as reducing the vast costs of haulage away from the digester site.

However, the finanacial value of the end product of the process (NH_4-N saturated clinoptilolite) remains unknown due to uncertainties surrounding the extent of beneficial properties to agricultural soils when used as a soil conditioner (Malekian et al. 2011; Lija et al. 2012; Aainaa et al., 2014). Therefore, in order for a treatment process, such as is proposed to be implemented, further research would be required into how saturated clinoptilolite effects soil and plant chemistry, soil structure and the availability of N to crops. These essential research questions would enable tangible financial valuations to be made of the market value of saturated clinoptilolite.

6.4 CONCLUSIONS

In this study the potential for non-regenerative media to be used in a passive system for NH_4-N recovery was investigated. Experiments were performed in batch mode to establish the more effective media to recover NH_4-N from high strength liquors (such as digestate filtrate and landfill leachate). Clinoptilolite had a greater NH_4-N adsorption capacity to biochar (9.3 and 4.8 mg NH_4-N/g media respectively) and a greater kinetic uptake rate (1.013 h^{-1} and 0.557 h^{-1} respectively) and, despite the high concentration of competing cations present in the wastewater, had preferable characteristics for this application. Dynamic column experiments were utilised to determine operational capacity with long EBCT (≥226 mins) established as the most important factor influencing media efficiency (23-4 kg/m^3 wastewater treated). Dynamic experiments carried out at different NH_4-N concentrations indicate that NH_4-N adsorption at high concentrations is faster and exhaustion will occur at a quicker rate. The effect of bed depth on NH_4-N uptake was marginal, with the greater footprint required for shallow clinoptilolite bed depths preventing application in this form. The configuration of a passive clinoptilolite bed (0.33 m depth with an EBCT ≥226 mins) operated in series is a feasible means for NH_4-N recovery from high strength liquors and it's potential for use in a wide range of applications is evident.

6.5 REFERENCES

Aainaa, H. N., Ahmed, O. H., Kasim, S. and Majid, N. M. A. (2014), "Use of Clinoptilolite Zeolite to Reduce Christmas Island Rock Phosphate use in Zea mays Cultivation on an Acid Soil.", *International Journal of Soil Science,* vol. 9, no. 2.

Antille, D. L., Sakrabani, R., Tyrrel, S. F., Le, M. S. and Godwin, R. J. (2013), "Characterisation of organomineral fertilisers derived from nutrient-enriched biosolids granules", *Applied and Environmental Soil Science,* vol. 2013.

APHA (2005), *Standard Methods for the Examination of Water and Waste Water,* American Public Health Association, Baltimore, U.S.A.

Barros, M., Arroyo, P. and Silva, E. (2013), "General Aspects of Aqueous Sorption Process in Fixed Beds- Advances in Sustainable Energy and Environment Oriented Numerical Modeling", in Dr. Hironori Nakajima (ed.) InTech, .

Baykal, B. B. and Guven, D. A. (1997), "Performance of clinoptilolite alone and in combination with sand filters for the removal of ammonia peaks from domestic wastewater", *Water Science and Technology,* vol. 35, no. 7, pp. 47-54.

Berg, U., Donnert, D., Weidler, P., Kaschka, E., Knoll, G. and Nüesch, R. (2006), "Phosphorus removal and recovery from wastewater by tobermorite-seeded crystallisation of calcium phosphate", *Leading-Edge Technology 2005-Wastewater Treatment,* vol. 53, no. 3, pp. 131-138.

Brune, M., Ramke, H. G., Collins, H. J. and Hanert, H. H. (1994), "Incrustation problems in landfill drainage systems", *Landfilling of waste: Barriers*, E & FN Spon, London, UK.

Bulut, E., Özacar, M. and Şengil, İ A. (2008), "Equilibrium and kinetic data and process design for adsorption of Congo Red onto bentonite", *Journal of Hazardous Materials,* vol. 154, no. 1–3, pp. 613-622.

Carley, B. and Mavinic, D. (1991), "The effects of external carbon loading on nitrification and denitrification of a high-ammonia landfill leachate", *Research Journal of the Water Pollution Control Federation,* , pp. 51-59.

Cockburn, A., Brambilla, G., Fernández, M., Arcella, D., Bordajandi, L. R., Cottrill, B., van Peteghem, C. and Dorne, J. (2013), "Nitrite in feed: From Animal health to human health", *Toxicology and Applied Pharmacology,* vol. 270, no. 3, pp. 209-217.

Cooney, E. L., Booker, N. A., Shallcross, D. C. and Stevens, G. W. (1999), "Ammonia removal from wastewaters using natural Australian zeolite. I. Characterization of the zeolite", *Separation Science and Technology,* vol. 34, no. 12, pp. 2307-2327.

Demir, A., Gunay, A. and Debik, E. (2002), "Ammonium removal from aqueous solution by ion-exchange using packed bed natural zeolite", *Water SA,* vol. 28, no. 3, pp. 329-336.

Dryden, H. T. and Weatherley, L. R. (1989), "Aquaculture water treatment by ion exchange: Continuous ammonium ion removal with clinoptilolite", *Aquacultural Engineering,* vol. 8, no. 2, pp. 109-126.

Du, Q., Liu, S., Cao, Z. and Wang, Y. (2005), "Ammonia removal from aqueous solution using natural Chinese clinoptilolite", *Separation and Purification Technology,* vol. 44, no. 3, pp. 229-234.

Erdoğan, B. C. and Ülkü, S. (2011), "Ammonium sorption by Gördes clinoptilolite rich mineral specimen", *Applied Clay Science,* vol. 54, no. 3, pp. 217-225.

FAO (2015), *World fertilizer trends and outlook to 2018,* 1st ed, Food and Agricultural Organisation of the United Nations, Rome, Italy.

Ferguson, G. and Pepper, I. (1987), "Ammonium retention in sand amended with clinoptilolite", *Soil Science Society of America Journal,* vol. 51, no. 1, pp. 231-234.

Fertilizers Europe (2013)*, EU Fertilizer Market - Key Graphs*, available at: http://www.fertilizerseurope.com/fileadmin/user_upload/publications/statistics_ publications/Stat_website.pdf (accessed 5th May 2015).

Fux, C., Boehler, M., Huber, P., Brunner, I. and Siegrist, H. (2002), "Biological treatment of ammonium-rich wastewater by partial nitritation and subsequent anaerobic ammonium oxidation (anammox) in a pilot plant", *Journal of Biotechnology,* vol. 99, no. 3, pp. 295-306.

Guest, J. S., Skerlos, S. J., Barnard, J. L., Beck, M. B., Daigger, G. T., Hilger, H., Jackson, S. J., Karvazy, K., Kelly, L. and Macpherson, L. (2009), "A new planning and design paradigm to achieve sustainable resource recovery from wastewater 1", *Environmental Science and Technology,* vol. 43, no. 16, pp. 6126-6130.

Guo, X., Zeng, L., Li, X. and Park, H. (2008), "Ammonium and potassium removal for anaerobically digested wastewater using natural clinoptilolite followed by membrane pretreatment", *Journal of Hazardous Materials,* vol. 151, no. 1, pp. 125-133.

Han, R., Wang, Y., Zou, W., Wang, Y. and Shi, J. (2007), "Comparison of linear and nonlinear analysis in estimating the Thomas model parameters for methylene blue adsorption onto natural zeolite in fixed-bed column", *Journal of Hazardous Materials,* vol. 145, no. 1-2, pp. 331-335.

Hankins, N., Pliankarom, S. and Hilal, N. (2005a), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

Hankins, N., Pliankarom, S. and Hilal, N. (2005b), "Removal of NH4 Ion from NH4Cl Solution Using Clinoptilolite: A Dynamic Study Using a Continuous Packed-

Demir, A., Gunay, A. and Debik, E. (2002), "Ammonium removal from aqueous solution by ion-exchange using packed bed natural zeolite", *Water SA,* vol. 28, no. 3, pp. 329-336.

Dryden, H. T. and Weatherley, L. R. (1989), "Aquaculture water treatment by ion exchange: Continuous ammonium ion removal with clinoptilolite", *Aquacultural Engineering,* vol. 8, no. 2, pp. 109-126.

Du, Q., Liu, S., Cao, Z. and Wang, Y. (2005), "Ammonia removal from aqueous solution using natural Chinese clinoptilolite", *Separation and Purification Technology,* vol. 44, no. 3, pp. 229-234.

Erdoğan, B. C. and Ülkü, S. (2011), "Ammonium sorption by Gördes clinoptilolite rich mineral specimen", *Applied Clay Science,* vol. 54, no. 3, pp. 217-225.

FAO (2015), *World fertilizer trends and outlook to 2018,* 1st ed, Food and Agricultural Organisation of the United Nations, Rome, Italy.

Ferguson, G. and Pepper, I. (1987), "Ammonium retention in sand amended with clinoptilolite", *Soil Science Society of America Journal,* vol. 51, no. 1, pp. 231-234.

Fertilizers Europe (2013)*, EU Fertilizer Market - Key Graphs*, available at: http://www.fertilizerseurope.com/fileadmin/user_upload/publications/statistics_ publications/Stat_website.pdf (accessed 5th May 2015).

Fux, C., Boehler, M., Huber, P., Brunner, I. and Siegrist, H. (2002), "Biological treatment of ammonium-rich wastewater by partial nitritation and subsequent anaerobic ammonium oxidation (anammox) in a pilot plant", *Journal of Biotechnology,* vol. 99, no. 3, pp. 295-306.

Guest, J. S., Skerlos, S. J., Barnard, J. L., Beck, M. B., Daigger, G. T., Hilger, H., Jackson, S. J., Karvazy, K., Kelly, L. and Macpherson, L. (2009), "A new planning and design paradigm to achieve sustainable resource recovery from wastewater 1", *Environmental Science and Technology,* vol. 43, no. 16, pp. 6126-6130.

Guo, X., Zeng, L., Li, X. and Park, H. (2008), "Ammonium and potassium removal for anaerobically digested wastewater using natural clinoptilolite followed by membrane pretreatment", *Journal of Hazardous Materials,* vol. 151, no. 1, pp. 125-133.

Han, R., Wang, Y., Zou, W., Wang, Y. and Shi, J. (2007), "Comparison of linear and nonlinear analysis in estimating the Thomas model parameters for methylene blue adsorption onto natural zeolite in fixed-bed column", *Journal of Hazardous Materials,* vol. 145, no. 1-2, pp. 331-335.

Hankins, N., Pliankarom, S. and Hilal, N. (2005a), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

Hankins, N., Pliankarom, S. and Hilal, N. (2005b), "Removal of NH4 Ion from NH4Cl Solution Using Clinoptilolite: A Dynamic Study Using a Continuous Packed-

Bed Column in Up-Flow Mode", *Separation Science and Technology,* vol. 39, no. 6, pp. 1347-1364.

Hasanoğlu, A., Romero, J., Pérez, B. and Plaza, A. (2010), "Ammonia removal from wastewater streams through membrane contactors: Experimental and theoretical analysis of operation parameters and configuration", *Chemical Engineering Journal,* vol. 160, no. 2, pp. 530-537.

Hedström, A. (2001), "Ion exchange of ammonium in zeolites: a literature review", *Journal of Environmental Engineering,* vol. 127, no. 8, pp. 673-681.

Huang, H., Xiao, X., Yan, B. and Yang, L. (2010), "Ammonium removal from aqueous solutions by using natural Chinese (Chende) zeolite as adsorbent", *Journal of Hazardous Materials,* vol. 175, no. 1, pp. 247-252.

Huo, H., Lin, H., Dong, Y., Cheng, H., Wang, H. and Cao, L. (2012), "Ammonia-nitrogen and phosphates sorption from simulated reclaimed waters by modified clinoptilolite", *Journal of Hazardous Materials,* vol. 229, pp. 292-297.

Hylander, L. D., Kietlińska, A., Renman, G. and Simán, G. (2006), "Phosphorus retention in filter materials for wastewater treatment and its subsequent suitability for plant production", *Bioresource technology,* vol. 97, no. 7, pp. 914-921.

Jeffery, S., Bezemer, T. M., Cornelissen, G., Kuyper, T. W., Lehmann, J., Mommer, L., Sohi, S. P., Voorde, T. F., Wardle, D. A. and Groenigen, J. W. (2015), "The way forward in biochar research: targeting trade-offs between the potential wins", *GCB Bioenergy,* vol. 7, no. 1, pp. 1-13.

Johansson, L. and Gustafsson, J. P. (2000), "Phosphate removal using blast furnace slags and opoka-mechanisms", *Water Research,* vol. 34, no. 1, pp. 259-265.

Jorgensen, S., Libor, O., Graber, K. L. and Barkacs, K. (1976), "Ammonia removal by use of clinoptilolite", *Water Research,* vol. 10, no. 3, pp. 213-224.

Karadag, D., Tok, S., Akgul, E., Turan, M., Ozturk, M. and Demir, A. (2008), "Ammonium removal from sanitary landfill leachate using natural Gördes clinoptilolite", *Journal of Hazardous Materials,* vol. 153, no. 1, pp. 60-66.

Kitano, M., Inoue, Y., Yamazaki, Y., Hayashi, F., Kanbara, S., Matsuishi, S., Yokoyama, T., Kim, S., Hara, M. and Hosono, H. (2012), "Ammonia synthesis using a stable electride as an electron donor and reversible hydrogen store", *Nature Chemistry,* vol. 4, no. 11, pp. 934-940.

Kleinübing, S. and da Silva, M. (2008), "Lead removal process modeling in natural zeolita clinoptilolita through dynamic and batch systems", *Scientia Plena,* vol. 4, no. 2.

Kučić, D., Markić, M. and Briški, F. (2012), "Ammonium adsorption on natural zeolite (clinoptilolite): adsorption isotherms and kinetics modeling", *The Holistic Approach to Environment,* vol. 2, no. 4, pp. 145-158.

Lija, W., Ahmed, O. and Kasim, S. (2012), "Reducing ammonia volatilization from compound fertilizers amended with zeolite", *African Journal of Biotechnology,* vol. 11, pp. 13903-13906.

Liu, B., Giannis, A., Zhang, J., Chang, V. W. and Wang, J. (2014), "Air stripping process for ammonia recovery from source-separated urine: modeling and optimization", *Journal of Chemical Technology and Biotechnology, .*

Malekian, R., Abedi-Koupai, J. and Eslamian, S. S. (2011), "Influences of clinoptilolite and surfactant-modified clinoptilolite zeolite on nitrate leaching and plant growth", *Journal of Hazardous Materials,* vol. 185, no. 2-3, pp. 970-976.

Martin, B. D., De Kock, L., Stephenson, T., Parsons, S. A. and Jefferson, B. (2013), "The impact of contactor scale on a ferric nanoparticle adsorbent process for the removal of phosphorus from municipal wastewater", *Chemical Engineering Journal,* vol. 215–216, no. 1, pp. 209-215.

Mashal, A., Abu-Dahrieh, J., A. Ahmed, A., Oyedele, L., Haimour, N., Al-Haj-Ali, A. and Rooney, D. (2014), "Fixed-bed study of ammonia removal from aqueous solutions using natural zeolite", *World Journal of Science, Technology and Sustainable Development,* vol. 11, no. 2, pp. 144-158.

McCarty, P. L., Bae, J. and Kim, J. (2011), "Domestic wastewater treatment as a net energy producer–can this be achieved?", *Environmental Science and Technology,* vol. 45, no. 17, pp. 7100-7106.

Metropoulos, K., Maliou, E., Loizidou, M. and Spyrellis, N. (1993), "Comparative studies between synthetic and natural zeolites for ammonium uptake", *Journal of Environmental Science and Health Part A,* vol. 28, no. 7, pp. 1507-1518.

Mosquera-Corral, A., Gonzalez, F., Campos, J. and Méndez, R. (2005), "Partial nitrification in a SHARON reactor in the presence of salts and organic carbon compounds", *Process Biochemistry,* vol. 40, no. 9, pp. 3109-3118.

Muherei, M. A. and Junin, R. (2009), "Equilibrium adsorption isotherms of anionic, nonionic surfactants and their mixtures to shale and sandstone", *Modern Applied Science,* vol. 3, no. 2, pp. p158.

Nguyen, M. and Tanner, C. (1998), "Ammonium removal from wastewaters using natural New Zealand zeolites", *New Zealand Journal of Agricultural Research,* vol. 41, no. 3, pp. 427-446.

Nivala, J., Headley, T., Wallace, S., Bernhard, K., Brix, H., van Afferden, M. and Müller, R. A. (2013), "Comparative analysis of constructed wetlands: the design and construction of the ecotechnology research facility in Langenreichenbach, Germany", *Ecological Engineering,* vol. 61, pp. 527-543.

Paksy, A., Powrie, W., Robinson, J. and Peeling, L. (1998), "A laboratory investigation of anaerobic microbial clogging in granular landfill drainage media", *Geotechnique,* vol. 48, no. 3, pp. 389-401.

Park, S. and Kim, M. (2015), "Innovative ammonia stripping with an electrolyzed water system as pretreatment of thermally hydrolyzed wasted sludge for anaerobic digestion", *Water Research,* vol. 68, pp. 580-588.

Parkinson, J. and Tayler, K. (2003), "Decentralized wastewater management in peri-urban areas in low-income countries", *Environment and Urbanization,* vol. 15, no. 1, pp. 75-90.

Pawełczyk, A. and Popowicz, A. (2006), "Application of natural zeolites in the production of controlledrelease fertilizers", *Book of Abstracts,* pp. 203.

Peeling, L., Paksy, A., Robinson, J. P. and Powrie, W. (1999), "Removal of volatile acids from synthetic landfill leachate by anaerobic biofilms on drainage aggregates: a laboratory study", *Waste Management and Research,* vol. 17, no. 2, pp. 141-149.

Porter, J., McKay, G. and Choy, K. (1999), "The prediction of sorption from a binary mixture of acidic dyes using single-and mixed-isotherm variants of the ideal adsorbed solute theory", *Chemical Engineering Science,* vol. 54, no. 24, pp. 5863-5885.

Renou, S., Givaudan, J. G., Poulain, S., Dirassouyan, F. and Moulin, P. (2008), "Landfill leachate treatment: Review and opportunity", *Journal of Hazardous Materials,* vol. 150, no. 3, pp. 468-493.

Rose, C., Parker, A., Collins, G. and Cartmell, E. (2015), "Nutrient recovery form high solids anaerobic digestion effluents in location x", *3rd International Faecal Sludge Management Conference,* 18-21 January, Hanoi, Vietnam, EAWAG, Dubendorf, Switzerland, .

Rowe, R. K., Armstrong, M. D. and Cullimore, D. R. (2000), "Particle size and clogging of granular media permeated with leachate", *Journal of Geotechnical and Geoenvironmental Engineering,* vol. 126, no. 9, pp. 775-786.

Saeed, T. and Sun, G. (2012), "A review on nitrogen and organics removal mechanisms in subsurface flow constructed wetlands: Dependency on environmental parameters, operating conditions and supporting media", *Journal of Environmental Management,* vol. 112, pp. 429-448.

Sarioglu, M. (2005a), "Removal of ammonium from municipal wastewater using natural Turkish (Dogantepe) zeolite", *Separation and Purification Technology,* vol. 41, no. 1, pp. 1-11.

Sarioglu, M. (2005b), "Removal of ammonium from municipal wastewater using natural Turkish (Dogantepe) zeolite", *Separation and Purification Technology,* vol. 41, no. 1, pp. 1-11.

Sarkhot, D., Ghezzehei, T. and Berhe, A. (2013), "Effectiveness of biochar for sorption of ammonium and phosphate from dairy effluent", *Journal of Environmental Quality,* vol. 42, no. 5, pp. 1545-1554.

Semmens, M., Klieve, J., Schnobrich, D. and Tauxe, G. W. (1981), "Modeling ammonium exchange and regeneration on clinoptilolite", *Water Research,* vol. 15, no. 6, pp. 655-666.

Sohi, S., Lopez-Capel, E., Krull, E. and Bol, R. (2009), "Biochar, climate change and soil: A review to guide future research", *CSIRO Land and Water Science Report,* vol. 5, no. 09, pp. 17-31.

Speer, S., Champagne, P. and Anderson, B. (2012), "Pilot-scale comparison of two hybrid-passive landfill leachate treatment systems operated in a cold climate", *Bioresource Technology,* vol. 104, no. 0, pp. 119-126.

Sprynskyy, M., Lebedynets, M., Terzyk, A. P., Kowalczyk, P., Namieśnik, J. and Buszewski, B. (2005), "Ammonium sorption from aqueous solutions by the natural zeolite Transcarpathian clinoptilolite studied under dynamic conditions", *Journal of Colloid and Interface Science,* vol. 284, no. 2, pp. 408-415.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Tetreault, G. R., Brown, C. J., Bennett, C. J., Oakes, K. D., McMaster, M. E. and Servos, M. R. (2013), "Fish community responses to multiple municipal wastewater inputs in a watershed", *Integrated environmental assessment and management,* vol. 9, no. 3, pp. 456-468.

Thornton, A., Pearce, P. and Parsons, S. A. (2007), "Ammonium removal from digested sludge liquors using ion exchange", *Water Research,* vol. 41, no. 2, pp. 433-439.

Ulyett, J., Sakrabani, R., Kibblewhite, M. and Hann, M. (2014), "Impact of biochar addition on water retention, nitrification and carbon dioxide evolution from two sandy loam soils", *European Journal of Soil Science,* vol. 65, no. 1, pp. 96-104.

US EPA (2007), *Wastewater Technology Fact Sheet: Sidestream Nutrient Removal,* , United States Environmental Protection Agency, Office of Water, U.S.A.

Vassileva, P., Tzvetkova, P. and Nickolov, R. (2009), "Removal of ammonium ions from aqueous solutions with coal-based activated carbons modified by oxidation", *Fuel,* vol. 88, no. 2, pp. 387-390.

Wang, S. and Peng, Y. (2010), "Natural zeolites as effective adsorbents in water and wastewater treatment", *Chemical Engineering Journal,* vol. 156, no. 1, pp. 11-24.

Wang, S. and Wu, H. (2006), "Environmental-benign utilisation of fly ash as low-cost adsorbents", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 482-501.

Wang, Y., Lin, F. and Pang, W. (2007), "Ammonium exchange in aqueous solution using Chinese natural clinoptilolite and modified zeolite", *Journal of Hazardous materials,* vol. 142, no. 1, pp. 160-164.

Wang, Y., Liu, S., Xu, Z., Han, T., Chuan, S. and Zhu, T. (2006), "Ammonia removal from leachate solution using natural Chinese clinoptilolite", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 735-740.

Widiastuti, N., Wu, H., Ang, H. M. and Zhang, D. (2011), "Removal of ammonium from greywater using natural zeolite", *Desalination,* vol. 277, no. 1, pp. 15-23.

Xu, G., Fan, X. and Miller, A. J. (2012), "Plant nitrogen assimilation and use efficiency", *Annual review of plant biology,* vol. 63, pp. 153-182.

CHAPTER 7

INTEGRATED SLUDGE DRYING BEDS AND NUTRIENT RECOVERY UTILISING NON REGENERATIVE MEDIA AND A SACRIFICIAL SAND BARRIER

7 INTEGRATED SLUDGE DRYING BEDS AND NUTRIENT RECOVERY UTILISING NON-REGENERATIVE MEDIA AND A SACRIFICIAL SAND BARRIER

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ABSTRACT

Anaerobic digestate is rich in nitrogen, phosphorus and potassium which are all nutrients required for plant growth. However, following dewatering processes the majority of the nitrogen and potassium is lost in the liquor fraction, leaving a biosolids product that, although rich in phosphorus, has unbalanced nutrient ratios and has a resultant low utility as an agricultural fertiliser. In this study we present a process to recover nutrients from digestate through the integration of an adsorption/ion exchange zone into a sludge drying bed, allowing the simultaneous dewatering and recovery of nutrients from the percolate fraction. The ability of biochar to capture ammonium in the configuration was limited with ammonium removal (13% removal) similar to that of a sand control (8% removal). In contrast, clinoptilolite performed optimally with ammonium removal rates of 62-99%, dependent on particle size and configuration. However, clogging of the media bed occurred rapidly (0.88-12.39 bed volumes) before full saturation of the media bed had occurred. A sacrificial sand barrier was

subsequently incorporated into the design to reduce media clogging (37-56 % TS removal) and intensify drying bed operation through the scraping of residual solids to prevent the onset of cake filtration (0.046-0.500 kg TS/m^2). Clinoptilolite was successfully utilised as a non-regenerative adsorbent in an integrated nutrient recovery sludge drying bed design with a sacrificial sand bed. The exhausted clinoptilolite (saturated in ammonium and potassium) can either be utilised directly or blended with dewatered biosolids (high in phosphorus) to ensure complete nutrient recovery and create an enriched biosolids product.

KEY WORDS

Ammonium recovery, sludge drying beds, ion exchange, faecal sludge management, nutrient recovery, clinoptilolite

HIGHLIGHTS

- A process to enable complete nutrient recovery using sludge drying beds was proposed.

- Clinoptilolite incorporated in bed profile to recover NH_4^+ and K^+ from percolate.

- Recovery rate of 62-99% ammonium in effluent percolate when clinoptilolite used.

- Sacrificial sand barrier needed to enable media saturation and intensify operation.

- Saturated clinoptilolite and biosolids gives high value enriched fertiliser product.

7.1 INTRODUCTION

Sewage sludge and faecal sludge (FS) are unavoidable by-products of wastewater treatment and on-site sanitation facilities. The average sewage sludge production in European countries is approximately 0.09 kg TS/cap/day^{-1} (Brester et al., 1998). Anaerobic digestion (AD) is the most common means of treatment and stabilisation of sewage sludge in Europe (Fytili and Zabaniotou, 2008) and is an expanding area for the decentralised treatment of faecal sludge in low income countries (Collins et al., 2013; Strande et al., 2014). The AD process breaks down pathogenic microorganisms, converts organic matter to methane (CH_4) and carbon dioxide (CO_2), and the residual solids (referred to as digestate) are rich in valuable nutrients (Speece, 2008). The effluent digestate can be utilised directly in agriculture as a liquid digestate (Paavola and Rintala, 2008) or, alternatively, can be further refined in order to create products of increased value. This provides a distinct opportunity for cost recovery of treatment and collection processes, following the shifting paradigm away from what must be removed from wastewater to what can be recovered from wastewater (Guest et al., 2009).

Nutrient levels in digestate are substantial, with the key plant fertilising nutrients nitrogen (N), phosphorus (P) and potassium (K) present. In effluent digestate, treating sewage and faecal sludge, large concentrations of ammonium-nitrogen (NH_4-N) are reported at 200-700 and 520-1853 mg NH_4-N L^{-1} respectively (Thornton et al., 2007b; Rose et al., 2015). In addition high levels of P and K have been reported at 154-1617 mg P L^{-1} and 390 mg K L^{-1} respectively (Parsons et al., 2001; Rose et al., 2015). Agricultural productivity relies heavily on N and P fertilisers in order to achieve high crop yields (Xu et al., 2012). Nitrogen fertiliser production amounted to 113 million tonnes in 2014, with forecasted growth of 1.4% annually until 2018 (FAO, 2015) and global phosphate rock production was estimated at 195 million tonnes in 2011 (Watson et al., 2014). Additionally, the recovery of NH_4-N also has global significance in regards to energy production and ecological drivers, with ~7% of the world's natural gas production used in 1990 to fix atmospheric N to satisfy global demand, there is a requirement to save energy by using re-cycled

nutrients in agriculture rather than spend energy removing fertilising elements from wastewaters (McCarty et al., 2011).

The direct application of anaerobic digestate to agricultural land is commonplace and does recover nutrients, however, prior to dewatering, digestate comprises > 90% water (Speece, 2008) resulting in high transportation and application costs. In addition, the spreading of liquid digestate can lead to the volatilisation and loss of ammonia (NH_3) as well as contributing to pollution of waterways due to direct run off and leaching of nitrate (Kowaljow et al., 2010). The dewatering of AD effluents is therefore necessary to address these issues, such as through settlement or centrifugal processes, which increases TS concentrations to typically 25-35% TS (Holm-Nielsen et al., 2009). However, within these dewatered biosolids it can be expected that 60-80% of the original P content of the digestate will be maintained, but only 11-25% of the N and 10-15% of the K (Martin, 2005; Møller et al., 2006). The lack of N in dewatered biosolids therefore lowers its utility as an agricultural fertiliser and inefficiencies in nutrient recovery are presented.

The increasing cost of artificial fertilisers (Heffer and Prud'homme, 2013) and low utility of biosolids in agriculture have given rise to recent research into enhancing biosolids to increase product value and reduce artificial fertiliser input requirements: for instance, Antille et al. (2013) investigated the coating of dewatered biosolids with urea to increase N content. This approach requires additional inputs of artificial fertilisers. However, if the substantial N and K concentrations lost during the dewatering process (75-89% N and 85-90% K (Martin, 2005; Møller et al., 2006)) could be recovered from liquors during the dewatering process and combined with the dewatered biosolids (already high in P) a complete fertiliser product could be generated with equal N-P-K proportions (e.g. 10-10-10 wt. %) as marketed in artificial fertiliser products (Lima et al., 2015).

These factors create a need to develop a treatment process that will dewater AD effluents and recover and concentrate NH_4-N and K^+ to create a final product that can be added to P rich biosolids. Existing options for NH_4-N

recovery from AD liquors requires the prior separation of solid and liquid fractions before recovery can take place. For instance, reverse osmosis, and other options, such as ammonia (NH_3) air stripping, require acid to convert NH_3 to marketable fertilisers and both have high energy demands (Park and Kim, 2015). Passive ion exchange and adsorption systems for NH_4-N recovery in anaerobic liquors, has been identified as a feasible means of recovery with low energy requirements (Chapter 6). However, the effect of the high solids nature of digestate percolate is yet to be investigated.

There are numerous methods of solid/liquid separation (e.g. centrifuge, screw or belt filter press); however, one of the simplest and least energy intensive processes are sludge drying beds which are the most widely used method of sludge dewatering in both low income countries such as Ghana (Koné and Strauss, 2004) as well as high income countries such as the United States (Tchobanoglous et al., 2003). However, sludge drying beds cannot operate in isolation; the percolate requires further treatment, often with the recirculation of effluents to the head of a sewage works (Abel, 1996). Alternatively, if anaerobic digesters are operating at a decentralised level away from wastewater treatment works (WwTW) (as is a growing trend in faecal sludge management (Gutterer et al., 2009)) then further treatment of percolate can be problematic. There is, therefore, a significant opportunity to modify sludge drying beds to make them a more efficient dewatering and nutrient recovering process and combining these two processes into one single process train could present substantial benefit.

The predominant mechanism for dewatering in sand sludge drying beds is through percolation, with approximately 75% of water loss occurring through this mechanism (van Haandel and Van Der Lubbe, 2007). In order to capitalise on this factor, there is potential for inserting an adsorption or ion exchange zone into the drying bed profile in order to capture nutrients that would have previously been lost in the percolate stream. However, one of the greatest costs and complications of adsorption and ion exchange systems in wastewater treatment processes is the regeneration of exhausted media (Wang and Wu, 2006), which requires increased infrastructure, expensive chemical regenerants

as well as final disposal issues for brine (Mormile et al., 1999). Synthetic ion exchange media, e.g. Mesolite, has a high purchase cost but a greater NH_4-N capacity (49 g NH_4-N kg^{-1}) (Thornton et al., 2007b), in comparison to abundant low-cost media, such as clinoptilolite (12.2 g NH_4-N kg^{-1}) which could be used as a non-regenerative media (Rose et al. 2015). This difference in media capacity will impact media bed life, with reduced treatment capacity by non-regenerative media before servicing of the media bed is required. Nevertheless, a lower purchase price and operational cost make the use of non-regenerative media attractive. In addition, due to the large footprint of sludge drying beds (Dodane and Ronteltap, 2014), and resultantly low hydraulic flow rates of percolate liquors, the greater capacity of high cost regenerative media is unnecessary. Consequently, there is a strong driver for the use of non-regenerative passive medias which, once saturated with nutrients, can be directly applied (or co-applied with biosolids) to agricultural soils.

The integration of sludge drying beds and non-regenerative media to achieve complete nutrient recovery presents multiple challenges. Previous studies (Rose et al 2015) have investigated the operational capacity and configuration of non-regenerative media (biochar and clinoptilolite) without the interference of TS in digestate. However, the percolate of sludge drying beds will still contain substantial amounts of TS (5700-6100 mg TS L^{-1} with removal of 80-81% TS reported by Cofie et al. (2006)) which could prove problematic to ion exchange and adsorption processes. In sludge drying beds, the predominant mechanism for volume reduction is through percolation (50-80%), in which predominantly free water is lost, within a time span of 1-3 days (Heinss et al., 1999; Tchobanoglous et al., 2003; van Haandel and Van Der Lubbe, 2007). The remaining bound water is subsequently lost through evaporation but this is highly dependent on climatic conditions (temperature, humidity, wind) and takes substantially longer. Therefore, the percolation rate will be the primary mechanism controlling the rate of NH_4-N recovery in an integrated sludge drying bed and nutrient recovery design.

However, the rate of percolation will not be consistent throughout the drying bed cycle due to gradual build-up of TS on the surface of the bed, known as cake

filtration. The onset of cake filtration is important as it provides an indication as to the TS loading before cake filtration commences; resulting in the incremental build-up of solids retained on top of the filter medium, which subsequently provides the majority of filtration action but at a reduced hydraulic rate. Therefore, optimising the hydraulic and TS loading of drying beds to ensure optimal percolation rates is necessary in order to establish nutrient recovery rates and operational footprint. A full investigation into influencing factors, such as the onset of cake filtration and TS loading rates, is essential to establish an integrated nutrient recovery drying bed design.

The aim of this study is to assess the feasibility of modifying sludge drying beds to incorporate a nutrient recovery zone in which non-regenerative media (biochar and clinoptilolite) will be used to capture and recover NH_4-N and K^+. In addition, the use of a sacrificial sand barrier to maintain media bed life and dewater P rich biosolids will be investigated. This study aims to present process design information along with a discussion centred around the relative trade-offs involved in the design and operation of the modified sludge drying bed concept. Specifically, the objectives are to a) establish the performance of a pilot scale media bed for the recovery of NH_4-N and K^+ from anaerobic digestate; b) determine the TS loading required before the onset of clogging commences at the media: sludge interface and on a sacrificial sand barrier; d) determine optimum TS loading rates in batch fed sludge drying beds; and e) propose design values for the operation of an integrated sludge drying bed and nutrient recovery system.

7.2 MATERIALS AND METHODS

7.2.1 Non-regenerative media description

Clinoptilolite (supplied by RS Minerals Ltd., Cleveland, UK) was screened into 3 fractions of 0.7-1.6 mm, 2-4 mm and 7-9 mm. The media was predominantly clinoptilolite (92%) with other clay minerals (5% smectite and 3% biotite) with a pH of 6.5-7.5, bulk density of 1.23 g/cm^3, real density of 2.25 g/cm^3, porosity of 84.5% and a specific surface area of 41 m^2/g. A deciduous mixed wood biochar (Biochar Foundation, Loanhead, UK) was used with production carried out

according to Ulyett et al. (2014). The biochar media had a pH of 10.2, C:N ratio of 117 and a cation exchange capacity of 66 cmol$^+$ kg^{-1} (Ulyett et al., 2014). The char was sieved to particle sizes of 6, 4, 3.5,2, 1 mm. Silica sand (Garside Sands, Leighton Buzzard, UK) with particle size of 0.63-0.85, 0.9-1.18 and 2-2.7mm was used. Gravel was used to enable under bed drainage and had a 6 mm particle size. Before use, all media was rinsed with deionised water to remove impurities, before drying at 103°C.

7.2.2 Anaerobic digestate used in experimental work

The effluent from a continuous stirred-tank mesophilic anaerobic digester, with thermal hydrolysis pre-treatment, fed with sewage sludge from a WwTW (p.e. of 288000) was used as a high solids anaerobically digested sewage sludge. The anaerobic digestate was diluted using tap water, in order to ensure the consistent presence of competing cations in the digestate dilutions according to Thornton et al. (2007a). This provided a range of total solids (TS) concentrations between 5000 - 50000 mg L^{-1} TS.

7.2.3 Dynamic column studies with anaerobic digestate

Digestate was used to determine the effect of high solids loading on the NH$_4$-N , K and P recovery process with three columns used in parallel (inside diameter: 0.15 m, length: 1.5 m: Figure 7.1) for the three different media types (sand, biochar and clinoptilolite). The anaerobic sludge was stored in a continuously stirred tank to prevent any settlement of solids occurring (Figure 7.1). Sludge was then pumped using a peristaltic pump (Watson Marlow, Falmouth, UK) to a gravity fed column with a fixed media bed volume of 8830 cm^3 and surface area of 0.178 m^2 (Figure 7.1). An empty bed contact time (EBCT) of 354 minutes was used and columns were operated continuously with free drainage until the bed clogged. A pressure trigger, defined as when there was >1 bed volume of influent sludge suspended above the surface of the media bed for a period >24 hours, was used to signal that clogging had occurred and the end of each experimental run was indicated. The media bed depth (0.5 m) and volume (8830 cm^3) remained constant throughout all experimental runs and was underlain by gravel (6 mm particle size) to support the media bed and enable

efficient drainage to occur (Figure 7.1). In order to investigate the effect of different particle sizes on filtration and nutrient capture, media beds were configured in two ways. Firstly, multilayer graded media beds were configured with the largest particle size at the top of the bed reducing in size to the smallest media particle size at the bottom of the bed (5.4, 2 and 1 mm in char; 2.35, 1.99 and 1.5 mm in sand; and 7, 4 and 2.3 mm in clinoptilolite) in order to prevent clogging at the media: sludge interface, secondly one bed of a single particle size was used (2.35 mm, 5.4 mm, 7 mm in sand, biochar and clinoptilolite respectively). All media configurations had a bed volume of 8830 cm^3, a bed depth of 0.5 m and were supported by a gravel bed (6 mm particle size) of an equal proportion. Influent (S_i) samples were taken daily from the feed tank and effluent (S_e) samples from the base of each column (Figure 7.1) to determine removal efficiencies.
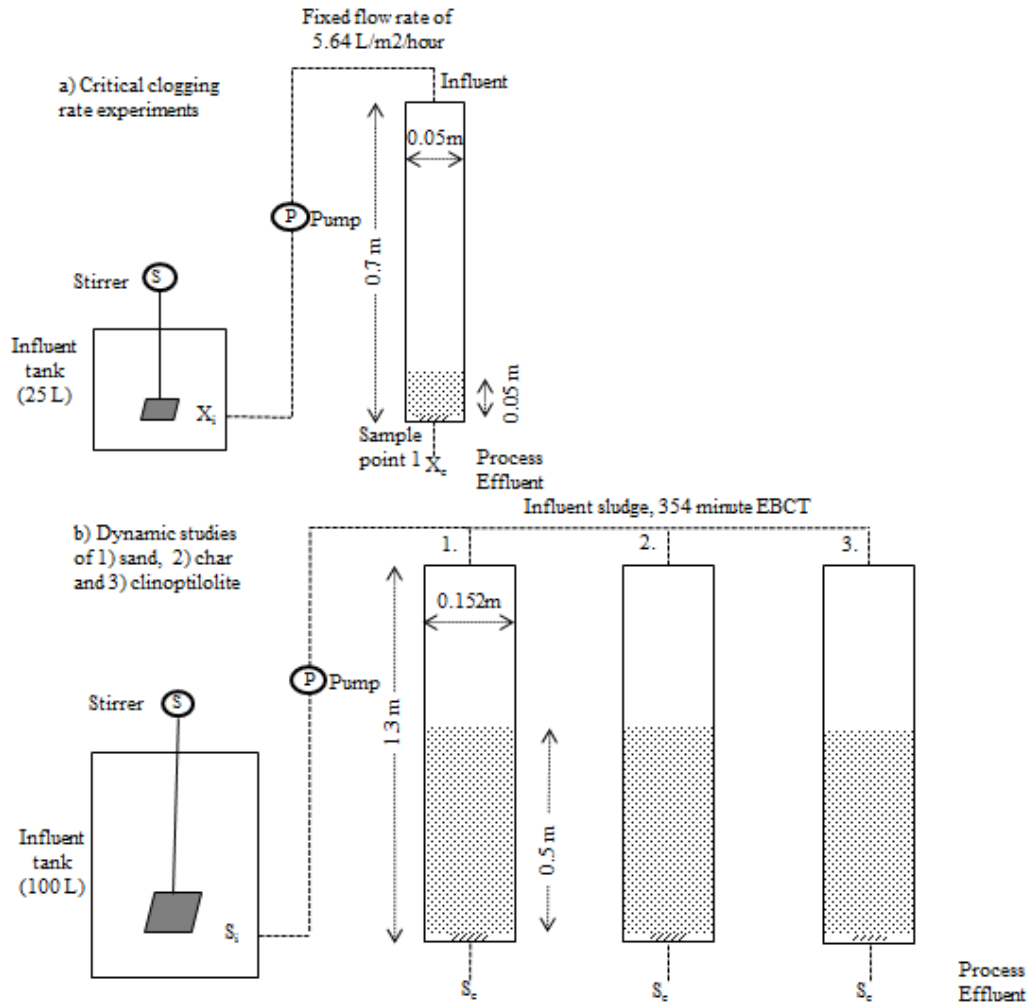
Figure 7.1 Experimental set-up for a) critical solids loading rates for the onset of cake filtration and b) dynamic studies investigating NH$_4$-N uptake by sand, clinoptilolite and biochar.

7.2.4 Sacrificial sand barrier

7.2.4.1 Onset of cake filtration through continuous flow experiments

The onset of cake filtration was determined on a sacrificial sand barrier with two different sand particle sizes (0.9-1.2 mm and 0.6-0.9 mm) and was established by operating a column (inside diameter: 0.05 m) with a bed surface area of 0.02 m^2 and a bed depth of 0.05 m (sand bed volume 98.17 cm^3) in down-flow operation at a fixed flow rate of 5.64 L/m^2/hour. Column effluent volume was continuously measured and the TS measured at 30 minute intervals. The critical

point in which sand beds were defined as clogged was when the volume of percolate exiting the columns was 10% of influent hydraulic flow rate. This process was repeated at 7 different TS concentrations (5000, 7500, 10000, 12500, 15000, 17500, 20000 mg TS L^{-1}) and experiments were carried out in duplicate. Influent sludge was continuously stirred to ensure no settlement took place prior to being pumped into each column using a peristaltic pump (Watson Marlow, Falmouth, UK).

7.2.4.2 Batch fed solids loading

The impact of using a sand barrier when carrying out batch-fed mode operation was assessed by establishing clogging rates, TS removal and percolation rates through the sand barrier. A column (inside diameter: 0.15 m) with a bed depth 0.05 m (bed volume 884 cm^3) with a sand (0.9-1.2 mm) and gravel drainage layer (0.1 m) used. Digestate (50000 mg TS L^{-1}) was applied at 9 different hydraulic loads (68-750 L/m^2) in order to provide 9 different TS loading rates (3-30 kg TS/m^2). The percolate was collected at the base of each column with the volume and TS concentration recorded regularly throughout the 32 day experimental run for each TS loading rate.

7.2.5 Media clogging

7.2.5.1 Combined media and sacrificial sand bed

A column (inside diameter: 0.05 m) with a bed height of 0.33 m (bed volume 590 cm^3) of clinoptilolite (0.7-1.6 mm) was configured with a sacrificial sand barrier (0.9-1.2 mm) of 0.05 m depth routinely applied on top of the media (sand bed volume 98 cm^3). The sacrificial sand barrier was removed along with dewatered solids every 24 hours (application rate of 1 BV of 0.5 % TS digestate/24 hours) and replaced with a new sand barrier. According to literature regarding TS removal in sludge drying beds (Cofie et al., 2006; van Haandel and Van Der Lubbe, 2007), the sacrificial sand barrier was not expected to achieve 100% TS removal. Therefore, in order to quantify clogging of the clinoptilolite media void spaces at different bed depths samples were extracted and solid matter was washed from the media and dried at 105°C according to Nivala et al. (2012).

7.2.6 Analytical procedures

In all dynamic column experiments analysis was carried out in duplicate: NH_4-N, K and P were all determined photometrically using a Spectroquant Nova 60 (Merck-Millipore, Darnstradt, Germany). Chemical oxygen demand (COD), total solids (TS), total suspended solids (TSS), volatile solids (VS) was carried out in triplicate according to standard methods (APHA, 2005). Particle size distribution of digestate was measured through a laser diffraction particle sizer (Mastersizer 2000, Malvern Instruments, Malvern, UK). Samples were pumped (60 mL min^{-1}) by peristaltic pump (Model 505, Watson Marlow, Falmouth, UK) with the particle size distribution measurements replicated six times with the average and standard deviation values reported. The pH of all samples and solutions was measured directly by pH meter (model 3540, Jenway, Dunmow, UK).

7.3 RESULTS AND DISCUSSION

7.3.1 Influent digestate composition

The particle size distribution of digestate used in all experiments is illustrated in Figure 7.2. All particles were >0.9 µm and <158.4 µm, with d (0.1), d (0.5) and d (0.9) values of 3.638 ± 0.036, 14.001 ± 0.1799 and 57.367 ± 2.014 µm respectively, which indicates that 10%, 50% and 90% of particles measured were less than or equal to the sizes stated. The d (0.5) values are within the range reported by Houghton et al. (2002) in digested sewage sludge (30.5-69.9 µm) consisting of a range of different primary:waste activated sludge ratios, but are less than d (0.9) values (245.3-300.3 µm). This reflects the thermal hydrolysis pre-treatment step that was undertaken on the digested sludge used in this study, which breaks down larger particles present in sewage sludge through high temperatures and pressure (Higgins et al., 2011). This factor is likely to have caused the low d (0.9) values and caused a more homogenous particle size distribution in comparison to digested sewage sludge without thermal pre-treatment (Figure 7.3).
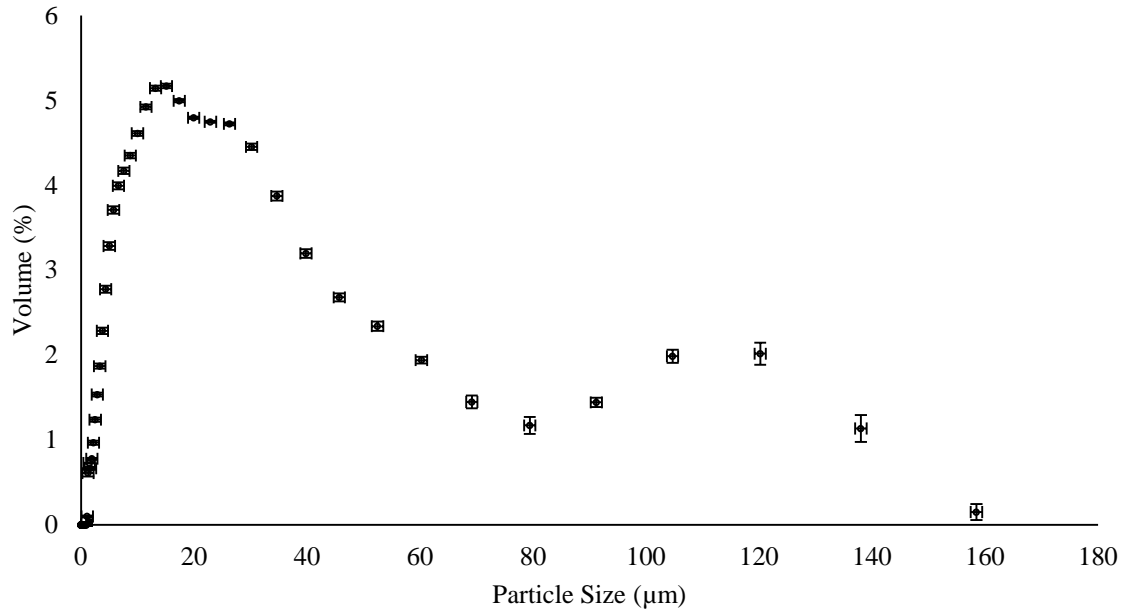
Figure 7.2 Particle size distribution of digestate from a mesophilic anaerobic digester fed with primary and waste activated sewage sludge utilising a thermal hydrolysis pre-treatment step.

7.3.2 Dynamic column studies utilising digestate and non-regenerative media

7.3.2.1 Multilayer graded media bed

The sand media bed had TS removal rates (43-65 % TS removal) within the range of biochar and clinoptilolite at all influent TS concentrations (Table 7.1). The sand filter beds in this study are therefore at the lower range of TS removal in conventional sand sludge drying beds with 60-70% TS removal (Drinan and Spellman, 2012).The graded media bed was constructed in order to provide a graded porosity throughout the bed depth and as the particles of the digestate in use were relatively fine (<57.367 ± 2.014 µm), the TS particles present had to impact on the walls of the channel and remain there by force (Matteson and Orr, 1998). This process culminates with the rapid build-up of solids on the surface of the media, which was of a similar particle size and grading between media types, providing a cake filtration effect at the surface and resultantly similar filtration characteristics between all media types (Table 7.1). Similarly, COD

removal across all media types was consistent, primarily as it is a function of TS removal, as under the short experimental time periods in which the filter beds were operational (< 10 days), biofilm growth to facilitate organic removal by attached microorganisms was not likely to have developed (Mara and Horan, 2003). The shorter operational time is also the reason why COD removal rates were lower in this study than other pilot scale sludge drying beds or filter bed systems, with 85-90% COD removal reported in a study by Cofie et al. (2006).

The ability of biochar to capture NH_4-N was limited with removal rates (13% NH_4-N removal) that were similar to sand (8% NH_4-N removal). The NH_4-N removal rate of sand and biochar only increased when the TS concentration increased (2.5% TS) as a result of clogging occurring almost immediately (0.442 and 0.579 BVs until pressure trigger reached in sand and biochar respectively), resulting in a dense sludge cake layer forming rapidly and providing increased filtration action with 73% and 61% TS removal in biochar and sand respectively at 25000 mg TS L^{-1}, in comparison to 45% and 51% TS removal at 5000 mg TS L^{-1}. In addition, the formation of a substantial cake filtration layer reduced the hydraulic flow rate of the percolate, increasing EBCT, which is beneficial for NH_4-N and other contaminant adsorption (Reungoat et al., 2012).

Table 7.1 Drying bed experiments utilising a column (id: 0.15 m, length: 1.25 m) fed with anaerobic digestate at three different TS concentrations (0.5 – 2.5 % TS). Media beds were configured with the largest particle size at the top of the bed reducing in size to the smallest media particle size at the bottom of the bed. Three different particle sizes were used in the beds; 5.4, 2 and 1 mm in char; 2.35, 1.99 and 1.5 mm in sand; and 7, 4 and 2.3 mm in clinoptilolite. All media beds had a volume of 8.83 L, a bed depth of 0.5 m and were supported by a gravel bed (6 mm particle size) of an equal proportion. EBCT of 354 mins

	(%)	(L)	(BVs/24 hours)	(%)		(%)		(%)		(g NH_4-N/m^3 media/day)		(g NH_4-N /g TS)	
Media	Influent. TS	Total BVs	BVs/day	TS removed		COD removed		NH_4-N removed		NH_4-N removed		NH_4-N removed	
Char	0.50	14.781	5.439	-0.458	(0.117)	-0.240	(0.168)	-0.128	(0.048)	165.885	(43.934)	0.684	(0.345)
Sand	0.50	11.573	11.573	-0.511	(0.101)	-0.211	(0.378)	-0.079	(0.044)	470.418	(706.889)	0.973	(0.450)
Clinoptilolite	0.50	8.176	8.176	-0.542	(0.040)	-0.253	(0.169)	-0.950	(0.084)	750.109	(94.953)	-0.996	(0.002)
Char	1.50	4.593	1.690	-0.629	(0.231)	-0.667	(0.239)	-0.216	(228.138)	171.391	(17.472)	1.680	(1.009)
Sand	1.50	2.756	1.014	-0.544	(0.270)	-0.769	(0.155)	-0.420	(173.047)	100.857	(125.148)	2.015	(1.237)
Clinoptilolite	1.50	2.930	1.078	-0.434	(0.411)	-0.864	(0.126)	-0.999	(560.327)	148.476	(148.300)	-0.997	(0.002)
Char	2.50	0.579	0.579	-0.737	*	-0.925	*	-0.985	*	583.455	*	-0.944	*
Sand	2.50	0.440	0.440	-0.619	*	-0.925	*	-0.925	*	415.719	*	-0.802	*
Clinoptilolite	2.50	0.461	0.461	-0.654	*	-0.993	*	-0.999	*	471.184	*	-0.998	*

*No data available

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The formation of a clogging layer at the surface of the media bed, commonly referred to as the schmutzdecke in slow sand and intermittent sand filtration (Muhammad and Hooke, 2003), is where complex microbial communities form in a biofilm and it is through this that the majority of NH_4-N removal takes place (Gimbel et al., 2006). This is reflected in the higher removal rates in literature values for intermittent sand filtration systems (85-99% NH_4-N removal (U.S. EPA, 1999; Rodgers et al., 2005; Healy et al., 2007)) in comparison to the sand filter used in this study (8-42% NH_4-N removal). However, as this study was carried out at much higher hydraulic and TS loading rates (5000-25000 mg TS L^{-1}) (EBCT 354 minutes), and over a shorter time period (<10 days) this did not allow time for biological nitrogen removal to take place and can explain the reduced NH_4-N removal rates by sand beds in this study.

However, NH_4-N recovery relies on biological nitrogen removal not taking place, with alternate uptake mechanisms used by non-regenerative media. It was previously reported that the NH_4-N uptake capacity of biochar is 5 mg NH_4-N/g at a 200 mg NH_4-N L^{-1} concentration under batch conditions (Rose et al. 2015) and 3-5.3 mg NH_4-N /g dynamic conditions (Sarkhot et al., 2013). However, when under dynamic experimental conditions with the presence of TS in the percolate (357-3019 mg TS L^{-1}), the performance of biochar was significantly hindered (Table 7.2). As the main mechanism for NH_4-N adsorption by biochar is physical adsorption (Halim et al., 2010), in a filter bed system, when digestate is applied, the TS matter in the percolate clogs the porous structure of the biochar and prevents the physical uptake of NH_4-N by pores in the media. This makes biochar an unsuitable media for applications in, for example, an integrated nutrient recovery sludge drying bed, where substantial interference from TS in the percolate will take place.

Clinoptilolite's NH_4-N removal efficiency was substantial at all TS loading rates (5000, 15000, 25000 mg TS L^{-1}) with average removal rates of 95%, 99% and 99% NH_4-N respectively (Figure 7.3), indicating that the ion exchange process was not adversely impacted by the high solids nature of digestate percolate. Removal rates increased following day one (Figure 7.3) due to the blocking filtration action of TS filling media void spaces, followed by the partial onset of cake filtration. The high removal rate in the graded bed configuration (95-99% NH_4-N removal) concurs with findings that the predominant mechanism for NH_4-N recovery in clinoptilolite is cation exchange (Demir et al., 2002; Hankins et al., 2005; Wang et al., 2008), rather than adsorption, and the effect of TS on the surface charge density of the clinoptilolite particles is not detrimental to the performance of the clinoptilolite. For this reason, clinoptilolite is a robust media and is highly suitable for NH_4-N and K^+ recovery in a sludge drying bed configuration. Nevertheless, low hydraulic loading rates, before clogging of the media bed occurred (0.46-8.18 total BVs), were still prevalent despite operation at long EBCT (354 minutes) (Table 7.2). This is significant due to the substantial potential capacity of clinoptilolite: with a capacity of 60 g NH_4-N /kg and >80 bed volumes of percolate required before full capacity of the media is obtained at an influent concentration of 200 mg L^{-1} (Chapter 6).
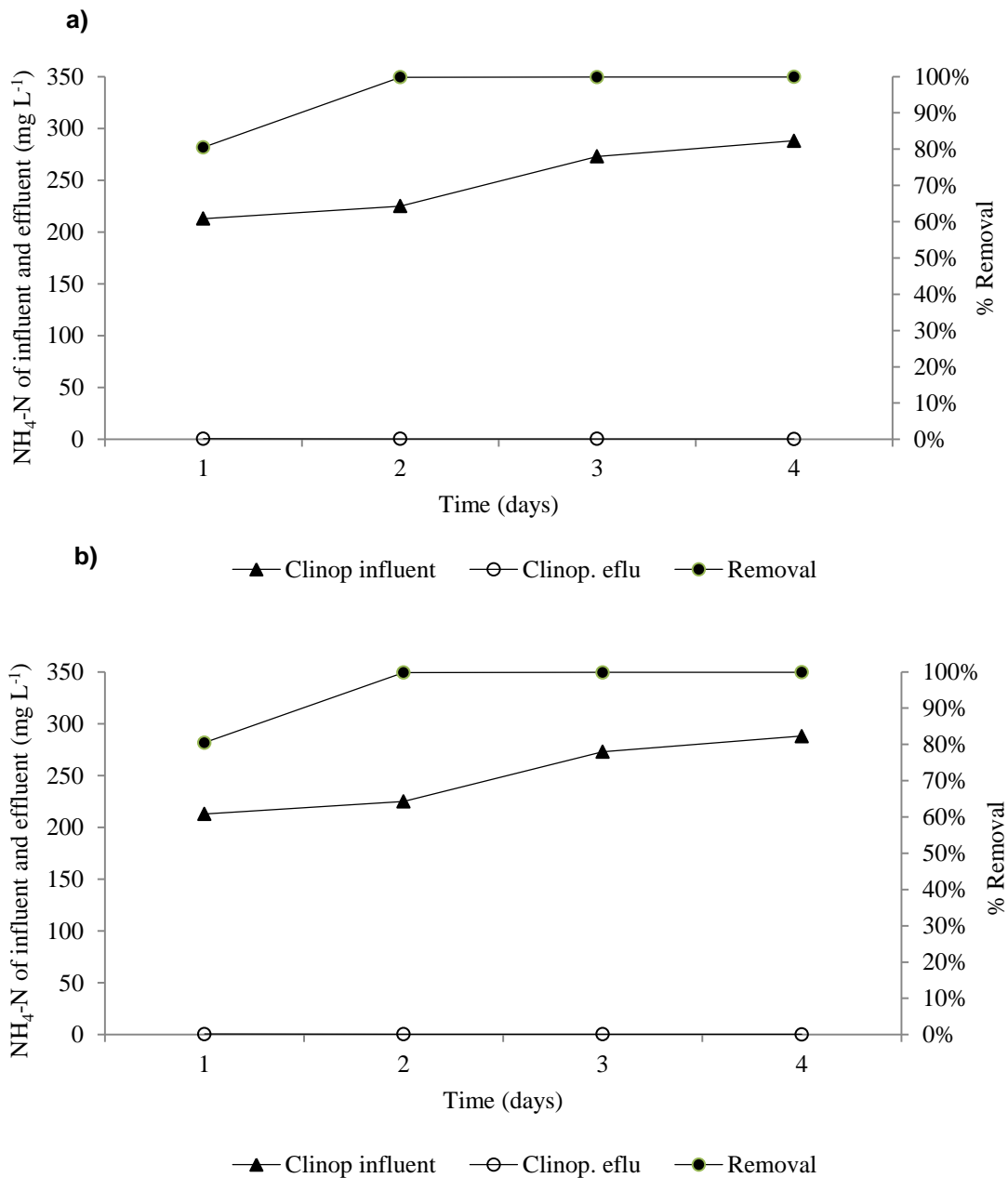
Figure 7.3 a) Influent, effluent and % removal by clinoptilolite (graded particle size media bed with larges media size on the top of the bed: 7, 4 and 2.3 mm , bed depth of 0.5m) with a flow rate of 3.01 BVs/day and 8.17 BVs in total being fed by influent TS concentration of 5000mg.L. b) NH$_4$-N removal by clinoptilolite media (particle size of 7mm, bed depth 0.5m) with a flow rate of 4.083 BVs/day and 15.3 bed volumes in total being fed by influent TS concentration of 5000 mg.L^{-1}.

7.3.2.2 Single layer media bed

In order to minimise media clogging and maximise contact between the sludge and media, the largest clinoptilolite particle size (7-9 mm) was utilised (Table 7.2). At the lowest TS loading rate (5000 mg TS $^{-1}$), the largest volume of sludge was treated (12.39 BVs) but with the lowest NH_4-N recovery efficiency (62% removal). However, at the highest TS loading rate (25000 mg TS L^{-1}), the greatest NH_4-N recovery efficiency was reached (99%) but a reduced volume was treated (total 0.87 BVs). This is due either to the onset of cake filtration at the media: sludge interface or the blocking of void spaces throughout the media bed occurring quicker with the high solids effluent. This has the effect of increasing the pressure drop within the column and reducing the hydraulic flow rate of percolate through the media bed, resulting in a longer EBCT, which increases media uptake rates (Rose et al. 2015). Although NH_4-N recovery efficiency was substantial at all TS loading rates, the total bed volumes (BVs) of percolate treated (0.88 – 12.39 BVs) were substantially lower than the BVs of percolate required to reach exhaustion of the clinoptilolite media (50-183 total BVs dependent on EBCT (Rose et al. 2015)). This factor promotes the need for either a TS reduction process prior to the media bed or an integrated TS barrier to prevent clogging of the bed and allow the full utilisation of the media to take place.

Table 7.2 Average removal efficiency of clinoptiolite beds fed with anaerobic digestion effluent at a range of TS concentrations (0.5 – 2.5 %) with a media configuration of a single fixed bed of clinoptilolite with a particle size of 7-9mm. The media bed had a volume of 8.83 L, a bed depth of 0.5 m and were supported by a gravel bed (6 mm particle size) of an equal proportion. EBCT of 354 mins.

(%)	(L)	(L/24 h)	% (-)		% (-)		% (-)		(g NH_4-N /m3 media.d)		% (-) (g NH_4-N /g TS)		% (-)		% (-)	
Influent TS	Total BVs	BVs/d	TS removed		COD removed		NH_4-N removed		NH_4-N removed		NH_4-N removed		P removal		K removal	
2.5	0.875	0.322	88.194	(0.039)	90.425	(0.054)	99.799	(0.002)	646.578	(337.945)	97.530	(0.321)	94.585	(0.027)	92.144	(0.068)
1.0	3.062	1.127	34.787	(0.037)	50.144	(0.025)	99.974	(0.000)	621.723	(23.565)	99.964	(0.000)	84.205	(0.006)	27.682	(0.177)
0.5	12.393	4.560	38.574	(0.227)	46.712	(0.197)	62.671	(0.057)	447.686	(142.542)	31.918	(0.231)	64.986	(0.405)	57.111	(0.153)

7.3.3 Sacrificial solids barrier to protect media bed

7.3.3.1 The onset of cake filtration under continuous flow conditions

The critical point at which the onset of cake filtration occurred (percolate <10% influent hydraulic flow rate) in the sand barrier ranged from 0.070-0.320 kg TS/m^2 with the smaller sand particle size (0.6-0.9 mm) and from 0.046-0.500 kg TS/m^2 when a larger sand particle size was used (0.9-1.2 mm) dependent on digestate TS concentration (0.5-2% TS) (Figure 7.5). The flow of TS matter through a sacrificial sand filter, as well as the subsequent rate at which cake filtration commences, is therefore dependent on the particle size of the sand. However, the particle size distribution of TS in the digestate is also important: as the particle size of the digestate is fine (<158.4 µm) and highly homogenous (Figure 7.2), physiochemical filtration is therefore likely to predominate over mechanical filtration, which prevails when larger particles are present (Herzig et al., 1970). The time for a sludge cake filtration layer to develop is within the reported range of TS loading rates for sludge drying beds (0.18 kg TS/m^2/application according to Tchobanoglous et al. (2003)), indicating that a mixture of both physical filtration by sand and sludge cake filtration occurs within a conventional sludge drying bed.

7.3.3.2 Solids removal before and after the onset of cake filtration

The mean TS removal rate of the sand barrier was 48% and 44% in the small and large sand particle size respectively (Figure 7.5). The difference in critical clogging points between different particle sizes (average values of 0.12 and 0.06 kg TS/m^2 in the large and small sand particle size respectively) can be attributed to the difference in void size as it will require an increased mass of TS in order to bridge larger pore sizes (0.9-1.2 mm sand) than smaller pore sizes (0.6-0.9 mm sand). The TS removal before the onset of cake filtration was within a narrow range across media sizes and the range of influent TS concentrations (37-56 % removal). Similarly, following the commencement of cake filtration the TS concentration was consistent across all influent TS concentrations independent of influent TS concentrations with values in the percolate between 1193-2653 mg TS L^{-1}(Figure 7.4). This differs to findings by

Wett et al. (2005) who reported increased filtration efficiency at higher influent TS concentrations in the filtration of sewage sludge by geotextiles. The difference to this study can be accounted for by the different filter mediums used and also by the difference in particle size distribution of the sewage sludge used, which was from a septic tank system which will have a larger more heterogeneous particle size distribution (Vincent et al., 2011) than the sludge used in this study, meaning that the onset of cake filtration is likely to be reached quicker.
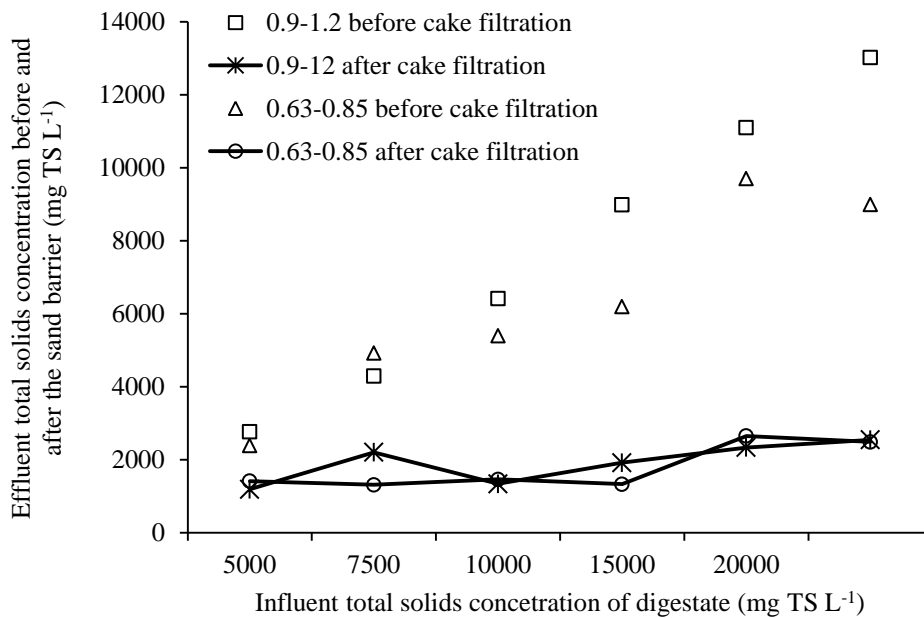

Figure 7.4 Total solids concentration of sand filter barrier (particle size 0.63-0.85 and 0.9-1.2 mm: 0.05 m bed depths) effluent before and after the onset of cake filtration (defined as when effluent percolate flow is < 10% of influent flow. Influent flow was consistent at 22.5 L/m^2/hour and was repeated at a range of TS concentrations (5000-20000 mg TS L^{-1}).

7.3.3.3 The onset of cake filtration in clinoptilolite

The particle size of the media directly impacts the NH_4-N adsorption capacity of the media (Rose et al. 2015); however, the particle size will also affect the speed in which a sludge cake layer will form on the media surface due to increases in particle void space. Clinoptilolite with a smaller particle size (0.7-1.6 mm), had a reduced TS loading capacity before the onset of cake filtration (with an influent 10000 mg TS L^{-1} concentration), than the larger (7-9 mm) media size (0.23 and 0.43 kg TS/m^2 respectively). The larger clinoptilolite particle size (7-9 mm) could therefore be loaded with 48% more TS than the smaller particle size (0.7-1.6 mm) before the onset of cake filtration. It was similarly observed by Brune et al. (1994) that finer grained material was more susceptible to clogging than coarser grained material when studying particle size ranges of 2-4,4-8, 8-16, 16-32 mm. Values of this study regarding clinoptilolite clogging (0.7-1.6 mm) are comparable to values of clogging in sand of 0.9-1.2 mm and 0.6-0.9 mm particle size (0.43 and 0.11 kg TS/m^2 respectively), demonstrating that over the initial period of filtration, the filtering mechanisms, through straining (fluid and gravitational forces) of clinoptilolite, are similar to that of sand when the same hydraulic and TS loading is applied.

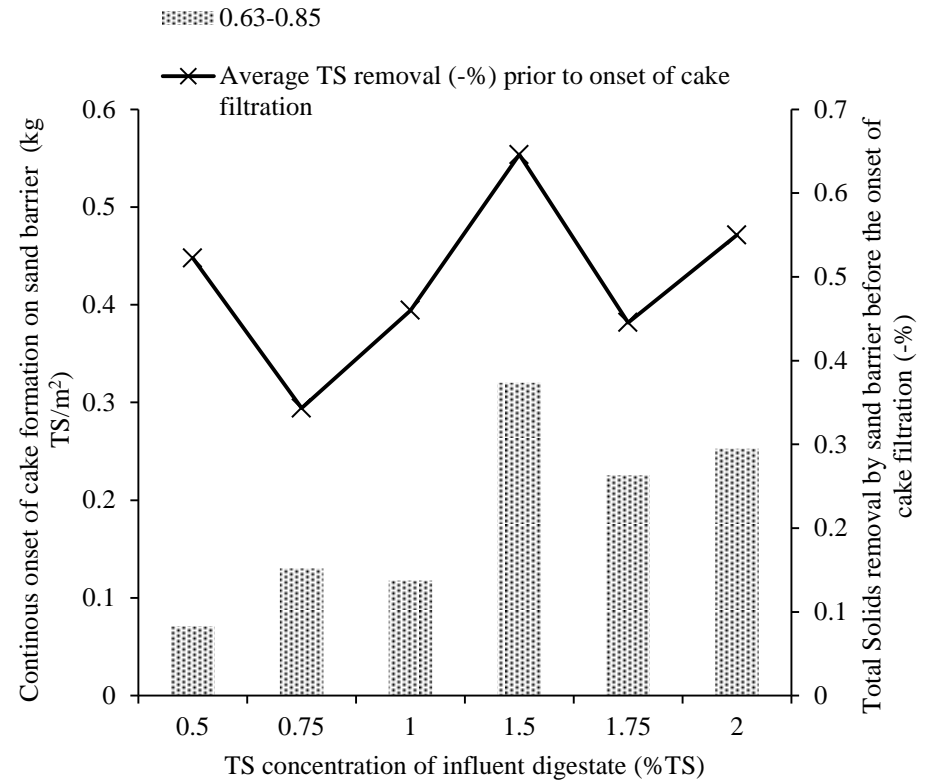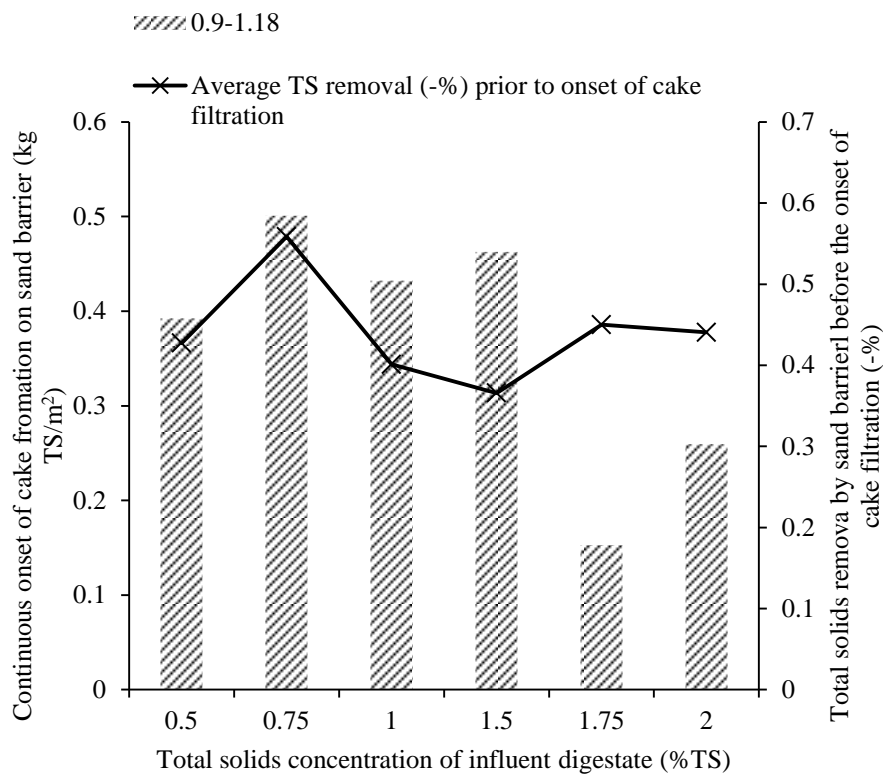**Figure 7.5 The total solids loading on a sand filter barrier (particle size 0.63-0.85 and 0.9-1.2 mm: 0.05 m bed depths) before the onset of cake filtration (effluent percolate flow rate is < 10% of influent flow rate). Influent flow rate remained at 22.5 L/m²/hour and was repeated at TS concentrations between 5000-20000 mg/L TS. Average TS removals before the onset of cake filtration are illustrated for each TS concentration.**

7.3.3.4 Batch fed solids loading of sacrificial sand barrier

The physical process of filtration by the sand barrier has an idealised blocking filtration model shape (Figure 7.6), meaning that if the flow rate is constant, the pressure drop increases exponentially with the quantity filtered, with the number of void spaces approaching zero (Ripperger et al., 2012). Following initial blocking filtration, cake filtration subsequently commences and it is assumed that TS is deposited on the filter medium as a homogenous porous layer with a constant permeability (Sparks, 2011). Therefore, if flow rate is constant, the pressure drop will increase linearly, proportional to the quantity of solids deposited (Figure 7.6).

Cumulative percolate volumes varied according to TS loading with a range of 35-167 L/m^2 (Figure 7.7). A spike in the concentration of the effluent TS can be observed on day one of the 32 day percolation cycle, however, once cake filtration has commenced, effluent TS concentrations remain consistent and within a narrow range (3532-13791 mg TS L^{-1}) (Figure 7.7). Greater than 50% of total percolation occurred on average within 27 hours (range of 15-60 hours), however, during this time period the highest concentrations of TS in the effluent were also present (Figure 7.7). This represents the time when blocking filtration is ongoing and physical filtration by the sand is the predominant mechanism. However, after cake filtration has commenced, this becomes the predominant method of filtration, with greater TS removal but at a reduced hydraulic rate (Figure 7.7).

The rate of percolation is therefore dependent on the TS application rate (Figure 7.7) but once cake filtration has commenced it will be independent of the particle size of the sand (or media) bed due to the increase in pressure drop that is a result of a build-up in the cake filtration layer. Total percolation volumes were less than theoretical values of dewatering times stated by Van Haandel and Van Der Lubbe (2007), who calculated that 75% of the water content is lost through percolation in sand sludge drying beds and it was estimated by Wang et al. (2010) that 60-80% of sludge water is drainable dependent on the extent of digestion that the influent sludge has undergone. Therefore, the wide range

of applied water collected in the percolate (10% - 71% of the water content applied) in this study is at the lower end of these theoretical values, largely due to the wide range in TS loading rates (3-30 kg TS/m^2) utilised in this study. However, in pilot scale studies of sludge drying beds by Cofie et al. (2006) it was reported that between 39-79% of sludge loaded on drying beds was present in the percolate fraction. These values are within those of this study and illustrate the varied nature of dewatering in the drying bed process.

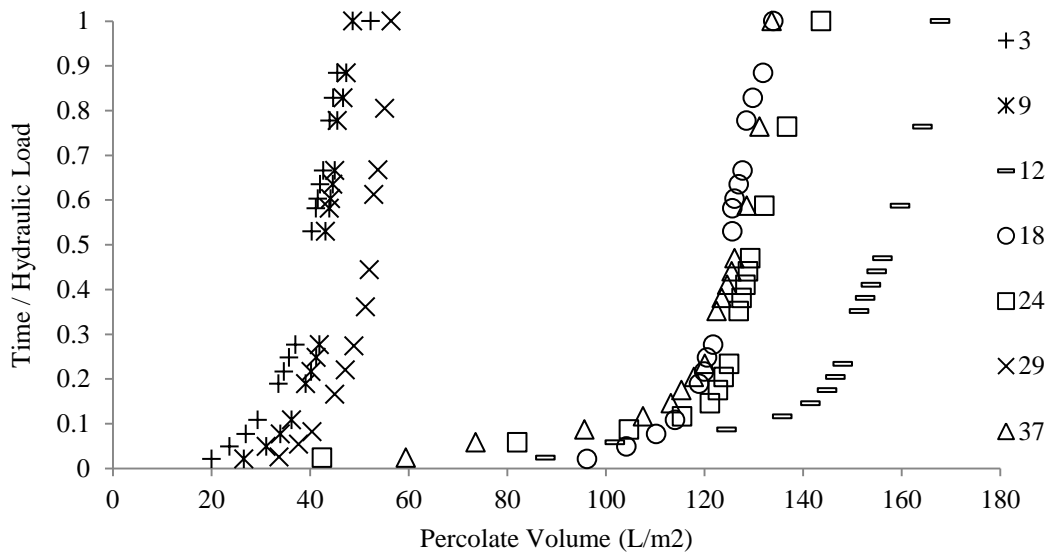**Figure 7.6 The physical filtration characteristics of a sand (0.9-1.2 particel size) filter barrier (0.05 m bed depth) batch fed with digestate at solids loading rates between 3-37 kg TS/m^2.**
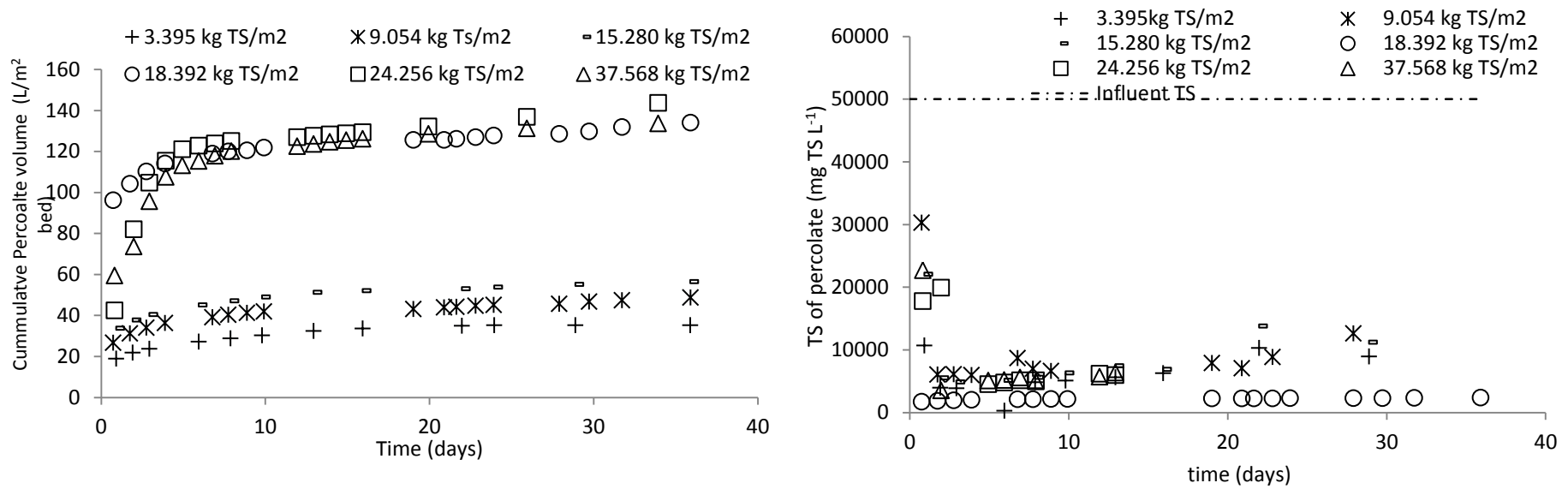
Figure 7.7 Batch fed percolation rate through sand bed (inside diameter: 0.05 m, bed height: 0.05 m) with a particle size 0.9-1.2 mm with varying hydraulic batch loads of anaerobic digestion effluent at TS.

7.3.3.5 Solids accumulation within integrated sand barrier and media bed

A clinoptilolite media bed with a sacrificial sand barrier was utilised in down-flow experiments. After 25 BVs of sludge (0.5% TS) the clinoptilolite bed was defined as clogged, due to percolation not being complete after 24 hours. The removal efficiency of NH_4-N by the sacrificial sand bed and clinoptilolite within the column was > 99%, with 5 mg NH_4-N/g media recovered. This value is still significantly lower than the theoretical operational capacity without solids interference (60 mg NH_4-N/g media (Chapter 6)), due to there being significant unused capacity of media in the bed before clogging took place. Accumulation of TS within the vertical profile of the bed illustrates that there was a positive linear correlation between TS accumulation and bed depth ($r = 0.949$, $p = 0.03$). This factor suggests that the majority of TS was being washed through the media bed and accumulating at the base of the bed where there was increased resistance to TS movement out of the column effluent port. This corresponds to work by Rowe et al. (2000) who found that the greatest accumulation of clogging materials (organic and inorganic) was near the leachate collection pipe in experimental work examining landfill leachates. However, results of the present study are in contrast to findings by Lianfang et al. (2009), who reported the majority of clogging occurred within the top 0.15 m of the bed depth in lab scale column experiments simulating clogging of a vertical flow constructed wetland made up of coarse sand above gravel. This difference can again be attributed to the particle size distribution of the digestate in use: consisting of fine particle sizes, with d (0.9) values of 57.367 ± 2.014 µm, in comparison to other types of sewage sludge and wastewater commonly applied to wetlands.

7.3.4 Design Considerations

7.3.4.1 Mechanisms and trade-offs in sludge drying beds and nutrient recovery

Sludge drying beds have a notoriously large footprint with low TS loading rates of 120-150 kg TS/m^2/year for the treatment of digested primary sludge (Tchobanoglous et al., 2003) and 196 -321 kg TS/m^2 for faecal sludge in Ghana (Cofie et al., 2006). Reducing footprint and increasing the rate of treatment is also essential in achieving nutrient recovery, as over conventional drying bed cycles (2-6 weeks according to Wang et al. (2010)) there is the potential for nitrification to commence (Mara and

Horan, 2003) which would not recover the N present in the percolate, which is the primary objective. The main mechanisms of dewatering in sludge drying beds can be described through filtration theory (Sparks, 2011; Ripperger et al., 2012): dewatering commences through rapid filtration action by the drying bed media (Figure 7.5). This is followed by blocking filtration until the onset of cake filtration (Figure 7.6), at which point the dewatering rate slows substantially (Figure 7.7).

Clinoptilolite proved a resilient media that can operate effectively despite high TS (1193-2653 mg TS L^{-1}) and competing cation concentrations (mean electrical conductivity value of 16746 µS/cm) in the percolate of drying beds (with 62-99% NH_4-N recovery rates). However, the use of non-regenerative passive media relies on increasing the media value, which only occurs if clinoptilolite is fully saturated (>80 BVs according to Rose et al. (2015)). The full saturation of media was not achieved in any of the configurations tested in this study without utilising a sand barrier, with a maximum of 14.7 bed volumes treated. It is consequently evident that the limiting factor of the proposed system is not the performance and capacity of the media but instead it is a hydraulics issue of enabling sufficient contact between the percolate and the media without the media bed becoming clogged.

7.3.4.2 Process options for nutrient recovery and sludge drying beds

7.3.4.2.1 Clinoptilolite as a direct replacement to sand in sludge drying beds

The performance of clinoptilolite as a filter medium was equally comparable to sand in regards to TS, COD and P removal (Table 7.2), consequently, clinoptilolite could directly replace sand in sludge drying beds, with the benefit of recovering NH_4-N and K^+. However, this would present inefficiencies in unused media capacity. The use of non-regenerative media is only beneficial if the product can be suitably enriched before re-sale (Cucarella et al., 2007; Kõiv et al., 2012). Therefore, if media is being removed before its full capacity has been reached, this would no longer present a cost efficient proposal. It is for this reason that either a prior solid/liquid separation process step, for instance waste stabilisation ponds, or the use of a low cost barrier to reduce the movement of TS into the media bed, is necessary to the integrated nutrient recovery sludge drying bed design.

7.3.4.2.2 Integrated nutrient recovering sludge drying bed and sacrificial sand barrier

Other researchers have achieved the intensification of sludge drying beds through the use of increased solar drying conditions: for example, researchers have investigated the use of greenhouse solar drying and enhancing ventilation to improve drying times (Seginer et al., 2007) and others such as Radaidah et al. (2011) achieved a 30% reduction in drying times through the use of solar heated water pipes running through conventional sludge drying beds. These methods of intensification all predominate around the evaporation step and the removal of bound water from the sludge through increased evaporation. However, digestate is predominantly composed of free water as AD causes bound water to be partly converted to free water due to decomposition and cell lysis (Luo et al., 2013). The high fraction of free water is reflected in this study by the high rates of percolation achieved (Figure 7.7) when minimal evaporation was taking place due to the sealed nature of the filter columns (Figure 7.1). Therefore, the intensification of drying beds can also be achieved through de-coupling the two main processes of free water removal in sludge dewatering: initial percolation, in which over 50% of total percolate volume occurs on average over the first 1.14 days, and the subsequent cake filtration step, where percolation rates are substantially reduced (Figure 7.7a). Bound water in digestate will not contribute to the nutrient recovery process and could subsequently be carried out in a separate conventional drying bed as this relatively small water fraction is harder to separate and evaporation or disruption of the cells is required for moisture removal.

If semi-dewatered solids (15-25 % TS), with the remaining bound water present, were scraped from the surface of the sacrificial sand barrier following completion of the initial percolation step (0.5 kg TS/m^2: Figure 7.5), then loading rates could be increased by 35 % allowing greater saturation of the clinoptilolite bed before the commencement of biological nitrogen removal. A sacrificial sand barrier would provide a semi-dewatered material containing phosphorus (4.28% P TS) and carbon (32% C TS), providing benefits to agricultural soils such as improving microbial communities, micronutrient availability and soil structure (Lu et al., 2012). There is, subsequently, the potential benefit of co-application of dewatered solids with the saturated clinoptilolite beneath the sand barrier, creating a balanced fertiliser product

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with consistently high and controlled NH_4-N and K^+ content which is often a limitation in the valorisation of dewatered digestate (Antille et al., 2013).

The use of a sacrificial sand bed with the scraping of semi-dewatered solids after the initial percolation step is complete, provides multiple benefits. It would allow process intensification by utilising TS application rates of 3 kg TS/m^2/day (Figure 7.7) and utilising daily scraping of the bed surface, TS loading rates could be increased from conventional loading rates of 120-150 kg TS/m^2/year (Tchobanoglous et al., 2003) to 1095 kg TS/m^2/year. A sacrificial sand bed also prevents an inhibitory pressure drop occurring from TS blocking media void spaces, reducing TS movement into the clinoptilolite bed (34-65 % TS removal in continuous fed systems and 65-98% TS removal in batch fed operation). In addition, in batch fed systems, a sand barrier will reduce the hydraulic flow of digestate through the media bed beneath, improving NH_4-N recovery due to an increase in EBCT.

However, it should be noted that through the intensification of sludge drying beds, the removal of the cake filtration layer is likely to cause a decrease in the process performance of drying beds, as TS reduction is substantially greater once cake filtration has commenced (Figure 7.4). This factor was similarly observed by Koné et al. (2007) who reported reductions from 5600 to 3600 mg COD L^{-1} and from 600 to 290 mg TSS L^{-1} between the first day and last day of a drying cycle on pilot scale sludge drying beds. The cake filtration component is therefore an attribute in regards to pollutant removal but a constraint in regards to loading rates and nutrient recovery.

Despite this, the regular removal and reapplication of a sacrificial sand bed will be beneficial in regards to reductions in drying bed performance following repeat applications. For instance, Cofie et al. (2006) reported a dewatering time of 11 days on the third application cycle, which was only 73% and 50% complete after the same time in subsequent cycles 4 and 5. The use of a sacrificial sand bed could, therefore, be used to reduce footprint and increase the rate of nutrient recovery and is a feasible means of extending bed life over repeat applications. This would in turn allow complete nutrient recovery to take place with both the solid and liquid percolate fractions fully utilised.

7.4 CONCLUSIONS

This study has conducted a series of experiments in order to establish the design of an integrated sludge drying bed and nutrient recovery system utilising non regenerative media.

- Clinoptilolite can effectively be used as part of a sludge drying bed configuration to capture NH_4-N from digestate percolate under high solids loading conditions and the saturated media can be used directly as a high value fertiliser product presenting a complete nutrient recovery process in an integrated nutrient recovery drying bed design.

- Dewatering in sludge drying beds commences through rapid filtration action by the drying bed media. This is followed by blocking filtration until the onset of cake filtration occurs. As a result, matching the dewatering mechanisms of sludge drying beds to the uptake capacity of clinoptilolite presents the primary challenge in balancing TS loading rates and nutrient recovery.

- The use of a sacrificial sand bed, that can be scraped away with the accumulated TS after initial blocking filtration is complete, will substantially reduce the large footprint of conventional sludge drying beds and enable the full saturation of the clinoptilolite media bed to take place.

- The combined dewatered solids (high in P) can then be blended with the media beneath (saturated in NH_4-N and K^+) to create an enriched biosolids product with balanced nutrient ratios for agricultural application.

7.5 REFERENCES

Abel, P. D. (1996), *Water Pollution Biology,* CRC Press, Boca Raton, USA.

Antille, D. L., Sakrabani, R., Tyrrel, S. F., Le, M. S. and Godwin, R. J. (2013), "Characterisation of organomineral fertilisers derived from nutrient-enriched biosolids granules", *Applied and Environmental Soil Science,* vol. 2013.

APHA (2005), *Standard Methods for the Examination of Water and Waste Water,* American Public Health Association, Baltimore, U.S.A.

Brester, A. R., Coulomb, I., Deak, B., Matter, B., Saabye, A., Spinosa, L. and Utvik, A. O. (1998), *Sludge Treatment and Disposal, Management Approaches and Experiences,* , Environmental Issue Series, European Environmental Agency, Copenhagen, Denmark.

Brune, M., Ramke, H. G., Collins, H. J. and Hanert, H. H. (1994), "Incrustation problems in landfill drainage systems", *Landfilling of Waste: Barriers,* E & FN Spon, London, UK.

Chen, G., Yue, P. L. and Mujumdar, A. S. (2002), "Sludge dewatering and drying", *Drying Technology,* vol. 20, no. 4-5, pp. 883-916.

Cofie, O. O., Agbottah, S., Strauss, M., Esseku, H., Montangero, A., Awuah, E. and Kone, D. (2006), "Solid–liquid separation of faecal sludge using drying beds in Ghana: Implications for nutrient recycling in urban agriculture", *Water Research,* vol. 40, no. 1, pp. 75-82.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd Water Research Conference,* Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Cucarella, V., Zaleski, T., Mazurek, R. and Renman, G. (2007), "Fertilizer potential of calcium-rich substrates used for phosphorus removal from wastewater", *Polish Journal of Environmental Studies,* vol. 16, no. 6, pp. 817.

Demir, A., Gunay, A. and Debik, E. (2002), "Ammonium removal from aqueous solution by ion-exchange using packed bed natural zeolite", *Water SA,* vol. 28, no. 3, pp. 329-336.

Dodane, P. and Ronteltap, M. (2014), "Unplanted Drying Beds", *Faecal Sludge Management: Systems Approach for Implementation and Operation,* Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.),1st ed, IWA Publishing, London, UK

Drinan, J. E. and Spellman, F. (2012), *Water and wastewater treatment: a guide for the nonengineering professional,* Crc Press, Boca Raton, USA.

FAO (2015), *World fertilizer trends and outlook to 2018,* 1st ed, Food and Agricultural Organisation of the United Nations, Rome, Italy.

Fytili, D. and Zabaniotou, A. (2008), "Utilization of sewage sludge in EU application of old and new methods-A review", *Renewable and Sustainable Energy Reviews,* vol. 12, no. 1, pp. 116-140.

Gimbel, R., Graham, N. J. D. and Collins, M. R. (2006), *Recent progress in slow sand and alternative biofiltration processes,* IWA Publishing, London, UK.

Guest, J. S., Skerlos, S. J., Barnard, J. L., Beck, M. B., Daigger, G. T., Hilger, H., Jackson, S. J., Karvazy, K., Kelly, L. and Macpherson, L. (2009), "A new planning and design paradigm to achieve sustainable resource recovery from wastewater 1", *Environmental Science and Technology,* vol. 43, no. 16, pp. 6126-6130.

Gutterer, B., Sasse, L., Panzerbieter, T. and Reckerzügel, T. (2009), "Decentralised wastewater treatment systems (DEWATS) and sanitation in developing countries", *A practical guide, Publishing House: Borda Bremen-Germany,* , pp. 20-22.

Halim, A. A., Aziz, H. A., Johari, M. A. M. and Ariffin, K. S. (2010), "Comparison study of ammonia and COD adsorption on zeolite, activated carbon and composite materials in landfill leachate treatment", *Desalination,* vol. 262, no. 1, pp. 31-35.

Hankins, N., Pliankarom, S. and Hilal, N. (2005), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

Healy, M. G., Rodgers, M. and Mulqueen, J. (2007), "Treatment of dairy wastewater using constructed wetlands and intermittent sand filters", *Bioresource Technology,* vol. 98, no. 12, pp. 2268-2281.

Heffer, P. and Prud'homme, M. (2013), "Fertilizer outlook 2013–2017", May 2013, Chicago, Illinois, USA, Paper No.: A/13/78, pp. 8.

Heinss, U., Larmie, S. A. and Strauss, M. (1999), *Characteristics of faecal sludges and their solids-liquid separation,* 1, EAWAG/SANDEC, Zurich, Switzerland.

Herzig, J., Leclerc, D. and Goff, P. L. (1970), "Flow of suspensions through porous media—application to deep filtration", *Industrial and Engineering Chemistry,* vol. 62, no. 5, pp. 8-35.

Higgins, M., Murthy, S. N., Schafer, P., Cooper, A., Kasirga, E., Lee, K., Machisko, J., Fountain, P. and Kelleher, K. (2011), "Dewatering Characteristics of Cambi Thermal Hydrolysis Biosolids: Centrifuges vs. BFPs", *Proceedings of the Water Environment Federation,* vol. 2011, no. 17, pp. 678-684.

Holm-Nielsen, J. B., Al Seadi, T. and Oleskowicz-Popiel, P. (2009), "The future of anaerobic digestion and biogas utilization", *Bioresource Technology,* vol. 100, no. 22, pp. 5478-5484.

Houghton, J. I., Burgess, J. E. and Stephenson, T. (2002), "Off-line particle size analysis of digested sludge", *Water Research,* vol. 36, no. 18, pp. 4643-4647.

Kõiv, M., Ostonen, I., Vohla, C., Mõtlep, R., Liira, M., Lõhmus, K., Kirsimäe, K. and Mander, Ü (2012), "Reuse potential of phosphorus-rich filter materials from subsurface flow wastewater treatment filters for forest soil amendment", *Hydrobiologia,* vol. 692, no. 1, pp. 145-156.

Koné, D. and Strauss, M. (2004), "Low-cost options for treating faecal sludges (FS) in developing countries–Challenges and performance", *9th International IWA Specialist Group Conference on Wetlands Systems for Water Pollution Control and to the 6th International IWA Specialist Group Conference on Waste Stabilisation Ponds,* Vol. 27, 27 Sept. – 1 Oct., 2004, Avignon, France, IWA, London, UK, .

Kone, D., Cofie, O., Zurbrügg, C., Gallizzi, K., Moser, D., Drescher, S. and Strauss, M. (2007), "Helminth eggs inactivation efficiency by faecal sludge dewatering and co-composting in tropical climates", *Water Research,* vol. 41, no. 19, pp. 4397-4402.

Kowaljow, E., Mazzarino, M. J., Satti, P. and Jiménez-Rodríguez, C. (2010), "Organic and inorganic fertilizer effects on a degraded Patagonian rangeland", *Plant and Soil,* vol. 332, no. 1, pp. 135-145.

Lianfang, Z., Wei, Z. and Wei, T. (2009), "Clogging processes caused by biofilm growth and organic particle accumulation in lab-scale vertical flow constructed wetlands", *Journal of Environmental Sciences,* vol. 21, no. 6, pp. 750-757.

Lima, A. F., Da Costa, M. C., Ferreira, D. C., Richter, E. M. and Munoz, R. A. A. (2015), "Fast ultrasound-assisted treatment of inorganic fertilizers for mercury determination by atomic absorption spectrometry and microwave-induced plasma spectrometry with the aid of the cold-vapor technique", *Microchemical Journal,* vol. 118, no. 0, pp. 40-44.

Lu, Q., He, Z. L. and Stoffella, P. J. (2012), "Land application of biosolids in the USA: A review", *Applied and Environmental Soil Science,* vol. 2012, no. Article ID 201462, pp. 1-11.

Luo, F., Dong, B., Dai, L., He, Q. and Dai, X. (2013), "Change of thermal drying characteristics for dewatered sewage sludge based on anaerobic digestion", *Journal of Thermal Analysis and Calorimetry,* vol. 114, no. 1, pp. 307-312.

Mara, D. and Horan, N. J. (2003), *Handbook of water and wastewater microbiology,* Academic press.

Martin, J. H. (2005), *An evaluation of a Mesophilic, modified plug flow anaerobic digester for dairy cattle manure,* EPA Contract No. GS 10F-0036K Work Assignment/Task Order 9, Eastern Research Group, Inc, Lexington, USA.

Matteson, M. J. and Orr, C. (1998), *Filtration: Principles and practices. 2,* 2nd ed, PennWell Publishing Corp., Tulsa, OK (United States).

McCarty, P. L., Bae, J. and Kim, J. (2011), "Domestic wastewater treatment as a net energy producer–can this be achieved?", *Environmental Science and Technology,* vol. 45, no. 17, pp. 7100-7106.

Møller, H. B., Nielsen, A. M., Andersen, G. H. and Nakakubo, R. (2006), "Process performance of biogas plants integrating pre-separation of manure", *DIAS report,* pp. 125.

Mormile, M. R., Romine, M. F., Garcia, M. T., Ventosa, A., Bailey, T. J. and Peyton, B. M. (1999), "Halomonas campisalis sp. nov., a denitrifying, moderately haloalkaliphilic bacterium", *Systematic and Applied Microbiology,* vol. 22, no. 4, pp. 551-558.

Muhammad, N. and Hooke, A. M. (2003), "Biomass characterization of slow sand filtration schmutzdecke and its effects on filter performance", *Environmental Technology,* vol. 24, no. 1, pp. 43-50.

Nivala, J., Knowles, P., Dotro, G., García, J. and Wallace, S. (2012), "Clogging in subsurface-flow treatment wetlands: measurement, modeling and management", *Water Research,* vol. 46, no. 6, pp. 1625-1640.

Paavola, T. and Rintala, J., (2008), *Effects of storage on characteristics and hygienic quality of digestates from four co-digestion concepts of manure and biowaste.*

Park, S. and Kim, M. (2015), "Innovative ammonia stripping with an electrolyzed water system as pretreatment of thermally hydrolyzed wasted sludge for anaerobic digestion", *Water Research,* vol. 68, pp. 580-588.

Parsons, S., Wall, F., Doyle, J., Oldring, K. and Churchley, J. (2001), "Assesing the potential for struvite recovery at sewage treatment works", *Environmental Technology,* vol. 22, no. 11, pp. 1279-1286.

Radaidah, J. A. and Al-Zboon, K. K. (2011), "Increase the efficiency of conventional sand drying beds by using intensive solar energy: a case study from Jordan", Vol. 7, 26-28 February, Singapore, IACSIT Press, pp. 389.

Reungoat, J., Escher, B., Macova, M., Argaud, F., Gernjak, W. and Keller, J. (2012), "Ozonation and biological activated carbon filtration of wastewater treatment plant effluents", *Water Research,* vol. 46, no. 3, pp. 863-872.

Ripperger, S., Walter, G. and Alt, C. (2012), *Filtration 1: Fundamentals,* Wiley VCH, Weinheim, Germany.

Rodgers, M., Healy, M. and Mulqueen, J. (2005), "Organic carbon removal and nitrification of high strength wastewaters using stratified sand filters", *Water Research,* vol. 39, no. 14, pp. 3279-3286.

Rose, C., Parker, A., Jefferson, B. and Cartmell, E. (2015), "The Characterisation of faeces and urine; a review of the literature to inform advanced treatment technology", *Critical Reviews in Environmental Science and Technology,* vol. 45, no. 17, pp. 1827-1879.

Rowe, R. K., Armstrong, M. D. and Cullimore, D. R. (2000), "Particle size and clogging of granular media permeated with leachate", *Journal of Geotechnical and Geoenvironmental Engineering,* vol. 126, no. 9, pp. 775-786.

Sarkhot, D., Ghezzehei, T. and Berhe, A. (2013), "Effectiveness of biochar for sorption of ammonium and phosphate from dairy effluent", *Journal of environmental quality,* vol. 42, no. 5, pp. 1545-1554.

Seginer, I., Ioslovich, I. and Bux, M. (2007), "Optimal control of solar sludge dryers", *Drying Technology,* vol. 25, no. 2, pp. 401-415.

Sparks, T. (2011), *Solid-Liquid Filtration: A user's guide to minimizing cost & environmental impact, maximizing quality & productivity,* Elsevier.

Speece, R. E. (2008), *Anaerobic biotechnology and odor/corrosion control for municipalities and industries,* 1st ed, Archae Press, Tennessee, USA.

Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) (2014), *Faecal Sludge Management: Systems Approach for Implementation and Operation,* 1st ed, IWA Publishing, London, UK.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Thornton, A., Pearce, P. and Parsons, S. (2007a), "Ammonium removal from solution using ion exchange on to MesoLite, an equilibrium study", *Journal of Hazardous Materials,* vol. 147, no. 3, pp. 883-889.

Thornton, A., Pearce, P. and Parsons, S. A. (2007b), "Ammonium removal from digested sludge liquors using ion exchange", *Water Research,* vol. 41, no. 2, pp. 433-439.

U.S. EPA (1999), *Wastewater Technology Fact Sheet: Intermittent Sand Filters,* , United States Environmental Protection Agency, Washington, D.C.U.S.A.

Ulyett, J., Sakrabani, R., Kibblewhite, M. and Hann, M. (2014), "Impact of biochar addition on water retention, nitrification and carbon dioxide evolution from two sandy loam soils", *European Journal of Soil Science,* vol. 65, no. 1, pp. 96-104.

van Haandel, A. and Van Der Lubbe, J. (2007), *Handbook Biological Waste Water Treatment-Design and Optimisation of Activated Sludge Systems,* 2nd ed, Quist Publishing, Leidschendam, Netherlands.

Vincent, J., Molle, P., Wisniewski, C. and Liénard, A. (2011), "Sludge drying reed beds for septage treatment: Towards design and operation recommendations", *Bioresource Technology,* vol. 102, no. 17, pp. 8327-8330.

Wang, K., Li, Y., Shammas, N. k. and Sakellaropoulos, G. P. (2010), "Drying Beds", in Wang, k. (ed.) *Handbook of Environmental Engineering,* 2nd ed, The Humana Press Inc, Totowa, NJ, USA, pp. 403-431.

Wang, S. and Wu, H. (2006), "Environmental-benign utilisation of fly ash as low-cost adsorbents", *Journal of Hazardous Materials,* vol. 136, no. 3, pp. 482-501.

Wang, Y., Lin, F. and Pang, W. (2008), "Ion exchange of ammonium in natural and synthesized zeolites", *Journal of Hazardous Materials,* vol. 160, no. 2, pp. 371-375.

Watson, I., van Straaten, P., Katz, T. and Botha, L. (2014), "Mining and Concentration: What Mining to What Costs and Benefits?", in *Sustainable Phosphorus Management,* Springer, New York. USA.

Wett, B., Demattio, M. and Becker, W. (2005), "Parameter investigation for decentralised dewatering and solar thermic drying of sludge", *Water Science and Technology,* vol. 51, no. 10, pp. 65-73.

Xu, G., Fan, X. and Miller, A. J. (2012), "Plant nitrogen assimilation and use efficiency", *Annual Review of Plant Biology,* vol. 63, pp. 153-182.

CHAPTER 8

THESIS DISCUSSION

8 THESIS DISCUSSION

Household sanitation services in low income countries are predominantly provided through off-grid on-site sanitation (OSS) facilities, with more than 2 billion urban dwellers using facilities such as pit latrines and septic tanks for excreta and wastewater management (Kone, 2010). These facilities differ substantially to that of water-borne piped sewage networks and subsequent waste water treatment works (WwTW) that prevail in high income countries. The sewage of water-borne piped networks is well characterised (Tchobanoglous et al., 2003) but there is a paucity of information regarding the composition of the residual waste of OSS facilities (faecal sludge), which consequently hinders the advancement and development of downstream treatment and resource recovery processes for FS (Niwagaba et al., 2014). Faecal sludge containment, treatment and resource recovery requires simple treatment flow sheets in order to meet the challenging socioeconomic conditions that prevail in low income countries. This has led to a surge in research aimed at the advancement of OSS facilities, treatment mechanisms and resource recovery processes for FS. High rate AD has been identified as a treatment mechanism that can provide safe FS stabilisation (Collins et al., 2013) and resource recovery will take place through methane (CH_4) capture. However, digestate, rich in nutrients, is often underutilised with impracticable nutrient recovery products created and this is where previous studies have been insufficient in providing a solution that addresses the need for simple treatment processes combined with effective nutrient recovery.

This research commenced with a review reporting current knowledge of the physical and chemical composition of faeces and urine. It was hypothesised that variation in production rates, physical and chemical composition could be accounted for by human factors such as diet and also that composition will directly influence the selection, design and development of new OSS technologies. Following this, waste characterisation was undertaken in order to establish the physical, chemical and biological characteristics of a range of different types of faecal sludge from OSS facilities (pit latrines, un-sewered public toilets and portable toilets). It was hypothesised that any variation that occurred across different OSS facilities could be attributed to differences in facility types, including retention time and storage

conditions within the OSS facility. The FS characterisation data collected also enabled the establishment of a baseline of FS composition and AD as a treatment process was evaluated.

In order to assess the digestibility of FS, operation of laboratory scale biochemical methane potential (BMP) assays enabled the quantification of CH_4 production for each FS substrate. The residence time in the OSS facility was hypothesised to impact BMP values of FS, with shorter residence times (<4 days in portable toilets) resulting in greater BMP values than OSS facilities with greater residence times (1-10 years in pit latrines). To elucidate the lower (<95% of average values) CH_4 production observed in 30% of portable toilets tested, the toxicity of the chemical toilet additives, which was hypothesised to cause CH_4 inhibition, was calculated through an assessment of the toilet chemical's active ingredients (glutaraldehyde and bronopol) half maximal inhibitory concentration (IC_{50}) values.

Nutrient analysis of FS was carried out to enable nutrient recovery pathways to be evaluated and allow the informed selection of the primary target for nutrient recovery, NH_4-N. It was hypothesised that through the identification of current secondary treatment processes and feasible nutrient recovery technologies that are suitable for the challenging socio-economic conditions of low income countries, the identification of a potential nutrient recovery system could be established. This led to the identification of sludge drying beds and adsorption/ion exchange processes for the recovery of NH_4-N, as well as the need to develop simple treatment flow sheets. These factors culminated in the research hypothesis that sludge drying beds could be modified by having suitable media incorporated into the drying bed configuration in order to achieve simultaneous dewatering and nutrient recovery.

Each individual component of this concept was tested under controlled conditions with three different media (sand, biochar and clinoptilolite) using a synthetic solution replicating digestate, as well as at greater scale using digested sewage sludge. These tests allowed the suitability of the media to recover NH_4-N as part of a sludge drying bed configuration to be assessed. It was hypothesised that high concentrations of cationic ions (>50 mg L^{-1} Ca, Mg, Na, K), organic matter (8000 mg COD L^{-1}) as well as the high solids nature of digestate percolate (20000-500 mg TS

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L^{-1}) would hinder the NH$_4$-N uptake capacity of the media. In addition the effect of empty bed contact time (EBCT), influent NH$_4$-N concentration and bed depth were all investigated to enable an effective design to be constructed. The drying bed configuration utilised a sand barrier above the media bed; it was hypothesised that this sand layer provided the majority of filtration and solids removal in a conventional sludge drying bed and sludge cake filtration was not the predominant mechanism. This information was utilised and incorporated into the design of a sludge drying bed configuration with a sacrificial sand barrier to enable simultaneous dewatering and nutrient recovery.

8.1 Potential for treatment of faecal sludge through anaerobic digestion

Faeces and urine composition was assessed in relation to a broad range of treatment processes (physical separators, chemical, biological, and thermal processes), with biological processes, notably AD, highlighted as the most numerous treatment mechanism under development (Chapter 2). However, the transformation of faeces and urine to FS through storage in OSS facilities was a key area of uncertainty to the application of biological processes such as AD. Previously reported studies on the characterisation of faecal sludge are focussed on a single FS type in a defined location and were therefore insufficient in order to assess the treatability of this waste stream by AD. A lack of comprehensive FS characterisation data are evident, largely due to the challenging environments in which data are collected and a lack of infrastructure capacity to carry out analysis in these localities.

It was hypothesised that FS characteristics were influenced by OSS facility type, retention time within the system and user behaviour. A large variation in the physical composition across OSS types was observed (4.56-19.24% TS: Chapter 3) demonstrating the site specific nature of FS, and was accounted for by the function of water inputs (dilution through flush water, urine, wash-water and chemical toilet additives) and outputs (leaching of water from storage facilities). Previous studies regarding the characterisation of FS also reported a wide range in TS and VS concentrations (Foxon et al., 2009; Salisbury et al., 2009; Coetzee et al., 2011) further emphasising the site specific type and nature of OSS facilities.

There was a wide range in COD concentrations (3.2-25.5 g COD_{sol} L^{-1}) across OSS types in this research, with the difference in readily digestible organics primarily influenced by the retention time of the facility type with BMP assays illustrating the most aged FS (pit latrines) had the lowest BMP values (50.6 mL CH_4/g VS_{added}) in comparison to that of the regularly emptied portable toilets (281 mL CH_4/g VS_{added}). However, no significant difference in BMP values were found between the top (0.15 m) and bottom sections (1 m) of pit latrines (Chapter 4) indicating that there was likely to be rapid aerobic degradation of the readily degradable fraction of the FS on the surface of the pit latrine followed by prolonged, slow anaerobic degradation in the succeeding layers. This confers with findings in pit latrines in South Africa by Buckley et al. (2008) who concluded that aerobic degradation through naturally occurring microorganisms occurred at the pit surface and is the predominant mechanism for organic removal and subsequent degradation once anaerobic conditions prevail is minor.

The high VFA concentrations seen in all FS types (9023-996 mg VFA L^{-1}) are produced by acidogenic and acetogenic bacteria, which reflects a kinetic uncoupling between acid producers and consumers (Ahring, 1995). Faecal sludge from OSS facilities with longer retention times, such as pit latrines and unsewered public toilets, have high acetic acid concentrations (55% of total VFA) indicating the hydrolysis stage, which is frequently identified as the rate-limiting step in AD (Li. and Noike, 1992; Wang et al., 1999; Tiehm et al., 2001), is complete. This means that the proteins, carbohydrates and fats have been broken down to amino acids, sugars and fatty acids by bacteria (Nielsen et al., 2007). Consequently, there is not the need for pre-treatment systems to be used in order to achieve higher rate digestion of this FS type. Therefore, AD as a treatment mechanism can be successful in the further stabilisation of FS from pit latrines despite relatively low BMP values (50.6 mL/g VS_{added}). However, high CH_4 production comparable to primary sewage sludge from conventional WwTW (358.4 mL CH_4/g VS_{added}) will only be generated by FS from OSS facilities that have a shorter retention time (<4 days) (such as portable toilets: 281 mL CH_4/g VS_{added}), where VFA build up is greatest (9023 mg VFA L^{-1}) and onward conversion to methane has not yet taken place (Chapter 3).

Urine has high urea concentrations (9300-23300 mg CH_4N_2O L^{-1}: Chapter 2), that increase substantially through storage due to urease decomposing urea into NH_3 and bicarbonate (Udert et al., 2003). This could cause ammonia (NH_3) toxicity in mixed waste streams. However, in a faeces only waste stream (such as portable toilets) causes of toxicity were observed because of secondary influences, such as chemical toilet additives (IC 50 values of $\geq$100 mg L^{-1} glutaraldheyde and $\geq$50 mg L^{-1} bronopol), rather than the human faeces components (Chapter 3).

8.2 Anaerobic digestion and nutrient recovery

Anaerobic digestion of FS will not reduce the mass of nutrients such as N and P, it only in-part mineralises organic N and P to inorganic forms (NH_4^+ and PO_4^-) (Möller and Müller, 2012). Consequently, the nutrient composition of FS prior to AD can reliably indicate nutrient flows in AD effluents (Chapter 5). The concentration of P in residual solids (12-26 g P kg^{-1}) were found to be marginally lower in pit latrines and unsewered public toilets in comparison to primary sludge from piped waterborne sewage networks (19-29 g P kg^{-1} (Mantovi et al., 2005; He et al., 2010)), this could be accounted for by minimal detergents/cleaning products entering OSS facilities: which often accounts for a high proportion of P received at WwTW (Yeoman et al., 1988), with toilet wastewater contributing only 59% of P received in the US (U.S. EPA, 2002). In contrast, high P concentrations were found in portable toilet waste (75 g P kg^{-1}), primarily as a result of the chemical toilet additive and not the human waste components (1.7-9.9 and 3.5-25 g P kg^{-1} in faeces and urine fractions respectively: Chapter 2). Concentrations of NH_4-N were high throughout all types of FS (520-1853 mg NH_4-N L^{-1}), in agreement to other studies investigating different FS types such as nightsoil (2000-5000 mg NH_4-N L^{-1}(Pradt, 1971; Heinss et al., 1998)) and septage (150-600 mg NH_4-N L^{-1} (Koottatep et al., 2004)). In addition significant organic nitrogen (2080-4561 mg N L^{-1}) present in FS will partially mineralise during AD causing a further increase in NH_4-N concentrations (Chen et al., 2008). Total N concentrations will vary according to OSS facility but N production in faeces and urine is high (1.8 and 11.0 g/cap/day N in faeces and urine respectively): for this reason, AD effluents from both OSS facilities and WwTW will have high NH_4-N concentrations with Parsons et al. (2001) reporting concentrations in the region of

426-957 mg NH_4-N L^{-1} across six different UK WwTW. The high concentration of NH_4-N in AD effluents therefore presents an opportunity for nutrient recovery.

8.3 Nutrient recovery sludge drying beds

The adaptation of sludge drying beds is a feasible method for the recovery of key macro-nutrients (N, P, K) required in agriculture (Chapter 6 and 7). However, the design (Figure 8.1) and treatment flow sheet (Figure 8.2) has to balance media capacity, particle size as well as hydraulic and solids loading rates which all in turn influence the performance, configuration and operation, as described below.

The use of batch adsorption and kinetic tests provided a useful foundation in regards to the selection of suitable media, as well as providing an indication of the extent to which varying factors impact performance (for instance organic matter and competing cationic ions) and these results have broadened the understanding of the potential for clinoptilolite and biochar to be used in a drying bed application. In this regard, clinoptilolite had the greatest potential for the drying bed application (NH_4-N capacities of 4.8 and 9.3 mg/g in biochar and clinpotiolite respectively) which was within the literature range for NH_4-N capture (3-5.3 and 5-15 mg/g in biochar and clinoptilolite respectively when applied to wastewater (Green et al., 1996; Nguyen and Tanner, 1998; Sarkhot et al., 2013)). However, it is only through dynamic column studies that the true operational capacity of the media was determined. For instance, the batch adsorption capacity of clinoptilolite at 200 mg NH_4-N L^{-1} (9.33 mg/g) was lower in comparison to the range of capacities at the same NH_4-N concentration utilising dynamic column experiments (5-60 mg/g dependent on operational conditions). This is because in dynamic experiments the influent NH_4-N concentration is constantly being replenished resulting in a higher solute gradient providing the necessary driving force to enable NH_4^+ ions to replace cations. In contrast, in batch equilibrium trials the NH_4-N solution reduces as uptake of the media increases (Du et al., 2005; Wang et al., 2006). Consequently, dynamic experiments at a range of EBCT, NH_4-N concentrations, and bed depths are essential in order to assess, design and scale-up a fixed media bed to be incorporated into a sludge drying bed configuration (Cooney et al., 1999).
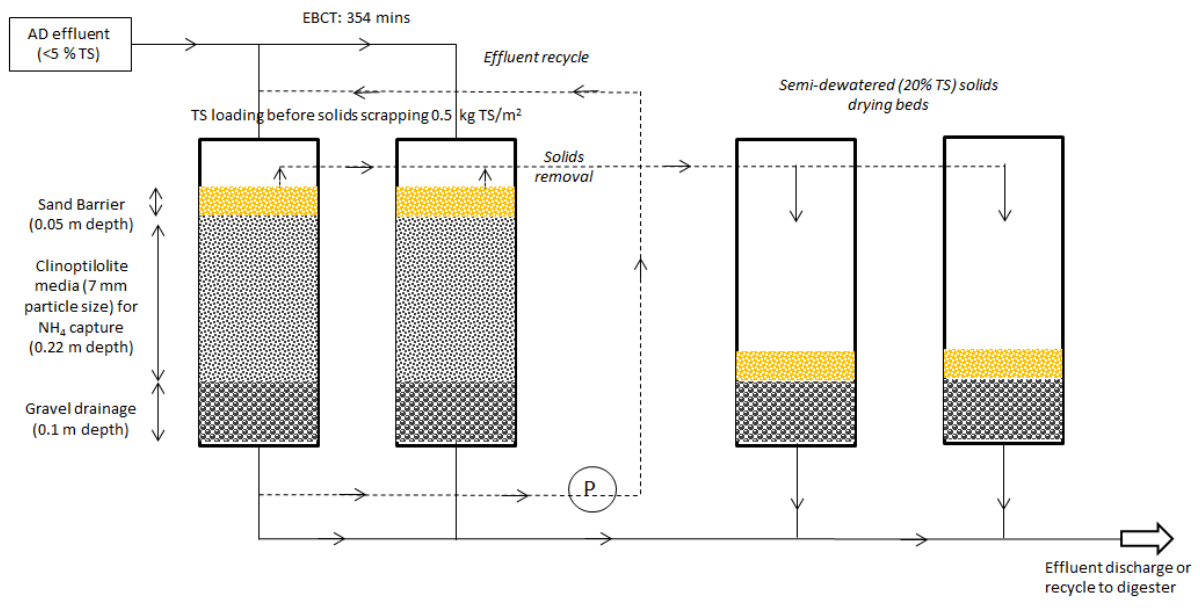
Figure 8.1. Nutrient recovery drying bed configuration consisting of 2 clinoptilolite beds operating in series with an EBCT of 354 minutes, with semi-dewatered solids scraping (application rate of 0.5 kg/m2) and application to secondary sludge cake drying beds.

The design and rate at which the nutrient recovery process is operated at will be determined by the desired footprint and service time of the individual site. For instance, the recommended bed depth of 0.22 m (Figure 8.1) is dependent on the desired service time of the media (79 days: Figure 8.2) rather than the efficiency of the media at different bed depths (4.05-11.89 kg/m^3 treated: Chapter 6). The desired service time is important as this will determine how quickly the media will become exhausted and require changing; consequently a more regular service time will result in increased operational costs, reducing efficiency. Similarly, operation at a low hydraulic loading rate (EBCT: 354 minutes) may be the most efficient in regards to NH$_4$-N capacity (60 g NH$_4$-N /kg clinoptilolite). However, the drying bed will have a significantly larger footprint (17.7 times greater) than a system with reduced EBCT (20 mins) and resultant media capacity (5 g NH$_4$-N /kg clinoptilolite) as reflected by the vastly increased hydraulic loading rates at low EBCT (3 and 0.17 bed volumes/hour respectively). Nevertheless, EBCT will be determined to the greatest extent by the rate of clogging by TS in the digestate, with TS loading rates of 0.04-

0.5 kg TS/m^2: (Chapter 7) indicating the critical point in which percolation is reduced and cake filtration predominates.
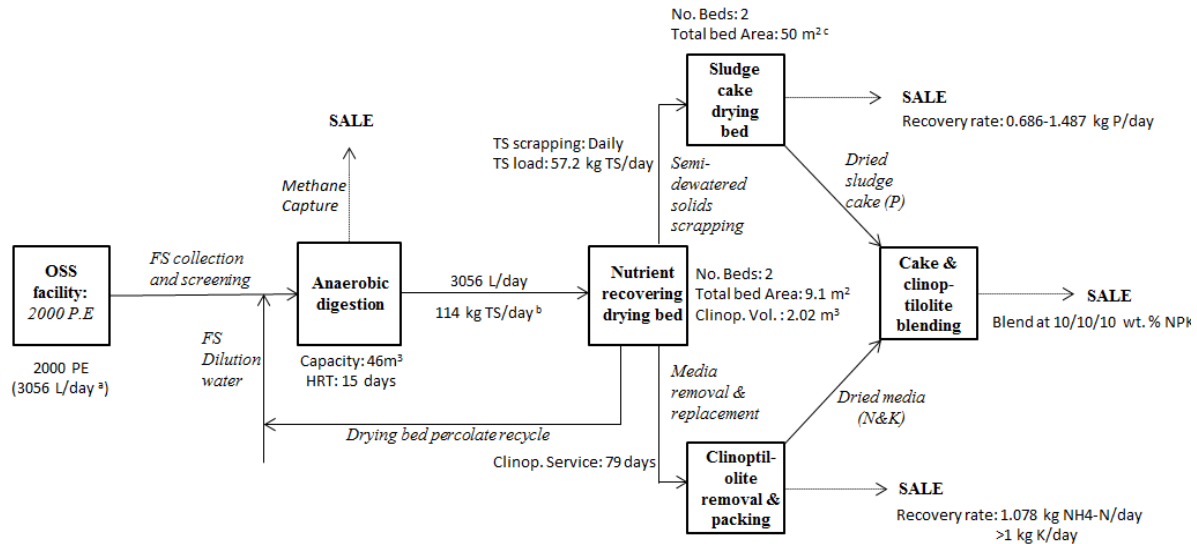
^{a} Production rate of faeces and urine from median literature values (Chapter 2)
^{b} TS loading rates utilised from AD projections (Chapter 5)
^{c} Sludge cake drying bed to remove 10% wt. moisture and highly dependent on climate as solar drying predominates (Chapter 6)

Figure 8.2 Proposed treatment flow sheet for faecal sludge management and nutrient recovery with a population equivalent (PE) of 2000 utilised for faeces and urine production rates.

Consequently, the hydraulic loading of the system should be based upon TS loading values of 0.04-0.5 kg TS/m^2 (dependent on sand particle size and the TS of influent digestate: Chapter 7, Figure 7.5), which is within the range of TS loading rates for conventional sludge drying beds (0.18 kg TS/m^2/application (Tchobanoglous et al., 2003)). However, in order to achieve intensification of the system and reduce the considerable footprint of sludge drying beds (120-321 kg TS/m^2/year (Tchobanoglous et al., 2003; Cofie et al., 2006)), scraping of TS from the sacrificial sand barrier used to protect the media bed is necessary (Figure 8.1). More than 50% of percolation occurs within the first 28 hours (Chapter 7: Figure 7.7), which corresponds to findings that 75% of sludge water percolates within 1-3 days in conventional sludge drying beds (van Haandel and Van Der Lubbe, 2007).

Therefore, if the semi-dewatered solids are scrapped from the surface of the nutrient recovery drying beds and reapplied to an additional sludge cake drying bed (Figure 8.1 and Figure 8.2) TS application could be increased by 15 fold (assuming a 30 day application period in conventional sludge drying beds). This intensification would in turn allow sufficient contact time between the digestate percolate and the fixed clinoptilolite bed beneath and would allow NH_4-N recovery to progress unhindered and achieve maximum recovery rates stated (60 g NH_4-N/kg clinoptilolite: Chapter 6).

The intensification of sludge drying beds is also beneficial for the nutrient recovery component; as over long time periods there is the potential for biofilm growth on and within media particles in which nitrifying bacteria may remove NH_4-N (Nguyen and Tanner, 1998). This would be beneficial in terms of NH_4-N removal; however, the process would not be adding to the capacity of the clinoptilolite and resultantly would not increase its fertiliser value. Therefore, conditions relating to the speed of growth of a biofilm may need to be taken into account: for instance substrate, temperature, pH, NH_4-N concentration and dissolved oxygen levels (Mara and Horan, 2003). However, no increase in nitrate concentrations of column effluents was observed during long-term (25 days) studies utilising anaerobic digestate (Chapter 6) indicating conditions were not optimal for the growth of nitrifying bacteria on the media with the sludge drying bed.

Media selection is also an essential factor in the design of nutrient recovery drying beds. Clinoptilolite with a larger particle size (7-9 mm) could be loaded with 48% more TS than that of a smaller particle size (0.7-1.6 mm) before the critical clogging point was reached. This factor is important as the sacrificial sand barrier will only remove on average between 44-48% TS (Chapter 7: Figure 7.5), therefore the media bed beneath may become clogged before the clinoptilolite has reached saturation. Therefore, despite larger media sizes being less efficient in NH_4-N capture than smaller particle sizes (20.27 and 14.40 mg NH_4-N/g media), the positive impact of allowing increased TS loading (0.43 and 0.22 kg TS/m^2 respectively) and reducing pressure drop build-up in the bed will outweigh any reduction in performance. Consequently, a particle size ≥5 mm is consequently recommended (Figure 8.1).

A distinct operational benefit of incorporating an ion exchange system over a conventional biological nitrogen removal system is the ability of clinoptilolite to effectively deal with a wide range of NH_4-N concentrations (20-200 mg NH_4-N L^{-1}) (Chapter 6: Figure 6.4) as well as the peak NH_4-N loads that would be expected in the percolate of batch fed drying beds (Jorgensen and Weatherley, 2003; Sica et al., 2014). Consequently, the effluent of a nutrient recovery drying bed system could consistently meet NH_4-N discharge consents if required or be recirculated for dilution of FS prior to AD (Figure 8.2). The gradual saturation and movement of the mass transfer zone through the media bed depth (Chapter 6: Figure 6.1), means the effluent of beds will therefore gradually increase in NH_4-N concentration causing rising concentrations in the discharge/recycle stream and inefficiency in the NH_4-N recovery process. For this reason, the operation of the dying bed configuration is optimal in series of at least two beds (Figure 8.1).

The full saturation of the clinoptilolite in the configuration outlined (Figure 8.2) would provide a fertiliser product with a grade of 5.9 wt. % NH_4-N. In addition, due to high K concentrations (277 mg K L-1: Chapter 6) present in the sludge liquor fraction the media will have an equally high proportion of K^+ due to the favourable selectivity sequence of clinoptilolite ($K^+ > NH_4^+ > Na^+ > Ca^{2+}$) (Papadopoulos et al., 1996; Cooney et al., 1999; Hankins et al., 2005). These values are lower than common artificial N fertiliser grades: such as ammonium nitrate (34% N), ammonium sulphate (21% N) and urea (45% N) (DAFVM, 2014) but are not far removed in N and K from commonly used 10/10/10 wt. % N-P-K fertilisers (Lima et al., 2015). Saturated media therefore provides consistent values for NH_4-N and K^+, however to provide a balanced fertiliser product, the dewatered sludge cake solids from the secondary sludge drying bed (4.2 wt. % P) (Figure 8.1) can be blended with the clinoptilolite as part of the treatment flow sheet (Figure 8.2): this will provide a complete N-P-K fertiliser product and can be blended to the desired proportions according to market demand in the locality. This has the additional advantage of providing organic matter to soils (32 wt. % C) that has additional benefits to soil structure and microbial communities (Lu et al., 2012). Consequently the use of a secondary sludge drying bed for the solar drying of semi-dewatered cake that accumulates on the sand barrier surface (Figure 8.1) could have multiple benefits: the intensification of drying

beds enabling nutrient recovery at a faster rate and the creation of a P rich bi-product that can be blended with saturated media to create a complete N-P-K fertiliser and an enriched biosolids product (Figure 8.2).

8.4 Implications and practicalities for the sector

On-site sanitation facilities (such as pit latrines) in urban areas have often been considered as temporary solutions until sewer based systems could be introduced (Seck et al., 2014), however, an estimated 1.7 billion people worldwide use some form of pit latrine (Graham and Polizzotto, 2013) and the FS produced consequently requires urgent removal, treatment and re-use according to these defined storage conditions (Chapter 3). Consequently, adapting a treatment and nutrient recovery process to adequately address this waste stream is a research priority. In addition, new innovative forms of OSS facilities (such as portable toilets) are currently being introduced (Clean Team, 2012) and these facilities need to use FS characterisation information such as that presented here. For instance, portable toilet waste will require extended residence time and a resultant greater digester capacity in order to break down the chemical toilet additive components and the chemical additives introduce heavy metals (27251 mg Cu kg^{-1}: Chapter 3) to the effluent which will exceed regulatory limits for sewage sludge application (European Union, 1986). In addition it is also probable that chemical toilet additives (at current concentrations) will hinder potential nutrient recovery systems such as ion exchange (Chapter 6 and 7) due to vast concentrations of competing cationic ions, dye components and heavy metals (e.g. 27251 mg Cu kg^{-1}) present.

Overall, this highlights the need for a holistic approach that encompasses the complete system in order to engineer OSS facilities to collect FS by a means that can enable resource recovery opportunities, e.g. by avoiding chemical usage through systems such as the 'rotating odour barrier' (Parker, 2014) in household toilet design. Regular collections would be required to ensure the maximum CH$_4$ yield (Chapter 4), however, a short retention time is not required for the recovery of key macro nutrients (Chapter 5). Consequently, if the majority of cost recovery is expected through nutrient recovery the collection period can be extended, such as in pit latrines (1-10 year collection frequency), and the marginal gain of regular

collection from this OSS facility type was highlighted as being minimal (Top: 136 mL CH_4/g VS_{added}, bottom: 130 mL CH_4/g VS_{added}).

Multiple process flows in WwTW presents challenges to operation and maintenance (Bassan and Robbins, 2014). Consequently, if a nutrient recovery system could be combined with sludge dewatering in one simple treatment process this would present great benefit to the sector (Figure 8.2). The ability of clinoptilolite to perform as both a solids filter (44-48 % TS removal: Chapter 7) as well as maintain high NH_4-N operational capacity (60 g NH_4-N /kg clinoptilolite), demonstrates a highly suitable media for this application. However, this reduction in cost of a pre-treatment step may be negated by the increased complication in design, which would require a site specific design and installation, which could prove challenging in a low income environment where skilled process engineers are both scarce and expensive.

Experimental work in this study (Chapter 7) was carried out utilising digestate from a mesophilic AD at a UK WwTW that utilised a Thermal Hydrolysis Process (THP) (Blytt, 2009) pre-treatment step. This was primarily due to logistical ease, however, poses the question of how transferable the results are to digestate expected from AD in low income countries. The expected NH_4-N concentrations (Chapter 5) and the effect of any variation on the media uptake capacity was explored (Chapter 6), however, the greatest factor influencing the drying bed design is likely to be the clogging and dewatering characteristics of the digestate, primarily influenced by particle size distribution (Karr and Keinath, 1978; Neyens and Baeyens, 2003). Particle size distribution was measured in FS of pit latrines and public toilets (Chapter 3: Appendix 2) and had a range in values similar to primary sewage sludge (>7mm 5-20%, 1-7mm 9-33%, <1mm 50-88%), the predominant feedstock of AD at WwTW, which would indicate a comparable FS type. However, the thermal hydrolysis pre-treatment step in use changes the rheology of the digested sewage sludge due to high temperature and pressure: this results in a more highly compressible sludge (improving dewaterability), which consequently differs to conventional digestion of sewage sludge (Higgins et al., 2011) and is reflected by the digestate particle size distribution d (0.9) value of 57.367 ± 2.014 µm). Therefore, percolation rates of this study (Chapter 6: Figure 7.7) may be amplified, although conversely the smaller particle sizes of the digestate used in this study may cause

increased rates of clogging within the media bed due to greater penetration into the sacrificial sand layer and subsequent build up in the media layer beneath. The results of this study regarding clogging rates should therefore present guideline figures and it is clear that site specific testing with the actual digestate and sand particle size would be beneficial to accurately gauge percolation rates.

Sanitation provision and wastewater treatment in low income countries predominantly revolves around the ease of implementation and cost, therefore appropriate media selection is a pivotal factor (Chapter 5). Caution should remain in the use of clinoptilolite due to its heterogeneous nature with varying degrees of impurities within the media (e.g. Clinoptilolite used in this study had 92% purity) presenting uncertainties in process design such as media capacity and kinetic uptake rates. Nevertheless, clinoptilolite is an abundant and low cost media (Shokrian et al., 2015), available extensively in countries such as China, Turkey and India at potentially viable prices (approximately 50-100 $USD/tonne). Although the direct use of saturated clinoptilolite requires large volumes of media, as regeneration is not required, the high regeneration costs (including infrastructure, chemicals, chemical storage and brine disposal) are redundant which further increases the viability of using ion exchange in a drying bed configuration.

The feasibility of nutrient recovery drying beds to increase the value of low cost media and create a valuable product that has high defined NH_4-N and K^+ concentrations, with the media combined with dewatered solids (4.2 wt. % P) to form a balanced fertiliser product, has been illustrated (Figure 8.1 and Figure 8.2). However, its implementation success will be dependent on a full economic analysis of the production costs and market demand in the locality. Nevertheless, additional benefits of incorporating a nutrient recovery stage to the treatment process flow sheet (Figure 8.2) beyond cost recovery; include the potential for the media beds to adsorb any non-biodegradable organics that could not be broken down in the AD process, either reducing concentrations in effluent recycle streams or preventing discharge to the environment.

8.5 Further Work

The results of this study have illustrated that clinoptilolite can be utilised as part of a sludge drying bed design to enable the recovery of NH_4-N from anaerobic digestate. However, the optimisation of this process along with the subsequent use of the fertiliser product created requires further research, with particular emphasis on the following issues:

- Operation at long EBCT has proven the most efficient operational mode in regards to media capacity, however, the presence of nitrifying bacteria on the media's long term operational capacity requires further investigation to determine whether nitrification may result in the loss of NH_4-N, negating the primary objective of nutrient recovery.

- Additional uncertainties remain regarding the desorption properties of clinoptilolite as when media has reached exhaustion there is the potential for NH_4-N to be removed by influent percolate, this could lead to effluent NH_4-N concentrations being greater than influent NH_4-N concentrations in the drying bed configuration. This factor would therefore be important to consider and will also provide essential information regarding the application of saturated clinoptilolite to agricultural soils.

- The fertiliser properties of the saturated clinoptilolite product created is the most important issue requiring further research. A full investigation into the exact nutrient composition, along with pot and field trials will be necessary in order to establish whether the saturated clinoptilolite brings value as a fertiliser product. In addition, the effect of the addition of saturated clinoptilolite on soil structure and chemistry are vital issues. Other factors to consider will be desorption of NH_4^+ and K^+ from clinoptilolite into the soil over short and prolonged timescales. In addition the relative benefits of co-applying dewatered solids (as a source of P), that accumulate on the top of the sand barrier with the saturated clinoptilolite will inform agricultural use as well as inform treatment flow sheets. The fertiliser benefits to agricultural production will in turn help to establish a market value for the product.

- A full economic assessment should be undertaken in order to establish the full cost of construction, maintenance and operation of the integrated nutrient recovery sludge drying beds. The purchase cost of the original clinoptilolite as well as the final market value of the saturated clinoptilolite and blended biosolids product will be dependent on geographical location and local market demand but is an essential element to the success of an integrated nutrient recovery sludge drying bed system.

8.6 References

Ahring, B. K. (1995), "Methanogenesis in thermophilic biogas reactors", *Antonie van Leeuwenhoek,* vol. 67, no. 1, pp. 91-102.

Bassan, M. and Robbins, D. M. (2014), "Operation, Maintenance and Monitoring of Faecal Sludge Treatment Plant", in Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) *Faecal Sudge Management,* 1st ed, IWA Publishing, London, UK, pp. 231-242.

Blytt, L. D. (2009), "The Cambi Process and Agriculture, a Sustainable Solution?", 9-11 November 2009, Leeds, UK, AquaEnviro, pp. 1.

Buckley, C., Foxon, K. M., Broukaert, C. J., Rodda, N., Nwaneri, C., Balboni, E., Couderc, A. and Magagna, D. (2008), *Scientific support for the design and operation of Ventilated Improved Pit latrines (VIPs) and the efficacy of pit latrine additives,* WRC project no K5/1630, Water Research Commission, Durban, South Africa.

Chen, Y., Cheng, J. J. and Creamer, K. S. (2008), "Inhibition of anaerobic digestion process: a review", *Bioresource Technology,* vol. 99, no. 10, pp. 4044-4064.

Clean Team (2012)*, Clean Team Toilets*, available at: http://www.cleanteamtoilets.com/ (accessed August 2014).

Coetzee, M., Van der Merwe, M. and Badenhorst, J. (2011), "Effect of nitrogen loading rates on nitrogen removal by using a biological filter proposed for ventilated improved pit latrines", *International Journal of Environmental Science and Technology,* vol. 8, no. 2, pp. 363-372.

Cofie, O. O., Agbottah, S., Strauss, M., Esseku, H., Montangero, A., Awuah, E. and Kone, D. (2006), "Solid–liquid separation of faecal sludge using drying beds in Ghana: Implications for nutrient recycling in urban agriculture", *Water Research,* vol. 40, no. 1, pp. 75-82.

Collins, G., Kennedy-Walker, R., Connelly, S., Shin, S. G., Nguyen, V., Rose, C., R. Dillon, R., Morciano, C., Parsons, S., A. Parker, A., Cartmell, E. :. A., J., Raffo, D., Mehta, L. and Biggs, C. (2013), "The Transforming Waste Consortium: A Socio-Technical Approach to Sanitation in Peri-Urban Environments in Developing Countries", van Loosedrecht, M. (ed.), in: *2nd Water Research Conference,* Vol. 1, 20 - 23 January 2013, Singapore, IWA, London, pp. 89.

Cooney, E. L., Booker, N. A., Shallcross, D. C. and Stevens, G. W. (1999), "Ammonia removal from wastewaters using natural Australian zeolite. I. Characterization of the zeolite", *Separation Science and Technology,* vol. 34, no. 12, pp. 2307-2327.

DAFVM (2014)*, Soil Fertility and Fertilizers*, available at: http://msucares.com/crops/soils/mgfertility.html (accessed May 2015).

Du, Q., Liu, S., Cao, Z. and Wang, Y. (2005), "Ammonia removal from aqueous solution using natural Chinese clinoptilolite", *Separation and Purification Technology,* vol. 44, no. 3, pp. 229-234.

European Union, (1986), *EC Directive on the Use of Sewage Sludge in Agriculture (1986/278),* EU Directive ed., 86/278/EEC, Brussels, Belgium.

Foxon, K., Mkhize, S., Reddy, M. and Buckley, C. (2009), "Laboratory protocols for testing the efficiency of commercial pit latrine additives", *Water SA,* vol. 35, no. 2, pp. 228-235.

Graham, J. P. and Polizzotto, M. L. (2013), "Pit Latrines and Their Impacts on Groundwater Quality: A Systematic Review", *Environmental Health Perspectives,* vol. 121, no. 5, pp. 521.

Green, M., Mels, A. and Lahav, O. (1996), "Biological-ion exchange process for ammonium removal from secondary effluent", *Water Science and Technology,* vol. 34, no. 1, pp. 449-458.

Hankins, N., Pliankarom, S. and Hilal, N. (2005), "An Equilibrium Ion-Exchange Study on the Removal of NH4 Ion from Aqueous Effluent Using Clinoptilolite", *Separation Science and Technology,* vol. 39, no. 15, pp. 3639-3663.

He, Z., Zhang, H., Toor, G. S., Dou, Z., Honeycutt, C. W., Haggard, B. E. and Reiter, M. S. (2010), "Phosphorus distribution in sequentially extracted fractions of biosolids, poultry litter, and granulated products", *Soil Science,* vol. 175, no. 4, pp. 154-161.

Heinss, U., Larmie, S. A. and Strauss, M. (1998), *Solids separation and pond systems for the treatment of faecal sludges in the tropics: lessons learnt and recommendations for preliminary design,* 05/98, EAWAG/SANDEC, Dubendorf, Switzerland.

Higgins, M., Murthy, S. N., Schafer, P., Cooper, A., Kasirga, E., Lee, K., Machisko, J., Fountain, P. and Kelleher, K. (2011), "Dewatering Characteristics of Cambi Thermal Hydrolysis Biosolids: Centrifuges vs. BFPs", *Proceedings of the Water Environment Federation,* vol. 2011, no. 17, pp. 678-684.

Jorgensen, T. C. and Weatherley, L. R. (2003), "Ammonia removal from wastewater by ion exchange in the presence of organic contaminants", *Water Research,* vol. 37, no. 8, pp. 1723-1728.

Karr, P. R. and Keinath, T. M. (1978), "Influence of particle size on sludge dewaterability", *Journal of the Water Pollution Control Federation,* vol. 50, no. 8, pp. 1911-1930.

Kone, D. (2010), "Making urban excreta and wastewater management contribute to cities' economic development: a paradigm shift", *Water Policy,* vol. 12, no. 4, pp. 602-610.

Koottatep, T., Surinkul, N., Polprasert, C., Kamal, A., Koné, D., Montangero, A., Heinss, U. and Strauss, M. (2004), "Treatment of septage in constructed

wetlands in tropical climate–Lessons learnt after seven years of operation", *Water Science and Technology,* vol. 51, no. 9, pp. 119-126.

Li., Y. Y. and Noike, T. (1992), "Upgrading of anaerobic digestion of waste activated sludge by thermal pretreatment", *Water Science and Technology,* vol. 26, no. 3-4, pp. 857-866.

Lima, A. F., Da Costa, M. C., Ferreira, D. C., Richter, E. M. and Munoz, R. A. A. (2015), "Fast ultrasound-assisted treatment of inorganic fertilizers for mercury determination by atomic absorption spectrometry and microwave-induced plasma spectrometry with the aid of the cold-vapor technique", *Microchemical Journal,* vol. 118, no. 0, pp. 40-44.

Lu, Q., He, Z. L. and Stoffella, P. J. (2012), "Land application of biosolids in the USA: A review", *Applied and Environmental Soil Science,* vol. 2012, no. Article ID 201462, pp. 1-11.

Mantovi, P., Baldoni, G. and Toderi, G. (2005), "Reuse of liquid, dewatered, and composted sewage sludge on agricultural land: effects of long-term application on soil and crop", *Water Research,* vol. 39, no. 2–3, pp. 289-296.

Mara, D. and Horan, N. J. (2003), *Handbook of water and wastewater microbiology,* Academic press.

Möller, K. and Müller, T. (2012), "Effects of anaerobic digestion on digestate nutrient availability and crop growth: A review", *Engineering in Life Sciences,* vol. 12, no. 3, pp. 242-257.

Neyens, E. and Baeyens, J. (2003), "A review of thermal sludge pre-treatment processes to improve dewaterability", *Journal of Hazardous Materials,* vol. 98, no. 1–3, pp. 51-67.

Nguyen, M. and Tanner, C. (1998), "Ammonium removal from wastewaters using natural New Zealand zeolites", *New Zealand Journal of Agricultural Research,* vol. 41, no. 3, pp. 427-446.

Nielsen, H. B., Uellendahl, H. and Ahring, B. K. (2007), "Regulation and optimization of the biogas process: Propionate as a key parameter", *Biomass and Bioenergy,* vol. 31, no. 11-12, pp. 820-830.

Niwagaba, C. B., Mbéguéré, M. and Strande, L. (2014), "Faecal Sludge Quantification, Characterisation and Treatment Objectives", in Strande, L., Ronteltap, M. and Brdjanovic, D. (eds.) *Faecal Sludge Management,* 1st ed, IWA Publishing, London, UK, pp. 19.

Papadopoulos, A., Kapetanios, E. and Loizidou, M. (1996), "Studies on the use of clinoptilolite for ammonia removal from leachates", *Journal of Environmental Science and Health Part A,* vol. 31, no. 1, pp. 211-220.

Parker, A. (2014), "Membrane technology plays key role in waterless hygienic toilet", *Membrane Technology,* vol. 2014, no. 12, pp. 8.

Parsons, S., Wall, F., Doyle, J., Oldring, K. and Churchley, J. (2001), "Assesing the potential for struvite recovery at sewage treatment works", *Environmental Technology,* vol. 22, no. 11, pp. 1279-1286.

Pradt, L. A. (1971), "Some recent developments in night soil treatment", *Water Research,* vol. 5, no. 8, pp. 507-521.

Salisbury, R. H., Lorentz, S., Buckley, C. A., Bhagwan, J. and Still, D. (2009), "Finding a sustainable solution to the overflowing pit problem: The role of SRF in the beneficiation of VIP sludge", *Water, sanitation and hygiene. Sustainable development and multisectoral approaches. Preceedings of the 34th WEDC International conference,* Vol. 1, 18th-22nd May 2009, Addis Ababa, Ethiopia, 34th WEDC International Conference, WEDC, Loughborough, UK, pp. 1.

Sarkhot, D., Ghezzehei, T. and Berhe, A. (2013), "Effectiveness of biochar for sorption of ammonium and phosphate from dairy effluent", *Journal of Environmental Quality,* vol. 42, no. 5, pp. 1545-1554.

Seck, A., Gold, M., Niang, S., Mbéguéré, M., Diop, C. and Strande, L. (2014), "Faecal sludge drying beds: increasing drying rates for fuel resource recovery in Sub-Saharan Africa", *Journal of Water, Sanitation and Hygiene for Development,* vol. 5, no. 1, pp. 72-80.

Shokrian, F., Solaimani, K., Nematzadeh, G. and Biparva, P. (2015), "Removal of Sodium Chloride from aqueous solutions by using clinoptilolite", *International Journal of Farming and Allied Sciences,* vol. 4-1, no. 50-54, pp. 2322-4134.

Sica, M., Duta, A., Teodosiu, C. and Draghici, C. (2014), "Thermodynamic and kinetic study on ammonium removal from a synthetic water solution using ion exchange resin", *Clean Technologies and Environmental Policy,* vol. 16, no. 2, pp. 351-359.

Tchobanoglous, G., Burton, F. and Stensel, H. (2003), *Wastewater Engineering Treatment and Reuse; Metcalf and Eddy,* 4th ed, McGraw-Hill Company, London, UK.

Tiehm, A., Nickel, K., Zellhorn, M. and Neis, U. (2001), "Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization", *Water Research,* vol. 35, no. 8, pp. 2003-2009.

U.S. EPA (2002), *Onsite Wastewater Treatment Systems Manual,* EPA/625/R-00/008, Office of Water Office of Research and Development U.S. Environmental Protection Agency, USA.

Udert, K. M., Larsen, T. A. and Gujer, W. (2003), "Estimating the precipitation potential in urine-collecting systems", *Water Research,* vol. 37, no. 11, pp. 2667-2677.

van Haandel, A. and Van Der Lubbe, J. (2007), *Handbook Biological Waste Water Treatment-Design and Optimisation of Activated Sludge Systems,* 2nd ed, Quist Publishing, Leidschendam, Netherlands.

Wang, Q., Kuninobu, M., Kakimoto, K., I.-Ogawa, H. and Kato, Y. (1999), "Upgrading of anaerobic digestion of waste activated sludge by ultrasonic pretreatment", *Bioresource Technology,* vol. 68, no. 3, pp. 309-313.

Wang, Y., Liu, S., Xu, Z., Han, T., Chuan, S. and Zhu, T. (2006), "Ammonia removal from leachate solution using natural Chinese clinoptilolite", Journal of Hazardous Materials, vol. 136, no. 3, pp. 735-740.

Yeoman, S., Stephenson, T., Lester, J. N. and Perry, R. (1988), "The removal of phosphorus during wastewater treatment: A review", Environmental Pollution, vol. 49, no. 3, pp. 183-233.

CHAPTER 9

CONCLUSIONS

9 CONCLUSIONS

This study has designed a nutrient recovery system and process for the capture of nutrients from anaerobic digestion effluents treating faecal sludge. These conclusions correspond to the objectives that were outlined in Chapter 1 of this thesis.

1. To review current knowledge of faeces and urine characteristics in order to determine how the physical and chemical composition will impact different treatment process types and nutrient recovery potential.

   - The rate of production (51-796 g/cap/day) as well as the physical (12-81 g TS/cap/day) and chemical composition of faeces is variable (7.0-38.3 g N kg^{-1}, 1.8-9.9 g P kg^{-1}, 1.8-4.9 g K kg^{-1}, 0.8-4.94 g Na kg^{-1}) and the greatest factor causing variation was indigestible dietary fibre intake of the target population. Similarly, urine production rates are wide ranging (0.6-2.6 L/cap/day), primarily due to differences in the water balance of the body. The substantial variation subsequently presents difficulties in assigning standard design values for on-site sanitation treatment systems and means collection/treatment systems must be robust and flexible to deal with this uncertainty.

   - Biological treatment processes are well suited for faeces treatment, although substantial urea concentrations in urine (9300-23300 mg CH_4N_2O L^{-1}) have the potential to cause NH_3 toxicity issues. This factor could promote source separation and would equally increase efficiency of dewatering and thermal treatment processes.

   - The largest proportion of N (90%), P (50-65%) and K (50-80%) was present in the urine fraction, however, the nutrient recovery potential of faeces should not be underestimated with production rates of 1.8 g/cap/day N, of which 50% is water soluble, and significant quantities of P (0.47 g/cap/day) are voided in the faeces fraction which could be recovered. In addition high concentrations of macro-nutrients (8140 mg N

L^{-1}, 4650 mg P L^{-1}, 1314 mg K L^{-1}) as well as micro-nutrients in the urine fraction that are readily available.

2. To establish the physical, chemical and biological characteristics of different types of faecal sludge from on-site sanitation facilities and evaluate how the results will impact anaerobic digestion as a treatment process.

- The physical composition of pit latrine FS (19.2% TS) is more suited to suspended growth reactors rather than up-flow or down-flow high rate processes whereas portable toilet (4.5% TS) and unsewered public toilet waste (6.1% TS) are more feasible for increased rate AD processes without the need for significant dilution. High municipal solid waste content (12% of total volume) in pit latrine FS could also prove problematic without substantial screening prior to AD.

- The mean C:N ratio was similar in pit latrines and unsewered public toilets (11.58 and 10.99 respectively), but was slightly reduced in portable toilets (10.43). The concentration of NH_4-N was greatest in pit latrine FS (1853 mg NH_4-N L^{-1}) but similarly high concentrations were observed in unsewered public toilets and portable toilet waste (846 and 949 mg NH_4-N L^{-1} respectively).

- Values of COD_{sol} varied vastly from 3.26 g COD_{sol} L^{-1} in unsewered public toilets to 25.5 g COD_{sol} L^{-1} in portable toilets. Gross energy values were greatest in FS that was stored in OSS with a shorter retention time (22.241 MJ/kg TS and 10.242 MJ/kg TS in portable toilets and pit latrines respectively).

- High acetic acid formation (55% of total VFA concentration in pit latrines) indicates the hydrolysis stage is complete, which is often the first and general rate limiting step in AD and could negate the need for separate hydrolysis/thermal hydrolysis steps before digestion.

3. To assess the digestibility of faecal sludge from a range of on-site sanitation facilities and establish the biochemical methane potential of each substrate.

- The highest BMP values of 276.0 ± 151.3 mL/g VS_{added} were recorded in portable toilet waste. In contrast relatively low BMP values were recorded in FS from pit latrines and public toilets (50.6 ± 19.4 mL/g VS_{added} and 36.6 ± 19.8 mL/g VS_{added} respectively). These values of OSS facilities with long retention times differ substantially to BMP values of primary sewage sludge (358.4 mL CH_4/g VS_{added}) that was used as a benchmark for CH_4 production.

- In pit latrines the relatively low BMP values (50.6 mL CH_4/g VS_{added}) demonstrate that there is a significant proportion of organic material that is not readily degradable, with rapid aerobic degradation likely on the surface of the pit latrine. Therefore designing systems using standard rates of VS or COD destruction would lead to overestimation of methane yields.

- The use of odour suppressant chemical toilet additives within portable toilets causes problems such as high Cu concentrations (27251 mg/kg Cu) and the active ingredients glutaraldehyde and bronopol were proven to cause CH_4 production inhibition at IC50 values of ≥100 mg L^{-1} and ≥50 mg L^{-1} respectively.

4. To determine technically feasible methods in which to recover nutrients in a low income context.

- High concentrations of 1440 mg NH_4-N L^{-1} in AD effluents presented a valuable nutrient recovery resource. Phosphorus concentrations (26950 mg P_{tot} kg^{-1}) although significant, were bound to particulate matter with low soluble concentrations of 27 mg P_{sol} L^{-1} making NH_4-N the more desirable target for nutrient recovery.

- Current NH_4-N recovery process options (reverse osmosis, NH_3 stripping and struvite precipitation) present significant challenges to application in a low income context primarily due to significant energy needs, high chemical costs and logistical challenges in the supply chain.

- Due to the simplicity and wide application of sludge drying beds in developing countries adapting a hybrid between a sludge drying bed and

an adsorption or ion exchange system is a feasible and realistic target for the recovery of NH_4-N (Chapter 5).

5. To examine the feasibility of using a modified sludge drying bed system for the recovery of nutrients from high solids anaerobic digestion effluents.

- Clinoptilolite had both a superior capacity (12.2 mg NH_4-N/g) as well as the fastest rate of uptake (1.013 h^{-1}) in comparison to biochar exhibiting comparative uptake rates of 5 mg NH_4-N/g and a slower kinetic uptake rate of 0.557 h-1.

- Clinoptilolite is a resilient media that can operate effectively despite high TS and competing cation concentrations (23.8% reduction in the uptake capacity) in the percolate of the drying beds (Chapter 6).

- Increasing contact time (20 mins - 354 mins) reduces the media usage rate (23 – 4 kg/m^3 treated) and increases operational capacity (5 - 60 g NH_4-N /kg clinoptilolite).

- The TS loading for sludge cake filtration to commence on sand is relatively low, at 0.070-0.320 kg TS/m^2 (0.6-0.9 mm sand particle size) and from 0.046-0.500 kg TS/m^2 (0.9-1.2 mm sand particle size) dependent on digestate TS (0.5-2% TS), indicating that the predominant mechanism in sludge drying beds is through cake filtration and not the physical filtration action of the sand.

6. To propose design values for the construction and operation of nutrient recovering drying beds.

- The ion exchange process allows clinoptilolite to capture NH_4-N under high solids loading conditions and its most efficient operation is at long EBCT (>354 mins). This ensures that the maximum fertiliser value is obtained (60 g NH_4-N/kg clinoptilolite) and optimum removal efficiency from the percolate (95% NH_4-N removal) occurs. It is necessary to operate two beds in series to enable the continual capture of NH_4-N as the first

media bed gradually reaches exhaustion and effluent concentrations increase.

- A larger media particle size (7 mm) is recommended despite reductions in capacity (26% reduction in NH_4-N capacity in comparison to a 1 mm particle size) because of the greater TS loading rate before clogging occurred (TS loading capacity 48% greater) decreasing the rate at which clogging of the bed occurs, leading to inhibitory pressure drops in the bed. Bed depth should also be maintained below 0.33 m to avoid inhibitory pressure drops in the system.

- To reduce the footprint and intensify nutrient recovery sludge drying beds the use of a sacrificial sand bed (0.05 m depth) is necessary in which semi-dewatered sludge cake can be scrapped away and applied to a secondary drying bed once cake filtration has commenced (0.5 kg TS/m^2) and percolation rate has reduced. This will suitably extend the service time of the bed to enable full saturation of the clinoptilolite to take place enabling efficient capture of NH_4-N by the media.

- Clinoptilolite can effectively be used as part of a sludge drying bed configuration to remove NH_4-N from digestate and the saturated media can be used directly as a fertiliser product or could additionally be blended with the dried solid fraction to create a balanced NPK fertiliser product (5.9 wt. % NH_4-N/4.2 wt. % P/≥6.0 wt.% K^+) with minimal capital and operational expenditure.

10 APPENDICES

Appendix A Description of sampling sites and detailed sampling methodologies

A.1 Sampling sites

Survey sites in peri-urban localities of *location x* and Kumasi, Ghana were selected for the detailed characterisation of faecal sludge (FS). Four different types of FS were selected for sampling and analysis: in *location x* pit latrines and unsewered public ablution blocks as well as portable toilets and an intermediate bulk container (IBC) that was in use as a holding tank for portable toilet waste in Kumasi.

Location x is located on the central African plateau, 1300 m above sea level. Temperatures are moderate with a maximum average temperature of 31.2^{o} C (October) and minimum average temperature of 9.6^{o}C (July); there is an average annual rainfall of 803 mm with the majority of this falling in the rainy season between November and April. *Location x* is a peri-urban district of a African city and was used for all FS sampling. The geology of the area is predominantly dolomitic marble with shallow depths of fine sandy soils containing large numbers of laterite pisoliths. Human behavioural practice in *location x* consisted of 'wiping' as an anal cleansing method using a range of different available materials (e.g. toilet paper, newspaper, vegetation) all of which were disposed within the pit latrine or flush toilet bowl.

A pilot scheme using portable chemical toilets was selected in Kumasi, Ghana. Kumasi is approximately 260 m above sea level and has a wet semi-equatorial climate with temperatures averaging 28°C and has an annual average rainfall of 1340mm (Keraita et al., 2003). Common user behavioural practice in Kumasi was 'wiping' for anal cleansing practices with toilet paper being disposed of in the toilet bowl along with faeces only (urine diverting toilets).

A.2 Pit latrines detailed sampling methods

In *location x*, a manual emptying team was accompanied over a period of one month in March 2013, during which samples were taken from a total of ten pit latrines, one UDDT, as well as from three holding tanks from community ablution blocks within the

district of *location x*. Sites were selected by accompanying the team on alternate days with the first site visited on each day chosen for sampling. The approximate age of FS was estimated through questioning of the clients when the previous emptying took place.

Pit latrines were emptied manually using an assortment of tools (long handled shovel, pitch fork) into 60 L barrels. A sub sample of 0.5 L was taken from the centre of each barrel of FS removed by a modified sample jar and drain rod, and subsequently combined together to make one composite sample (6-12 L) representative of the entire contents removed from the pit. An additional pair of samples were collected in December 2013, one from the surface layer and one sample from the bottom section of the pit latrine, in order for a comparative analysis to be undertaken of the different layers.

A.3 Portable toilets detailed sampling methods

Portable toilets were selected at random. In total, 36 toilets were sampled and analysed over a period of 6 weeks in July/August 2013. In order to gain a homogenous sample toilet units were firstly manually mixed for 5 minutes before three samples (0.5 L) were taken providing a vertical cross section of the unit. Samples were then combined and manually mixed again before a final sub sample (0.5 L) could be taken. A vertical cross section of the IBC storage tank was also constructed by taking 0.5 L grab samples at depths of 0.3 m intervals from the surface to the base of the tank. Accumulation rates and chemical analysis, expressed as per capita/day, were calculated from the number of users divided by the frequency of waste collection and excluding the initial volume of chemical additive.

References

Keraita, B., Drechsel, P. and Amoah, P. (2003), "Influence of urban wastewater on stream water quality and agriculture in and around Kumasi, Ghana", *Environment and Urbanization,* vol. 15, no. 2, pp. 171-178.

X Water and Sewage Company (2013), *Environmental and social impact assessment report: drainage report,* 1, USACE Europe District/Millennium Challenge Corporation, Ministry of Local Government and Housing.

Appendix B Analytical Methodology

B.1 DNA extraction and sequencing methodology

Homogenous sludge samples were taken from11 pit latrines and 3 public toilets as well as additional samples from the top (0.05 m), middle (1 m) and bottom (2.5 m) of an additional pit latrine (total depth 2.9 m) were stored at -20°C prior to DNA extraction. Extraction and purification was carried out using a Fast DNA Spin Kit for Soils (MP Biomedical, Irvine, USA) according to manufacturer's guidelines. DNA extractions were quantified using the Broad-Range Qubit Assay (Life Technologies, Darmstadt, Germany) and stored at -20°C until used in library preparation methods for Next Generation Sequencing (NGS). Library construction was applied to all DNA samples. Prior to PCR preparation, each DNA sample was thawed, normalised to 1 ng/µl template DNA by dilution in PCR grade water (Qiagen Nuclease Free water, Cat No. 129115), and stored at -20°C until PCR preparation. Golay barcoded PCR primers (F515/R806) were used for amplification of the V4 region of the 16S rRNA gene (Caporaso et al., 2012). An additional degeneracy on the Golay primers was utilised; base-N substituted for base-C in third position of F515 forward primer; for improved detection of Archaea. Each sample processed was amplified in triplicate 25 µl reactions. PCR amplification reactions were prepared using reagents from the KAPA HiFi HotStart PCR Kit with dNPTs (KAPA Biosystems) and a single 25ul reaction contained: 11ul PCR grade water (Qiagen as previous), 5ul 5X KAPA HiFi Fidelity Buffer, 0.75ul dNTP Mix, 0.75ul of forward and reverse primers at 0.3uM concentration, 0.5ul KAPA HiFi Taq, 6.25 µl template DNA at 1ng/µl concentration. Reactions were held at 950C for 5 minutes then 980C for initial denaturation of the DNA, with amplification proceeding for 25 cycles of 980C for 20 seconds denaturation, 600C for 15 seconds for annealing, and 720C for 40 seconds for extension, followed by 720C for 1 minute final extension. Triplicate reactions were pooled and PCR product was gel-purified (Zymoclean Gel DNA Recovery Kit) and quantified prior to sequencing using the High-Sensitivity Qubit Assay (Life Technologies). PCR products were normalised to 5 ng/µl DNA in nuclease free water, pooled to a total volume of 50 µl and stored at -20°C until sequenced. The

pooled multiplexed library was sequenced using the Illumina Miseq bench-top sequencer at the Centre for Genomic Research, Liverpool.

References

Caporaso, J. G., Lauber, C. L., Walters, W. A., Berg-Lyons, D., Huntley, J., Fierer, N., Owens, S. M., Betley, J., Fraser, L. and Bauer, M. (2012), "Ultra-high-throughput microbial community analysis on the Illumina HiSeq and MiSeq platforms", The ISME journal, vol. 6, no. 8, pp. 1621-1624.

Appendix C Images of on-site sanitation facilities sampled

Pit latrine (a)

Unsewered public toilet (b)

Portable household toilet c)

Figure A - 1 On-site sanitation facilities in which sampling took place in faecal sludge characterisation work. Pit latrines (*location x*) were predominantly emptied from a hole in the side wall (a), unsewered public toilets were emptied from a specially designed emptying port (b) and portable toilets were emptied direct from the collection chamber (c).

Pit Latrine Top   *Pit Latrine Bottom*

Figure A-2 Comparative photos detailing the top and bottom sections of pit latrines in *location x.*

Appendix D Supplementary faecal sludge characterisation data
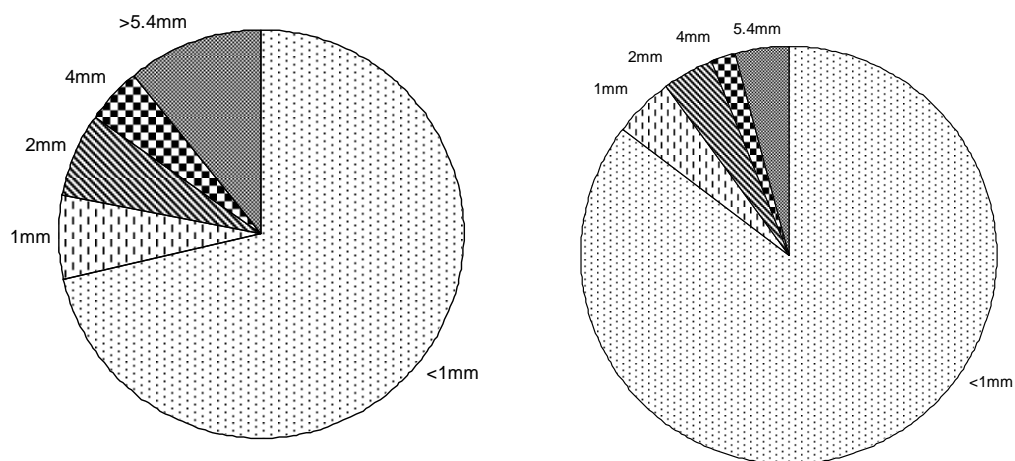
D.1 Particle Size distribution

Figure A-3 Particle size distribution (mean values) of a) pit latrines and b) public toilet holding tanks in *location x* **determined on the bulk sample collected from each on-site sanitation facilities**

D.2 Volatile Fatty Acids

Table A-1 Volatile fatty acid (VFA) concentrations in pit latrines, unsewered public toilets, portable toielts adn a holding tanks for portable toilet waste

	Pit Latrines	Unsewered Public toilets	Portable Toilets	Portable Toilet IBC
Acetic	2057 (1207)	452 (456)	1449 (786)	1745 (874)
Propionate	851 (998)	517 (595)	2170 (1150)	1839 (1055)
Iso-butyric	47 (76)	*	*	*
n-butyrate	146 (203)	*	1118 (516)	1058 (338)
Iso-valeric	204 (293)	*	677 (331)	1018(523)
n-valeric	430 (566)	26 (37)	1767 (1313)	3763(1353)
Total VFA	3736 (2810)	996 (1089)	7182	9423

Appendix E Images of Experimental Apparatus and Rigs

E.1 Biochemical methane potential and toxicity assays

Figure A-4 Biochemical methane potential (BMP) assay consisting of substrate and inoculum (at VSseed :VS substrate ratio of 2:1),deionised water to bring working volume to 60 mL and nutrient stock solution. Samples were incubated in a temperature controlled shaker at 37.5°C ± 0.5 at 150 RPM.
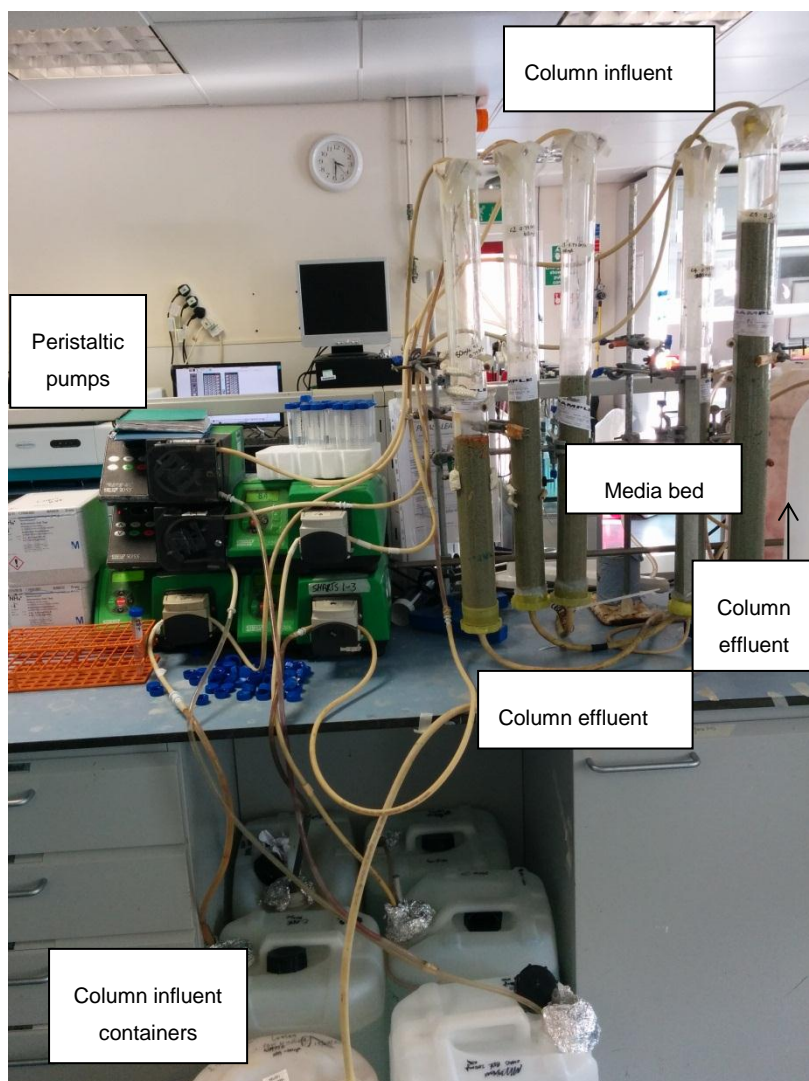
E.2 Lab scale dynamic column experiments

Figure A-5 Experimental rig for the investigation of operational capacity of media at different empty bed contact times, bed depths, NH$_4$-N concentrations and flow regimes.

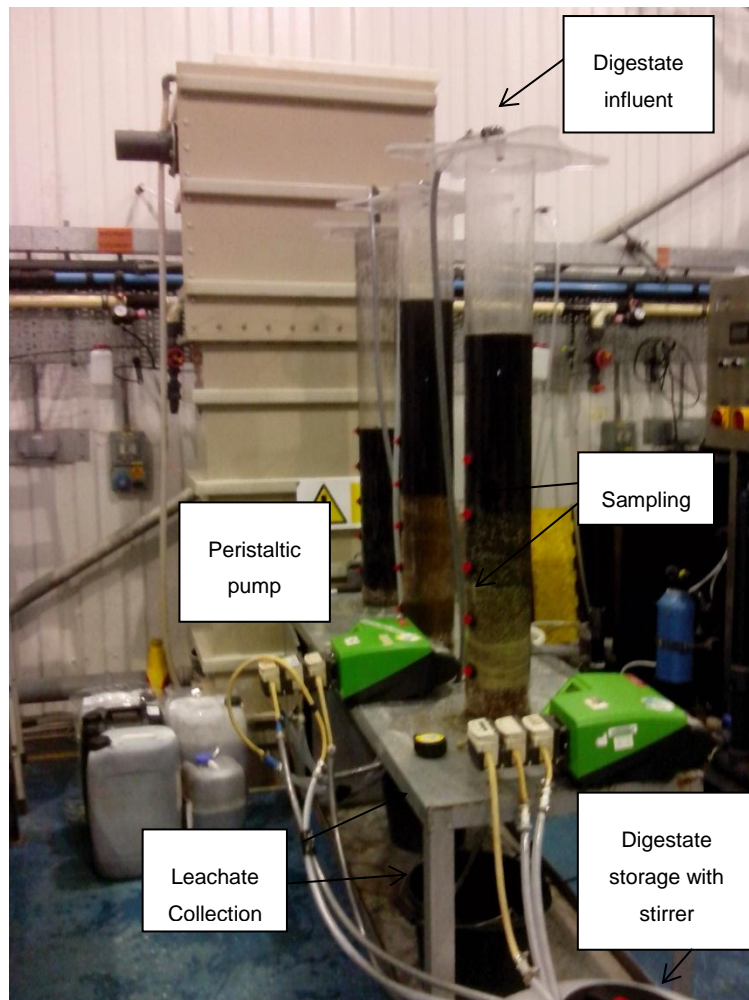## E.3 Pilot scale dynamic column experiments and percolation bed studies

Figure A-6 Pilot scale filter columns used for application with anaerobic digestate, large media particle sizes and batch fed percolation tests

Appendix F Additional results

F.1 Media physical and chemical properties

Figure A-7 Chemical composition (wt. %) of clinoptilolite and sand used in batch and dynamic experimental work

	Element	Clinoptilolite (wt. %)	Sand (wt.%)
SiO_2	Silica	70.5	96.36
Al_2O_3	Alumina	12.5	0.29
Fe_2O_3	Iron	0.72	2.49
TiO_2	Titania	0.10	0.024
CaO	Calcium	3.11	0.02
MgO	Magnesium	0.85	0.03
Na_2O	Sodium	0.13	0.04
K_2O	Potassium	2.14	0.04
P_2O_5	Phosphate	0.04	0.01
SO_3	Sulphate	0.11	-
L.O.I	Loss on ignition (1000ºC)	9.57	0.55