JULEN MENDIZABAL BENGOETXEA

UNDERSTANDING THE IMPACTS OF SEPTICITY ON WASTEWATER TREATMENT

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Academic Year: 2017 - 2022

Supervisor: Prof. Ana Soares
Associate Supervisor: Dr. Yadira Bajon Fernandez
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ABSTRACT

Wastewater septicity develops during wastewater conveyance through the sewerage network to the wastewater treatment plant (WWTP). The problems related to septicity have been mainly researched in sewerage networks and are almost exclusively related to hydrogen sulphide, such as concrete corrosion and odour nuisance. The aim of this work is to better understand the mechanisms governing septicity in wastewater and mitigate the impacts both in sewers and wastewater treatment plants. For doing so, a septicity measure that captures the key indicators was developed, which include sulphide, oxidation reduction potential (ORP), pH, soluble COD and ammonia. Furthermore, the impacts of septicity on a conventional wastewater treatment plant consisting of a primary settler, activated sludge plant and secondary settler were tested. Septic wastewater with 6.4 mg/L of sulphide was found to impact activated sludge flocs, with significant proliferation of filamentous bacteria, resulting also in a reduced COD removal by 55% and nitrification by 44%. Furthermore, sludge bulking in the secondary settler and consequent biomass washout was observed. Additionally, the impact on chemical phosphorus removal (CPR) was tested and septic wastewater was found to reduce the effectiveness of CPR starting at a 0.35 S:Fe molar ratio and only 10% phosphorus removal efficiency was measured at a 1.4 S:Fe molar ratio. Finally, a novel dissolved sulphide sensor was trialled to monitor sulphide at the inlet chamber of a WWTP. The data collected allowed the assessment of the efficiency of nitrate dosing at a rising main. Furthermore, it allowed to build up a data-driven sulphide prediction model utilising readily available data. Overall, the thesis provided the starting bricks for the development of a septicity management framework and highlighted that optimised nitrate dosing at the study rising main utilising the dissolved sulphide data was the most economic septicity management option.

Keywords:

Sewer, hydrogen sulphide, sulphate reducing bacteria, corrosion, activated sludge, chemical phosphorus removal, calcium nitrate, sulphide sensor, inhibition, bulking

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LIST OF ABBREVIATIONS

AD	Anaerobic digester
AGS	Aerobic granular sludge
AMO	Ammonia monooxygenase
anMBR	Anaerobic membrane bioreactor
ANN	Artificial neural network
AOB	Ammonia oxidising bacteria
ASM	Activated sludge model
ASP	Activated sludge plant
BNR	Biological nutrient removal
CAPEX	Capital expenditure
CAS	Conventional activated sludge
COD	Chemical oxygen demand
CPR	Chemical phosphorus removal
CSI	Combined septicity index
DO	Dissolved oxygen
DWF	Dry weather flow
EBPR	Enhanced biological phosphorus removal
EPS	Extracellular polymeric substances
FNA	Free nitrous acid
HRT	Hydraulic retention time
IVS	Immobilized volatile solids
LSTM	Long short-term memory
MBBR	Moving bed biofilm reactor
MBR	Membrane bioreactor
MICC	Microbially induced concrete corrosion
MLSS	Mixed liquor suspended solids
MWWTP	Municipal wastewater treatment plant
NLR	Nitrogen loading rate
NOB	Nitrite oxidising bacteria
NPV	Net present value
NSE	Nash-Sutcliffe efficiency
OLR	Organic loading rate

OPEX	Operational expenditure
ORP	Oxidation reduction potential
PAO	Phosphorus accumulating organisms
PE	Population equivalent
PHA	Polyhydroxyalkanoates
PS	Primary sludge
PSD	Particle size distribution
PST	Primary settlement tank
RAS	Return activated sludge
RBC	Rotating biological contactor
rb-COD	Readily biodegradable chemical oxygen demand
RM	Rising main
RMSE	Root mean square error
SAS	Surplus activated sludge
SBR	Sequencing batch reactor
sCOD	Soluble chemical oxygen demand
SDEV	Standard deviation
SI	Septicity index
SOB	Sulphide oxidising bacteria
SRB	Sulphate reducing bacteria
SRF	Specific resistance to filtration
SRT	Sludge retention time
SVI	Sludge volume index
TF	Trickling filter
TSS	Total suspended solids
UASB	Upflow anaerobic sludge blanket
UK	United Kingdom
VFA	Volatile fatty acids
VSS	Volatile suspended solids
WATS	Wastewater aerobic/anaerobic transformations in sewers
WW	Wastewater
WWTP	Wastewater treatment plant

1 INTRODUCTION

Sewerage networks are built to transport wastewater from where it is originated to centralised treatment facilities known as wastewater treatment plants (WWTPs). Sewerage networks are divided into different types of sewers, namely, gravity sewers and rising mains (or pressure sewers). Gravity mains are commonly partially filled (although they can be completely filled depending on hydraulics), whereas rising mains are completely filled. When wastewater travels through rising mains, the microorganisms contained in the wastewater and on the rising main biofilms consume the dissolved oxygen (DO). If the wastewater contains nitrate, the microorganisms will then consume nitrate until the wastewater has practically no DO or nitrate. At that point, the wastewater is in an anaerobic stage, also known as septicity. During the anaerobic stage, biochemical processes happen that change the characteristics of the wastewater. The most important changes are the sulphate reduction to sulphide by the sulphate reducing bacteria (SRB) activity and the hydrolysis and fermentation of organic matter resulting in the accumulation of VFAs (Hvitved-Jacobsen, Vollertsen and Nielsen, 2013). Several factors can impact the extent of the biochemical changes, mainly, the hydraulic retention time on the rising main, the wastewater COD and the temperature of the wastewater (Boon, 1995).

Wastewater septicity is a major problem in sewerage networks and WWTPs, as it contains sulphide that can volatilise as hydrogen sulphide and cause concrete and metal corrosion as well as being an odour nuisance and toxic to humans (Zhang *et al.*, 2008; Park *et al.*, 2014). In the last decade and presumably in the following ones, septicity issues are expected to increase, due to the water reduction strategies being applied at household levels (Sun *et al.*, 2015), which will result in a more concentrated but less volume of wastewater. Furthermore, climate change and global warming are going to further increase septicity issues (Cintra Campos and Darch, 2015). Therefore, it is paramount that septicity management options are available and can be economically feasible, as they are currently very costly (Vinck *et al.*, 2017). Dosing chemicals is the most popular method for septicity control. Currently, calcium nitrate dosing, aeration and ferric salts dosing are the most common methods (Ganigue *et al.*, 2011). As with most chemical dosing, optimising the dose based on the target parameter is essential to achieve its maximum potential. However, septicity is not a parameter and the most commonly associated compound, sulphide, is not trivial to monitor. Therefore, improved and precise real-time measurement of sulphide is required to optimise septicity management (Sutherland-Stacey *et al.*, 2008).

Wastewater septicity is also known to cause inefficiencies at WWTPs, with activated sludge bulking being reported at activated sludge sites with septicity issues and nitrification inhibition studies being published in the presence of sulphide (Echeverria, Seco and Ferrer, 1992; Bejarano-Ortiz *et al.*, 2015; Delgado-Vela, Dick and Love, 2018). However, there is commonly no link between septicity studies at sewerage networks and the impacts at the downstream WWTPs, even though septicity management needs to take into account not only corrosion and odour issues, but also the inefficiencies at the WWTPs.

1.1 Aims and Objectives

The overall aim of the thesis was to understand the mechanisms governing septicity in wastewater and mitigate the impacts both in sewers and at the wastewater treatment plant.

To achieve the aim, the following objectives were defined:

1. To provide a state of the art review on the impacts of septicity on wastewater treatment processes and identify gaps in knowledge
2. To identify the key indicators of septicity and develop a scale of septicity for raw wastewater
3. To investigate the impacts of septicity on wastewater treatment processes, including primary settling, chemical phosphorus removal and activated sludge processes and identify risks to utilities and operators
4. To understand the potential of dissolved sulphide sensors to monitor septicity in real-time and evaluate septicity management options such as dosing of nitrate in sewers

5. To develop a sulphide prediction model based on readily available data for a rising main
6. To develop an economical assessment of the potential septicity management options, quantifying the cost-benefits against a base case

1.2 Thesis plan

This thesis is presented as a series of chapters formatted as journal papers. All papers were written by the primary author, Julen Mendizabal and edited by Prof. Ana Soares and Dr. Yadira Bajon Fernandez. Industrial supervision was provided by Dr. Benjamin Martin and Dejan Vernon of Thames Water. All experimental work was designed and completed by Julen Mendizabal at Cranfield University. The full scale sulphide monitoring was designed in collaboration with Dejan Vernon (Thames Water) and was primarily run by Julen Mendizabal with help from Thames Water on the installation and calibration of the sensors.

Chapter 2 addresed objective 1 and investigated the scientific evidence on the impacts of septicity on most of the WWTP processes, both positive and negative impacts. The findings were utilised to design the experimental plan of Chapter 4.

Chapter 3 addressed objective 2 and investigated the key indicators of septicity in raw wastewater, by performing batch incubation tests of raw wastewater and monitoring the potential key indicators identified in a literature review (not included in the thesis). The key indicators were then used to develop a septicity measurement to create an alarm system, rather than just focusing on sulphide.

Chapter 4 addressed objective 3 and investigated the impacts of septic raw wastewater on the efficiency and operation of a typical WWTP, PST followed by an ASP and FST, at pilot scale. Furthermore, the impact on chemical phosphorus removal was also studied using jar tests. The results were then used as part of the economic assessment in Chapter 6.

Chapter 5 addressed objectives 4 and 5 and consisted of a full scale trial of a novel dissolved sulphide sensor at a Thames Water site with clear indication of septicity related corrosion. Three rising mains were monitored to allow prioritising of septicity management. The data collected for one of the rising mains was then utilised to develop a sulphide prediction model based on readily available data (flow, temperature and time of the day) using a long short-term memory (LSTM) model.

Chapter 6 addressed objective 6 and assessed the economic cost benefits of implementing different septicity management chemicals on one of the rising mains investigated in Chapter 5 and on the use of dissolved sulphide sensors. It further investigated the economic potential of dosing chemicals at the downstream section of the rising main rather than at the wet well.

Chapter 7 detailed the overall outputs of the thesis and discussed the contribution to knowledge and areas of future research.

Chapter 8 combines the overall conclusions of the project.

Table 1-1 Thesis plan

Chapter	Paper	Objective	Title
2	1	1	Impacts of septicity on wastewater treatment – A review
3	2	2	Development of a septicity scale for raw wastewater
4	3	3	Impacts of septicity on municipal wastewater treatment plants
5	4	4,5	Use of a novel dissolved sulphide sensor to monitor sulphide and build a prediction model using long short-term memory artificial neural network
6	5	6	Economic assessment of septicity prevention methods at rising mains discharging to wastewater treatment plants
7	-	1-6	Discussion
8	-	-	Conclusions

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2 IMPACTS OF SEPTICITY ON WASTEWATER TREATMENT – A REVIEW

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Abstract

Anaerobic processes that lead to wastewater septicity in sewers are known to change the characteristics of the wastewater. The most important changes are the presence of sulphide, decrease in oxidation reduction potential (ORP), the accumulation of volatile fatty acids (VFAs) and ammonia. The presence of sulphide can reduce the efficiency of chemical phosphorus removal as well as impact nitrification. In enhanced biological phosphorus removal (EBPR) processes the presence of sulphide and VFAs can result in the proliferation of filamentous bacteria and subsequently activated sludge bulking in the secondary settler. Overall, the impact of septicity is not clear in the efficiency of primary sedimentation processes, due to the solids particle size distribution in the septic wastewater; and the role of sulphide on: i) inhibition of nitrifying populations in full-scale WWTPs and ii) definition of the operational ranges at which filamentous bacteria are problematic in aerobic granular sludge (AGS) systems. Three different wastewater treatment plant (WWTP) configurations have been assessed to illustrate the impact septicity would have on their effluent quality and increased total suspended solids, phosphorus and ammonia have been modelled in the effluent.

This review provides an overview of the impacts of septicity on wastewater treatment technologies, which enables a quicker identification of septicity related problems and aids in the decision making of septicity prevention measures.

Keywords: Dewaterability, Bulking, Anaerobic digestion, Sulphide, Inhibition.

2.1 Introduction

In most countries, wastewater is collected at source and transported through sewerage systems to a centralised wastewater treatment plant (WWTP) where it receives treatment to the required standards before is discharged into a receiving water body, usually rivers or the sea. During transport and sometimes at the WWTP, unintended anaerobic reactions happen which lead to septicity (Figure 2-1). Several factors have previously been found to influence septicity formation in sewers, such as hydraulic retention time (HRT), turbulence of the wastewater and strength of the wastewater (Boon, 1995; Hvitved-Jacobsen, Vollertsen and Nielsen, 2013; Sun *et al.*, 2015). During septicity development, most of the wastewater parameters change in a predictable way. Dissolved oxygen (DO) is depleted (Gudjonsson, Vollertsen and Hvitved-Jacobsen, 2002) leading to a reduction in oxidation-reduction potential (ORP) (Kaijun, Zeeman and Lettinga, 1995), while ammonia and soluble chemical oxygen demand (COD) concentrations increase (Æsoy et al., 1997; Bachmann et al., 2007) due to hydrolysis (Figure 2-1 and Table 2-1). After DO depletion, sulphate is reduced to sulphide by sulphate reducing bacteria (SRB) present in the sewer and volatile fatty acids (VFAs) accumulate, which results in a pH decrease to values of 6-8 (Table 2-1) (Guisasola *et al.*, 2008; Sharma *et al.*, 2008).

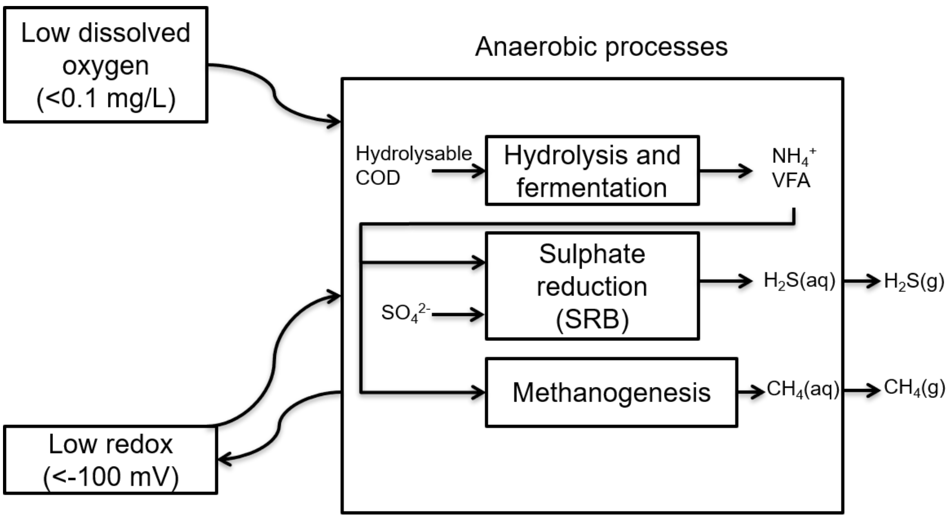

Figure 2-1 Biochemical processes that happen during septicity development

Wastewater septicity has several impacts both at sewer level and WWTPs. The most commonly identified impacts in literature are microbially induced concrete corrosion (MICC) (Nielsen, Raunkjaer and Hvitved-Jacobsen, 1998; Hvitved-Jacobsen, Vollertsen and Matos, 2002), odour nuisance (Talaiekhozani *et al.*, 2016) and health and safety risks to sewer workers (Park *et al.*, 2014). Changes in the wastewater characteristics also lead to inefficiencies and operational problems in WWTPs that have to be taken into account to provide suitable wastewater treatment. Furthermore, the impacts on WWTPs have to be considered when assessing septicity prevention or remediation measures, as these can have a significant influence on the costs of operation. For example, Kulandaivelu et al. (2020) proved that moving ferric dosing to the sewers provided multiple benefits, such as sulphide control in the sewer, phosphorus removal at the WWTP and hydrogen sulphide control in anaerobic digesters, which would significantly improve the economics of iron dosing in sewers.

The impact of septicity in WWTPs has been rarely reported. Some studies describe the individual impacts of septicity (or surrogate parameters) on individual processes, but to date no thorough study has captured the different impacts of septicity on WWTPs. Most research investigates this through modelling whereby sewer models and WWTP models merge by some sort of conversion of the state parameters (Fronteau, Bauwens and Vanrolleghem, 1997). Ashley et al. (2002) assessed the impacts of in-sewer storage of wastewater on a nutrient removing WWTP, which led to septicity development. Furthermore, Sharma et al. (2012) used integrated modelling for decision-making on chemical dosing in sewers to prevent septicity, demonstrating the cost-saving potential of having a holistic approach.

This review combines the knowledge available on the impacts of treating septic wastewater on individual processes and capture the state of the art on the impacts of septicity on WWTPs. Impacts of septicity on effluent quality compliance, depending on the WWTP configuration, were also estimated and discussed, and any research gaps identified. The aim of this review is not to include the control strategies for septicity or its impacts.

2.2 Overview of the impact of septicity on treatment processes

Wastewater treatment plants vary in their design and process selection, although most of them follow the same treatment structure, i.e., preliminary, primary, secondary treatment and sludge stabilisation processes. Wastewater passes through screens and grit removal followed by usually some kind of sedimentation step. The settled wastewater flows to the secondary treatment, which often consists of some form of biological treatment and, when required, is further polished at tertiary treatment. The larger WWTPs also include sludge stabilisation steps, which can include thickening, anaerobic digestion and dewatering. Changes in the influent wastewater characteristics can have implications on the process efficiency and overall effluent quality that can be obtained. Septicity has been demonstrated to induce severe changes in wastewater characterisation, such as 10 to 30% increase in soluble COD, mainly in the form of VFAs that can reach levels of up to 40-120 mg/L, leading to a decrease in pH to values as low as 6 (Table 2-1). Sulphate is reduced to sulphide that accumulates up to 0.5-17 mg S/L and there is a release of ammonia and orthophosphate leading to 10-25% higher concentrations of nutrients compared with fresh wastewater (Table 2-1).

Table 2-1 Characteristic values of fresh and septic municipal wastewater parameters

Parameter	Unit	Fresh WW	Septic WW	Change	Reference
DO	mg/L	0.5 – 3	0	Decrease 100 %	(Gudjonsson, Vollertsen and Hvitved-Jacobsen, 2002)
ORP	mV vs SHE	50 – 300	-180 – -310	-	(Kaijun, Zeeman and Lettinga, 1995)(Chapter 3)
pH	-	6.5 – 8.5	6 – 8	Decrease 5–20 %	(Kaijun, Zeeman and Lettinga,

					1995; Rudelle *et al.*, 2016)
Alkalinity	mg $CaCO_3$/L	50 – 200	50 – 200	~0 %	(Rudelle *et al.*, 2016)
sCOD	mg/L	50 – 300	60 – 400	Increase 10-30 %	(Bachmann, Saul and Edyvean, 2007)(Chapter 3)
Ammonia	mg N/L	20 – 60	25 – 80	Increase 10-25 %	(Æsøy et al., 1997; Kaijun et al., 1995)
Sulphate	mg S/L	20 – 60	3 – 30	Decrease 20-80 %	(Chapter 3)
Sulphide	mg S/L	0 – 0.2	0.5 – 17	-	(Æsøy et al., 1997; Hvitved-Jacobsen et al., 1995; Rudelle et al., 2016)
Orthophosphate	mg P/L	1 – 5	1.3 – 6	Increase 20 %	(Æsøy et al., 1997)
VFA	mg/L	10 – 50	40 – 120	Increase 10-100 %	(Bachmann, Saul and Edyvean, 2007; Rudelle *et al.*, 2016)

2.3 Chemical phosphorus removal

Phosphorus is a pollutant and must be removed from wastewater before the effluent is discharged as it causes eutrophication on receiving water bodies. Most of the wastewater discharge permits restrict phosphorus concentrations in the effluent to 0.1 – 2 mg/L (EC, 2000; UKTAG, 2013). Several approaches to phosphorus removal are applied at WWTPs, such as the addition of iron or aluminium based coagulants, enhanced biological phosphorus removal (EBPR) process and the use of adsorption media. The most commonly used process worldwide is the use of coagulants (usually iron-based) to precipitate phosphorus that can be subsequently removed in a clarification or filtration step (Wilfert *et al.*, 2015). Coagulants are added before primary settling, at the biological process and/or after secondary clarification. A parameter that has a direct impact on the coagulant demand is the wastewater orthophosphate concentration, which can increase by 20% due to septicity (Table 2-1). It is well known that the hydrolysis resulting from anaerobic processes occurring in the sewers can release phosphorus contained in organic matter (Æsøy et al., 1998; Sidebotham, 2012). The increased concentration of orthophosphate requires an equal augment in coagulant dose of 20% to reach the required effluent phosphorus concentration.

Furthermore, when septic wastewater is dosed with iron based coagulants, the sulphide present in the septic wastewater can also interact with the ferric ions. Under reducing conditions of < 0 mV ORP, ferric is chemically reduced to ferrous with sulphide as an electron donor (Equation (2-1)). Subsequently, ferrous iron can precipitate with sulphide in the form of ferrous sulphide (Equation (2-2)) (Nielsen *et al.*, 2005; Firer, Friedler and Lahav, 2008), which forms preferentially over iron phosphate due to its lower solubility (Hu *et al.*, 2019; Takashima, 2019; Wilfert *et al.*, 2020). Consequently, in a sulphide rich wastewater such as that associated with septic conditions, the coagulant dosage required to remove the same amount of phosphorus is increased. This dose increase varies based on other wastewater characteristics, but it can be estimated as 1.16 mg Fe/L per 1 mg S/L of sulphide based on stoichiometry. The iron coagulant dosing location which is most susceptible to be impacted by septicity is before the primary settling, (Gutierrez *et al.*, 2008).

$$2Fe^{3+} + HS^{-} \rightarrow 2Fe^{2+} + S^{0} + H^{+} \tag{2-1}$$

$$Fe^{2+} + HS^{-} \rightarrow FeS + H^{+} \tag{2-2}$$

Although it appears logical that ferrous sulphide precipitates would settle in the primary settler, Gutierrez et al. (2010) found that the hydraulic retention time (HRT) between the ferric dosing point and the primary settler determined the settling efficiency of the ferrous sulphide precipitates, reporting a higher settling efficiency at higher HRTs. Furthermore, in the subsequent aerobic biological process, the sulphide from the ferrous sulphide precipitates oxidises to sulphate and the iron is re-available to remove further phosphate. Hence, the increase in coagulant demand would not be as high as the 1.16 mg Fe/L per 1 mg S/L of sulphide stoichiometrically estimated, as long as the HRT between the dosing point and the primary settler is kept lower than two hours (Gutierrez *et al.*, 2010). Contrarily, the ferrous iron formed in environments with < 0 mV ORP can precipitate with phosphate to form vivianite (Wilfert *et al.*, 2015, 2016, 2018; Roussel and Carliell-Marquet, 2016), which was proven to be recoverable from sludge by magnetic separation (Prot *et al.*, 2019), offering the opportunity to recover both iron and phosphorus (Salehin *et al.*, 2020).

2.4 Primary settlement

Primary settlement is a solid-liquid separation process, where total suspended solids (TSS) present in the wastewater are removed by gravity and collected at the bottom of the tank that then needs periodical desludging. The most important control parameter in a primary settler is the sludge blanked height so that anaerobic conditions do not develop (Tchobanoglous *et al.*, 2014). Septic influents are already anaerobic or are prone to develop such conditions (Table 2-1). As such, if the primary settler desludging frequency is not adequately adjusted, anaerobic conditions can prevail, leading to gas formation and rise of settled sludge (Tchobanoglous *et al.*, 2014). In the WWTPs where return liquors from the sludge dewatering or thickening steps are recycled to the head of the works, anoxic conditions (no oxygen but nitrate present) can prevail in the primary settling tank leading to denitrification and conversion of nitrate to nitrogen gas, which again results in rising of settled sludge (van Dijk, Pronk and van Loosdrecht, 2018).

In septic sewers, enzymatic hydrolysis impacts the TSS and COD characteristics (Elmitwalli *et al.*, 2001). Æsøy et al. (1998) measured a reduction in the settling efficiency of TSS due to septicity conditions in a pilot plant, which was thought to be linked with changes in TSS particle size distribution due to septicity. In addition, soluble COD is poorly removed in solid-liquid separation processes, reducing the efficiency of the primary settler and leading to increases in the COD load to the downstream biological processes (Ashley *et al.*, 2002). Many WWTPs are operated at their design load or over-loaded, which means that they will not be able to cope with a further increase in COD loading (European Commission Urban Waste Water, 2016). Even if the biological process is not under stress, aerobic processes incur a higher aeration demand, which result in higher operational costs. Considering the average aeration energy need of 0.49 kWh/kg COD (Longo *et al.*, 2019) and the 10-30 % increase in soluble COD expected from septicity (Table 2-1), the operational cost on aeration would increase by 0.7-7 £/ML treated if septic conditions in the incoming sewage are developed (increase in sCOD of 10 mg/L and 100 mg/L respectively).

2.5 Biological processes

After removal of most of the suspended solids in the primary settler, the bulk of the wastewater COD is treated in biological processes of different nature, depending on the context of the treatment plant. The biological processes can be anaerobic or aerobic treatment. In the first, mainstream anaerobic treatment processes such as upflow anaerobic sludge blanket (UASB) and anaerobic membrane bioreactor (anMBR), benefit from the hydrolysis of TSS during septicity development, as hydrolysis is the rate limiting step in mainstream anaerobic treatment (Mahmoud *et al.*, 2004). Aerobic treatment processes can be further divided into suspended growth and biofilm processes and these can be impacted by septic wastewater in different ways as described below.

2.5.1 Suspended growth processes

The most common suspended growth technology in WWTPs is the activated sludge process (ASP), which can have varied configurations, but usually includes an aerated basin and a settling tank. Due to tightening of discharge standards, which include nutrients such as ammonia, total nitrogen and phosphorus (Defra, 2002), several more complex configurations of the ASP such as biological nutrient removal (BNR) and enhanced biological phosphorus removal (EBPR) are now widely applied (Tchobanoglous *et al.*, 2014).

2.5.1.1 Enhanced biological phosphorus removal (EBPR)

The EBPR process is comprised of an anaerobic stage followed by an aerobic stage. In the anaerobic stage, polyphosphate accumulating organisms (PAOs) uptake readily biodegradable COD, particularly volatile fatty acids (VFAs) and store them intracellularly as polyhydroxyalkanoates (PHAs) using energy generated from the hydrolysis of polyphosphate (Ong *et al.*, 2016). In the subsequent aerobic stage, PAOs use the stored PHAs as carbon source and accumulate phosphorus intracellularly as polyphosphate (Comeau *et al.*, 1986; Zhang *et al.*, 2013). Wastewater with low readily available COD was found to be one of the major causes of full-scale EBPR failures (Guerrero *et al.*, 2012). To compensate for the lack of carbon source, chemicals such as glycerol (Guerrero *et al.*, 2012), acetate (Wei *et al.*, 2014; Hu *et al.*, 2018), ethanol (Hu *et al.*, 2018) and an acetate-starch mix (Wei *et al.*, 2014) are added. Alternatively, the sludge produced on the WWTP can be used as a source of VFAs through sludge fermentation or disintegration (Kampas et al., 2009, Soares et al., 2010). The increase in VFA availability up to concentrations of 120 mg/L, especially acetate, due to septicity in the influent (Table 2-1) is beneficial for the EBPR process performance (Rudelle *et al.*, 2011, 2012). Ashley et al. (2002) assessed the impacts of septicity in the influent of an EBPR process combining the WATS sewer model (Hvitved-Jacobsen, Vollertsen and Nielsen, 2013) and the ASM2 model (Gujer *et al.*, 1995) and they found that the VFAs generated during septicity were essential to maintain the EBPR process performance. Even though the increase in VFA availability due to septicity might not be enough to sustain the EBPR process, it leads to a reduction in the addition of external carbon sources and consequently, operational costs.

On the side, in the anaerobic stage of the EBPR, sulphate reduction can also take place (Yamamoto, Komori and Matsui, 1991; Yamamoto-Ikemoto *et al.*, 1998; Baetens *et al.*, 2001; Rubio-Rincon, C. Lopez-Vazquez, *et al.*, 2017). Sulphate reducing bacteria (SRB) and PAO can coexist in the anaerobic stage (Figure 2-2), but there are contradictions in literature on the competition versus symbiosis between them. Yamamoto et al. (1991) observed that an increased SRB activity in the anaerobic stage reduced phosphate removal on the EBPR process. On a later study, the same authors reported that SRBs and PAOs worked in symbiosis as lactate and propionate were consumed by SRBs to produce acetate and acetate was subsequently consumed by PAOs (Yamamoto-Ikemoto *et al.*, 1998).

Furthermore, Rubio-Rincon et al. (2017a) found that the acetate and propionate consuming SRBs were more sensitive to oxygen than the lactate consuming SRBs. The latter increased the acetate and propionate concentrations and led to an increased phosphorus release and PHA accumulation by PAOs (Figure 2-2).

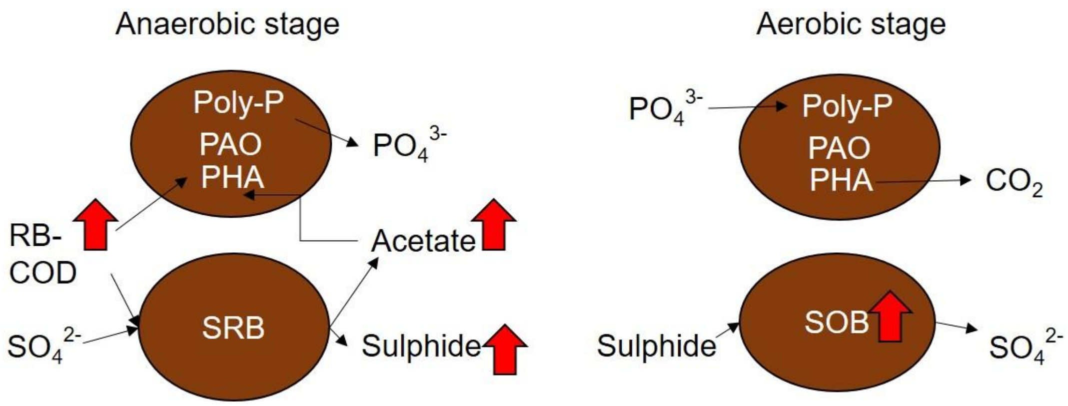

Figure 2-2 Interaction between sulphate reducing bacteria (SRB) and polyphosphate accumulating organisms (PAOs). Brown circles represent bacteria and red arrows show the impacted parameters due to the presence of septicity. RB-COD is readily biodegradable COD; SOB is sulphide oxidising bacteria.

Septic wastewater contains 0.5-17 mg S/L of sulphide, which can be further increased in the anaerobic stage of the EBPR process (Rubio-Rincon, C. Lopez-Vazquez, *et al.*, 2017). Contrarily, other studies suggest sulphide is oxidised in the aerobic stage by sulphide oxidising filamentous bacteria, which can lead to poor sludge settling (Figure 2-2) (Yamamoto, Komori and Matsui, 1991; Yamamoto-Ikemoto *et al.*, 1998; Baetens *et al.*, 2001; Valverde-Perez *et al.*, 2016; Rubio-Rincon, Welles, *et al.*, 2017). Yamamoto et al. (1991) observed in a EBPR pilot-plant the proliferation of the filamentous bacteria Type 021N when septic wastewater was fed that led to sludge volume indexes (SVI) higher than 500 mL/g and biomass washout. Similar observations were reported in a lab sequencing batch reactor (SBR) where a higher availability of sulphide led to development of sulphide oxidising filamentous bacteria and consequently sludge bulking (Yamamoto-Ikemoto *et al.*, 1998; Baetens *et al.*, 2001; Rubio-Rincon, Welles, *et al.*, 2017).

Sulphide concentrations higher than 8 mg H_2S-S/L have been shown to inhibit the anaerobic and mostly the aerobic metabolism of *Candidatus* Accumulibacter phosphatis type I (Rubio-Rincon, C. M. Lopez-Vazquez, *et al.*, 2017; Saad *et al.*, 2017). The inhibition was suggested to result from the diffusion of H_2S through the cell membrane and the alteration of the intracellular pH after the disassociation of sulphide inside the cell. However, the sulphide concentrations on a study by Rubio-Rincon et al. (2017c) were in the range of 42-189 mg TS-S/L and the inhibition constant for the anaerobic PAO metabolism was determined to be of 58.8 mg H_2S-S/L (Saad *et al.*, 2017), which are much higher than the sulphide concentrations of 0.5-17 mg TS-S/L found in septic wastewaters.

2.5.1.2 Nitrification inhibition

Nitrification is very sensitive to metals, inorganic and organic compounds (Arp, Sayavedra-Soto and Hommes, 2002; Singh and Verma, 2007). Sulphide, which is present in septic wastewaters, has been identified as a strong inhibitor to nitrification (Bejarano-Ortiz *et al.*, 2015), with different hypotheses regarding the mechanisms of sulphide inhibition formulated in literature (Delgado-Vela, Dick and Love, 2018). Sears et al. (2004) observed 96-100% inhibition on nitrification rates at sulphide concentrations of 0.5-3.5 mg S/L. However, when sulphide was completely removed, the nitrification activity was immediately recovered by 51-74%. Two modes of nitrification inhibition were suggested in this study: i) non-competitive inhibition, where the sulphide molecule attaches to the ammonia monooxygenase (AMO) enzyme at a different location than the active site hence changing its structure and inactivating the enzyme (Sears *et al.*, 2004); ii) metals such as copper were shown to play an important role in AMO activity (McCarty, 1999) and certain sulphur compounds, such as sulphide, can form complexes which can chelate copper and inhibit AMO activity (Juliette, Hyman and Arp, 1993). The latest studies support inhibition of both AOB and NOB populations through the non-competitive mechanism (Bejarano-Ortiz *et al.*, 2015; Kouba *et al.*, 2017; Delgado-Vela, Dick and Love, 2018), that can be modelled according to Equation (2-3).

$$\mu_{inh} = \frac{\mu_{max}[S]}{(1 + \frac{[I]}{K_i})(K_S + [S])} \quad \textbf{(2-3)}$$

where: μ_{inh} is the inhibited specific growth rate; μ_{max} is the uninhibited maximum specific growth rate; [S] is the concentration of electron donor, which in this case is ammonia or nitrite; [I] is the concentration of inhibitor, in this case sulphide; K_i is the inhibition constant; and K_S is the half saturation constant.

Ammonia oxidising bacteria (AOB) and nitrite oxidising bacteria (NOB) have different responses to sulphide and Erguder et al. (2008) observed only partial nitrification after addition of sulphide, indicating that NOBs are more prone to inhibition. The inhibition constants for AOBs are in the range of 1.4-150 mg/L and 0.22-10 mg/L for NOBs, which span by up to two orders of magnitude (Table 2-2). Differences in the inhibition constants were related to the original conditions of the inoculum added to the inhibition batch tests (Kouba *et al.*, 2017; Delgado-Vela, Dick and Love, 2018). Kouba et al. (2017) used an inoculum from an activated sludge plant that received average sulphide concentrations of ~3 mg/L and measured sulphide inhibition constants of 150 mg/L and 10 mg/L for AOB and NOB, respectively (Table 2-2). Contrarily, Bejarano-Ortiz et al. (2015) used sludge that had not been in contact with sulphide before and measured sulphide inhibition constants of 2.54 mg/L and 0.22 mg/L for AOB and NOB, respectively (Table 2-2). The difference between the measured sulphide inhibition constants suggested that activated sludge populations could acclimatise to the presence of sulphide on the long-term (Kouba *et al.*, 2017). Sekine et al. (2020) observed the progressive acclimation of nitrifying sludge when sulphide concentration was stepwise increased to 43 mg/L over 80 days, which induced a nitrifying population shift from *Nitrosomonas europaea* to *Nitrosomonas nitrosa* for AOB and from *Nitrospira* spp. to *Nitrobacter* spp. for NOB.

Few studies have researched whether the presence of soluble sulphides in unaerated or partially aerated zones might lead to nitrification inhibition, conditions which are likely to happen on BNR activated sludge plants fed with septic wastewater (Sears *et al.*, 2004). Zhou et al. (2013) reported that 6.2 mg/L of sulphide did not reduce the specific growth rate of nitrifiers under aerobic conditions, but after 0.5 h of unaerated exposure the specific growth rate was reduced by 76%. Furthermore, they found that the longer the anaerobic exposure with sulphide, the higher the inhibition with a drop in specific growth rate of up to 50% when exposed to sulphide concentrations of 10 mg/L for 1.5 hours. Delgado-Vela et al. (2018) concluded that there is a need for more in-depth experiments to further elucidate the mechanisms.

Table 2-2 Inhibition constants of sulphide on nitrification and the conditions at which they were measured

Sulphide concentration (mg/L)	Inoculum	Test type	DO (mg/L)	T (°C)	pH	$K_{I,AOB}$ (mg/L)	$K_{I,NOB}$ (mg/L)	Main effects	Reference
0-64	0.2 g SS L^{-1} of nitrifying sludge acclimatised to 0-128 mg $(L\ d)^{-1}$ sulphide loads	Batch	Saturated	30 ± 1	7.5 ± 0.05	1.43-5.82	-	Increase in sulphide resistance through biomass acclimation	(Sekine *et al.*, 2020)
2-35	1-1.7 g VSS L^{-1} of A2O process sludge/ 1.9-3 g VSS L^{-1} extended aeration sludge	Batch	>2	25	7.71	7.8-14	2.4-6.7	Decrease in AOB and NOB activity; NOB population diversity reduced the extent of inhibition	(Delgado-Vela, Dick and Love, 2018)
13-178	Nitrifying-denitrifying sludge 1.32 ± 0.17 g VSS L^{-1}	Batch	3-9	15 ± 0.5	7.9-8	150	10	Decrease in AOB and NOB activity	(Kouba *et al.*, 2017)
2.5-5	Nitrifying sludge 65 ± 6 mg L^{-1} microbial protein	Batch	3-6	30	7.5 ± 0.2	2.54 ± 0.12	0.22 ± 0.03	Decrease in AOB and NOB activity	(Bejarano-Ortiz *et al.*, 2015)

0-50	Activated sludge from A2O plant 2.66-2.98 g TSS L^{-1}	Batch	Anaerobic 1h then >4	20	7-9	36	-	Decrease in NH_4^+ utilisation rate after anaerobic contact with sulphide	(Zhou *et al.*, 2013)
3.1-112	Nitrifying sludge 320 ± 16 mg VSS L^{-1}	Batch	3-6	30	8 ± 0.4	2.6 ± 0.3	1.2 ± 0.2	Decrease in AOB and NOB activity	(Bejarano-Ortiz *et al.*, 2012)
1.7-18	Nitrifying biofilm 3.5 g VSS L^{-1} + 0.5 g IVS L^{-1}	Batch	4.5-5.5	24 ± 0.5	7-7.6	13	-	Decrease in the specific rates of NH_4^+ consumption and NO_3^- formation	(Beristain-Cardoso, Gomez and Mendez-Pampin, 2010)
1.3-80	Nitrifying sludge 2.7 ± 0.06 g VSS L^{-1}	SBR	4.6-8.6	23-28	7.5	-	-	Increase in the NO_2^--N to (NO_2^--N + NO_3^--N) accumulation ratio	(Erguder *et al.*, 2008)
0-3.5	Nitrifying sludge 1 g VSS L^{-1}	Batch	2-6	-	7-8	-	-	Decrease in the volumetric rate of NH_4^+ consumption	(Sears *et al.*, 2004)

0-2.5	-	MBBR pilot	5-8	14	7.2-7.7	-	-	Decrease in the volumetric rate of NH_4^+ consumption	(Æsøy et al., 1998)
2-5.4	-	Full-scale trickling filter	-	12-22	-	-	-	Decrease in the NH_4^+ consumption efficiency	(Bentzen *et al.*, 1995)

IVS: Immobilised volatile solids; MBBR: Moving bed biofilm reactor

2.5.1.3 Sludge bulking in secondary clarifiers

Secondary clarifiers promote the physical separation of solids and liquid and enable retention of the activated sludge biomass in ASP, while maintaining a clear effluent to meet the discharge consents. Due to improper operation or environmental factors, activated sludge flocs change its structure and reduce their settling efficiency, with filamentous bacteria overgrowth being the most common one. Several filamentous species are able to oxidise sulphide and store sulphur granules intracellularly, such as *Thiothrix* spp., Type 021N and *Beggiatoa* spp. (Nielsen, de Muro and Nielsen, 2000; Richard, 2003). Due to their high affinity for acetate (*Thiothrix* spp. 2.4 μM and Type 021N 1.8 μM), combined with the affinity for sulphide, both present in septic wastewater, these microorganisms can proliferate quickly, changing the structure of the ASP flocs and their settling properties (Table 2-3). Sulphide concentrations higher than 1 mg/L were found to increase the SVI of an activated sludge >200 mL/g and to cause bulking problems, although the specific filamentous morphotype was not identified (Echeverria, Seco and Ferrer, 1992).

Table 2-3 Filamentous bacteria observed to grow on ASPs fed with septic wastewater and their growth conditions

Bacterial specie	Conditions	References
Zoogloea ramigera	High organic acids and high F/M	(Seviour, 2010)
Type 021N	Presence of H_2S and septic wastewater, low F/M and long SRT	(Andreasen and Nielsen, 1997; Nielsen *et al.*, 1998)
***Thiothrix* spp.**	Presence of H_2S and septic wastewater, nitrogen or phosphorus deficiency, readily biodegradable soluble substrates	(Nielsen *et al.*, 1998, 2003; Nielsen, de Muro and Nielsen, 2000; Nielsen, Aquino and Nielsen, 2003)
***Beggiatoa* spp.**	Presence of H_2S and septic wastewater	(Williams and Unz, 1989; Baetens *et al.*, 2001)
N. *Limicola*	Readily biodegradable soluble substrates	(Seviour *et al.*, 2006)
Microthrix parvicella	Slowly biodegradable or particulate substrates and low DO	(Andreasen and Nielsen, 2000; Nielsen *et al.*, 2002)

2.5.1.4 Membrane bio-reactors (MBRs)

MBRs are similar to CAS in that they use activated sludge to treat the wastewater, with the difference of using membrane filtration for keeping the activated sludge in the biological tanks rather than a clarification step. The impact of septicity on the biological processes in MBRs is expected to be the same as in CAS, leading to nitrification inhibition and filamentous proliferation. Although septicity also results in activated sludge bulking in MBRs, the membrane filtration step is able to retain the biomass (Brindle and Stephenson, 1996). The main issue with filamentous proliferation in MBRs is the impact it has on membrane fouling (Meng and Yang, 2007). Several studies concluded that the proliferation of filamentous bacteria increased the fouling rate of the membrane filtration, increasing the transmembrane pressure and resulting in an increased cleaning frequency (Meng *et al.*, 2006). However, Wang et al. (2010) observed the opposite effect when operating two identical MBR pilot plants, one with low filamentous levels and another one with high filamentous abundance. Some authors have found that a small amount of filamentous abundance reduces the membrane fouling, due to the formation of a loose layer on the membrane wall, and an excessive filamentous abundance increased membrane fouling, due to the formation of a non-porous layer on the membrane wall (Meng *et al.*, 2006; Gkotsis *et al.*, 2020). There is still discrepancy on the effect of filamentous bulking on membrane fouling and some authors suggest the specific filamentous species and their length could play a big part in the different effects observed (Wang *et al.*, 2010).

2.5.2 Biofilm processes

Biofilm processes such as trickling filters (TF) and rotating biological contactors (RBC) are used widely in the UK and in small to medium size WWTPs, due to their low operational costs and high robustness. Other biofilm based technologies such as moving bed biofilm reactors (MBBR) are also popular to retrofit and increase capacity of existing ASP and more recently the aerobic granular sludge (AGS) process has received much attention and implementation worldwide (Pronk *et al.*, 2015). Most biofilm processes can be configured to remove some or all the nutrients in wastewater, depending on the aerobic/anaerobic stages involved (van Dijk, Pronk and van Loosdrecht, 2018). Biofilm processes have several advantages, such as higher biomass concentrations, resilience to environmental factors and low sludge production (Tchobanoglous *et al.*, 2014; Xue *et al.*, 2017; Winkler *et al.*, 2018). As the bacteria population in suspended growth systems and biofilm systems are quite similar and their functions are alike, a similar process impact when treating septic wastewater can be expected. Nevertheless, the biofilm structure and diffusion limitations enable a certain degree of protection to the bacteria, which can result in lower inhibition by sulphide (Miao *et al.*, 2018). Elevated sulphide levels, however, can still hinder process performance, for example, an increase in sulphide concentration from 2 mg/L to 5.4 mg/L reduced the nitrification efficiency by 10% in a full-scale TF (Bentzen *et al.* 1995). In a pilot plant MBBR, Æsøy et al. (1998) observed both the effect of competition between heterotrophs and nitrifiers at increasing COD concentrations from 100 mg/L to 170 mg/L and the inhibition of nitrifying activity due to sulphide inhibition. They concluded that a concentration of sulphide higher than 0.5 mg S/L reduced nitrification capacity by 30-40%.

High concentrations of VFAs and sulphide due to septicity are expected to have similar impacts in EBPR and in AGS processes, promoting the growth of filamentous bacteria. *Thiothrix* spp. was observed to grow in AGS systems fed with sulphide at 100 mg/L (Su *et al.*, 2012) and thiosulphate at 80 mg S/L (de Graaff, van Loosdrecht and Pronk, 2020). However, settleability problems were only observed in the study by Su et al. (2012), when high concentrations of 300 mg/L sulphide were fed to the system. de Graaff et al. (2020) observed an increase in the relative abundance of *Thiothrix* spp. from 0% to 51% with increasing thiosulphate concentrations from 0 mg S/L to 80 mg S/L, but even at an abundance of 51% the SVI of the granules was maintained at 13.3 mL/g. This was due to filamentous bacteria growth inside the granules rather than to deterioration of settling properties and formation of bulking sludge (Martins *et al.*, 2004).

Xue et al. (2017) observed a different impact, reporting an increase in phosphorus removal from 12% to 87% when the sulphate concentration of a synthetic saline wastewater was increased from 0 mg/L to 200 mg/L. When the sulphate concentration was increased further to 500 mg/L and 1000 mg/L, phosphorus removal decreased to 59% and 1.3%, respectively. The initial phosphorus removal increase was attributed to sulphide production during the anaerobic stage, which led to a higher phosphorus release by PAOs due to detoxification and to an increase in VFA uptake (Xue *et al.*, 2017). Nitrification inhibition by sulphide in AGS systems was not observed by Su et al. (2012) at sulphide concentrations in the range of 50-300 mg/L, whereas Xue et al. (2017) measured a drop in the nitrification efficiency from 53 % to 31 % at sulphide concentrations of 0 and 1000 mg/L, respectively.

2.6 Thickening and dewatering

Sludge is generated in WWTPs in primary settlers (primary sludge) and the biological processes, most importantly from suspended growth processes (surplus activated sludge). While primary sludge (PS) mainly contains organic particulates that come directly from domestic or industrial effluents, surplus activated sludge (SAS) is mainly composed of bacterial flocs, which contain microorganisms, water and extracellular polymeric substances (EPS) (Li and Ganczarczyk, 1990). PS is easier to dewater than SAS and hence research is mainly focused on the properties of SAS and how dewaterability changes with different conditions, like anaerobic storage or shear stress (Rasmussen *et al.*, 1994; Nielsen, Frolund and Keiding, 1996; Wilen, Keiding and Nielsen, 2000).

The location at which septicity develops dictates the impact it has on the dewatering processes. Septic influents or septic primary clarifiers (Figure 2-3, points 1 and 2) are expected to have an impact on the dewaterability of the sludge generated, both PS and SAS, but there is reported literature investigating those impacts. Septic PS would be expected to have smaller particle sizes due to the hydrolysis of particulate material, which would decrease the dewaterability rate (Novak *et al.*, 1988; Bruus, Nielsen and Keiding, 1992). For septic SAS, a higher abundance of filamentous bacteria within the sludge is expected, which was found to correlate with higher specific resistance to drainage, which is comparable to dewaterability rate (Dominiak, M. Christensen, *et al.*, 2011; Dominiak, M. L. Christensen, *et al.*, 2011). Research that links septic influents or septic primary clarifiers with dewatering efficiency is needed.

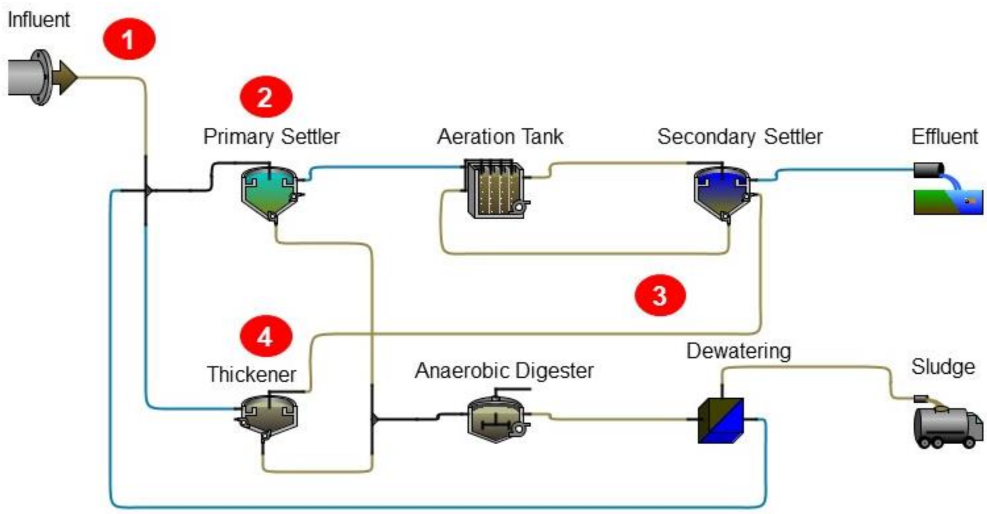

Figure 2-3 Locations at which septicity is potentially developed

Septicity development during sludge storage and thickening was also found to have an impact on sludge dewaterability (Figure 2-3, points 3 and 4). Storage prior to dewatering is frequent and it mainly depends on the dewatering cycles (Rasmussen *et al.*, 1994). Stored sludge lacks aeration and dissolved oxygen is depleted in a short time , leading to anaerobic conditions that result in SAS deflocculation and disintegration (Bruus, Nielsen and Keiding, 1992; Rasmussen *et al.*, 1994; Nielsen, Frolund and Keiding, 1996; Nielsen and Keiding, 1998; Wilen *et al.*, 2000; Wilen, Keiding and Nielsen, 2000). During the anaerobic state, hydrolysis, fermentation, ferric to ferrous reduction and sulphate reduction were found to be the main biochemical processes (Rasmussen *et al.*, 1994). Comprehensive studies of different factors that influenced the dewatering rate (measured as the specific resistance to filtration (SRF)) during anaerobic conditions were performed in the 90s. Nielsen et al. (1996) reported that during anaerobic storage EPS production was inhibited and the existing EPS were hydrolysed and solubilised, which was concluded would lead to a decrease in dewatering rate. Furthermore, it was observed that during 12 days of anaerobic storage of SAS the bulk liquid conductivity increased twofold, which indicated the displacement of divalent cations from the floc matrix, weakening the floc structure (Bruus, Nielsen and Keiding, 1992; Rasmussen *et al.*, 1994; Mangrum and Jenkins, 2019). The reduction of ferric to ferrous either biologically or chemically in the presence of sulphide was also found to cause SAS disintegration and increased the SRF by 50 % and, if soluble sulphide was present, the ferrous was precipitated as FeS and the SRF increased by a further 30% (Nielsen and Keiding, 1998; Wilen *et al.*, 2000; Kobayashi, Xu and Chiku, 2015). Einarsen et al. (2000) documented a 20% increase in the dewatering rate when septic conditions were avoided at a sludge thickener. Sludge was dosed with calcium nitrate, which induced anoxic conditions, thus avoiding sludge disintegration (Novak *et al.*, 1998; Wilen *et al.*, 2000; Wilen, Keiding and Nielsen, 2000).

Although anaerobic storage has been reported to reduce dewatering rate significantly, it has also been found to reduce some of the bound water content (Liao *et al.*, 2000). In the last decade, research on dewatering has focused on the development of methods to improve sludge dewaterability, both in terms of rate and extent. Methods that targeted lysis of microbial cells, such as enzyme addition (Chen *et al.*, 2015) or chemical acidification (Lu *et al.*, 2017), found that cell lysis increased the soluble fraction of EPS and hindered dewatering rate while simultaneously reducing the amount of bound water and increasing the dewatering extent. Anaerobic storage of sludge is expected to have a similar impact on microbial lysis and consequently on dewatering, although to the best of the authors knowledge there is no proof of it in literature. Chen et al. (2015) observed re-flocculation and removal of macromolecule biopolymers when dosing coagulants such as ferric chloride and polyaluminium chloride after enzymatic treatment, improving the dewatering rate to extents that match that of untreated sludge and improving the final dry solids content of the sludge, which reduced its moisture content to 82% compared to 86% of the untreated material. A similar behaviour would be expected from anaerobically stored sludge, but it should be demonstrated.

2.7 Anaerobic digestion

The operation of anaerobic digesters (ADs) is quite sensitive to changes in the input sludge and has to be carefully monitored to achieve optimised methane yields (Turovskiy and Mathai, 2006). Similar to the thickening and dewatering steps, the location at which septicity develops has different impacts in the AD process, with the main locations where septicity development will impact AD being the WWTP influent, the primary clarifier and the sludge storage prior to digestion (Figure 2-3).

Septic influents or septic primary clarifiers (Figure 2-3, points 1 and 2) are expected to change the volumes of primary sludge (PS) and activated sludge produced as septicity development (Figure 2-1) results in solubilisation of particulate COD, leading to a lower PS quantity and higher SAS generation. Overall, the total sludge quantity should not change significantly; however, PS is well known to have higher biodegradability than activated sludge, thus, leading to a reduction in biogas production (Carrere *et al.*, 2010).

Anaerobic sludge storage prior to AD stabilisation (Figure 2-3, point 3 and 4) is quite common (Rasmussen *et al.*, 1994). During storage, particulate COD and

EPS are broken down by hydrolysis and fermentation, leading to an accumulation of VFAs (Nielsen, Frolund and Keiding, 1996; Yuan, Sparling and Oleszkiewicz, 2011). Simultaneously, sulphate is reduced to sulphide by the activity of SRB (Wilen *et al.*, 2000). The processes that take place during anaerobic storage of sludge are similar to those occurring in ADs except for the final methanogenesis stage, which is not expected to be significant during anaerobic storage due to the lack of inoculum and low growth rates of methanogenic archaea (Tchobanoglous *et al.*, 2014). Anaerobic storage of sludge can hence aid the AD process, as hydrolysis is known to be the rate limiting step (Vavilin *et al.*, 2008; Batstone, Tait and Starrenburg, 2009), decreasing the hydraulic retention time (HRT) needed for the same performance in the AD (Carrere *et al.*, 2010). Unsurprisingly, anaerobic storage prior to AD has been previously intensified as a process to improve sludge biodegradability, known as thermophilic or mesophilic anaerobic fermentation (Ge, Jensen and Batstone, 2010), and even for the production of VFAs for EBPR processes (Chanona *et al.*, 2006; Yuan, Sparling and Oleszkiewicz, 2011). Feeding of high VFA sludge can, however, inhibit the methanogens (Wang *et al.*, 2009) if loading is not sufficiently controlled and causes a pH drop in the system below the optimum pH range of 6.8-7.4 (Turovskiy and Mathai, 2006; Ye *et al.*, 2013). Therefore, septic sludge feeding to AD should be performed in a controlled manner.

Similar to other microorganisms, methanogens were found to be inhibited by sulphide (O’Flaherty *et al.*, 1998; Lippens and De Vrieze, 2019), although the concentrations resulting in process inhibition were measured at 486 mg S/L (Khanal and Huang, 2003), 800 mg S/L (Celis-Garcia *et al.*, 2004) and 804 mg S/L (Khanal and Huang, 2006), which are not expected to be present at WWTPs treating domestic wastewater, measured up to 60 mg S/L (Matos *et al.*, 2018). The problem of sulphide inhibition to methanogens is exclusive to certain situations where there is seawater intrusion or when industrial trade effluents or wastes containing sulphur compounds are treated (Hulshoff Pol *et al.*, 1998).

2.8 Discussion

Overall, septicity results in significant changes in the wastewater characteristics leading to impacts at WWTPs that cannot be overlooked, such as sludge rising in primary settlers and nitrification inhibition (Table 2-4). Although individual impacts of septicity in WWTP processes have been investigated to a certain degree, these are usually not identified and managed accordingly. Some of the interfaces between septic wastewater and efficiency in WWTP processes have been defined, however, there are still controversies and lack of evidence to enable a full understanding of the impacts. Most importantly, the extent of septicity and the configuration of the specific WWTP dictates the consequences of septicity in process efficiency and effluent quality.

Table 2-4 Main impacts of septicity on wastewater treatment processes

Treatment process	Main impacts
Chemical phosphorus removal	Increase in coagulant demand
Primary settling	Rising sludge Worse settling characteristics Lower efficiency
Biological processes	Activated sludge bulking Inhibition of microbial processes Increased loading of pollutants Avoid or lower the need for external carbon (specific to BNR)
Thickening and dewatering	Reduced dewaterability rate Lower moisture on the dewatered cake
Anaerobic digestion	Lower biogas production (septicity in the influent) Higher biogas production (septicity in sludge storage)

To illustrate the different impacts that septicity can trigger depending on treatment flowsheet, three WWTPs with different configurations were assessed for their effluent quality when the influent was septic and not septic. The septic wastewater had the pollutant concentrations adjusted according to Table 2-1. All configurations comprised of preliminary, primary and secondary treatment and the influent flow was assumed as 1000 m^3/day. WWTP 1 included ferric dosing prior to the primary settling tank and a fully nitrifying ASP, with a discharge consent of 10 mg N/L of ammonia and 1 mg P/L of phosphorus (Defra, 2002). On WWTP 2 the secondary treatment was a nitrifying ASP with a discharge consent of 10 mg N/L of ammonia and no P removal (Table 2-5). In WWTP 3 the secondary treatment was a BNR process with a discharge permit of 10 mg N/L of ammonia and 1 mg P/L of phosphorus (Table 2-5). The TSS and soluble COD discharge consents where the same for all three configurations at values of 35 mg/L and 40 mg/L, respectively, according to Defra (2002). Mass balances for TSS, soluble COD, ammonia, orthophosphate and sulphide were completed for each one of the configurations. The process removals used to calculate the mass balances are shown in .

Table 2-5 Influent characteristics and effluent requirements of WWTP 1, 2 and 3

		Influent composition (after preliminary treatment)		Effluent requirements		
Parameters	**Units**	**Fresh Influent**	**Septic Influent**	**WWTP 1**	**WWTP 2**	**WWTP 3**
TSS	mg/L	450	400	35	35	35
sCOD	mg/L	200	240	40	40	40
Ammonia	mg N/L	40	47	10	10	10
Ortho-P	mg P/L	5	6	1	-	1
Sulphide	mg S/L	0	8	-	-	-

Table 2-6 Percent removal of wastewater pollutants used for the calculation of the mass balances for WWTP 1, 2 and 3

		Primary settler[1] (%)	Primary settler + Iron dosing[2] (%)	Nitrifying ASP[1] (%)	EBPR ASP[1] (%)
TSS	Steady state	60	75	90	90
	Under septic conditions	60	75	70[4]	70[4]
sCOD	Steady state	0	30	85	85
	Under septic conditions	0	30	85	85
Ammonia	Steady state	0	0	90	90
	Under septic conditions	0	0	70[5]	70[5]
Ortho-P	Steady state	0	85	0	90
	Under septic conditions	0	50[3]	50[3]	95
Sulphide	Steady state	0	100	100	100
	Under septic conditions	0	100	100	100

[1] (Tchobanoglous *et al.*, 2014); [2] (Shewa *et al.*, 2020); [3] Lower availability due to FeS precipitation and subsequent sulphide oxidation and re-precipitation with orthophosphate according to Gutierrez *et al.* (2010).[4] Increase in SVI due to filamentous proliferation; [5] Nitrification inhibition by sulphide.

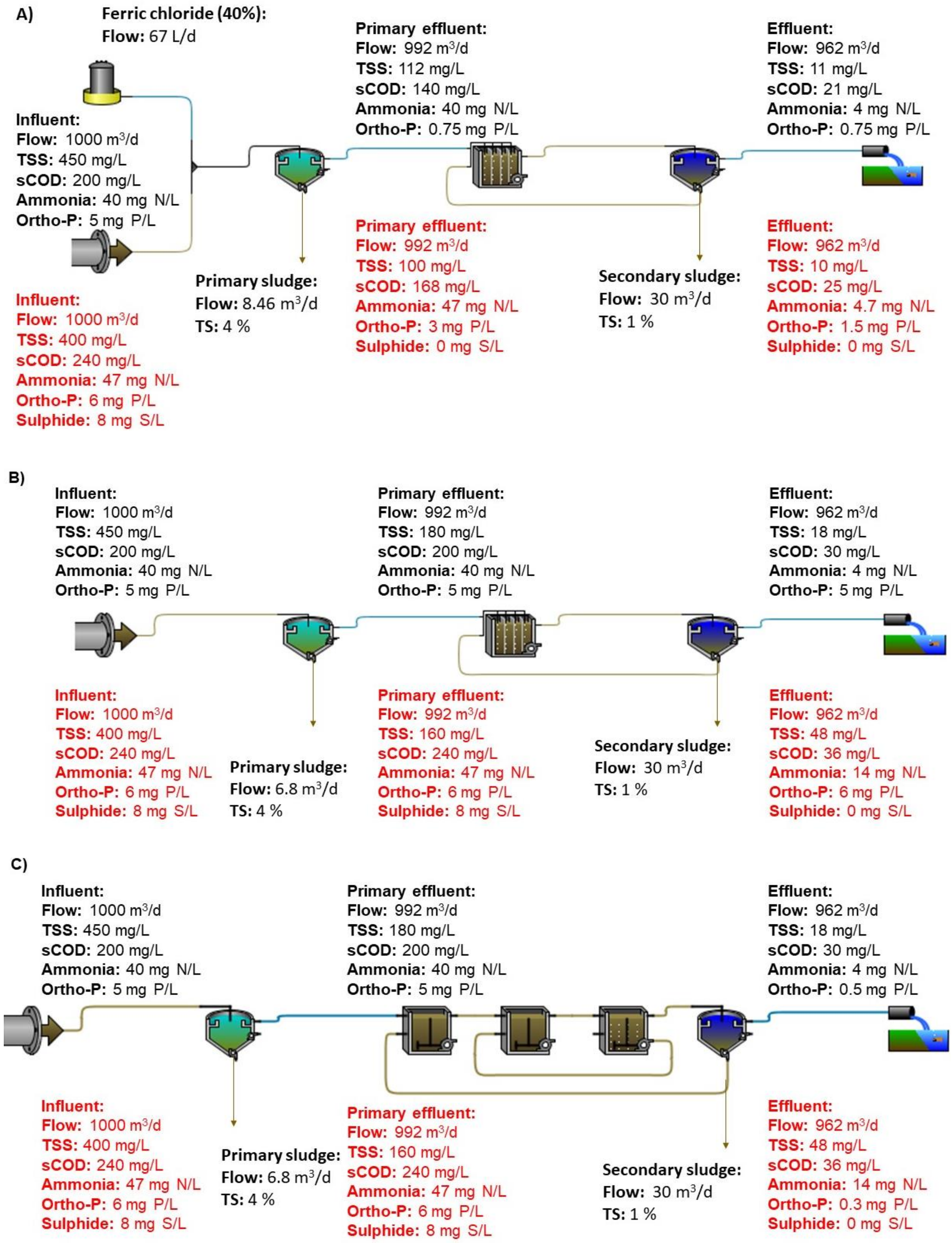

Figure 2-4 Mass balance of WWTP 1 (A), WWTP 2 (B) and WWTP 3 (C) with not septic influent (values in black) and with septic influent (values in red)

A septic influent in WWTP 1 led to an effluent orthophosphate concentration of 1.5 mg P/L, which breached the discharge standard of 1 mg P/L (Figure 2-4 A) due to the sulphide concentration of 8 mg S/L in the influent and competition between ortho-phosphate and sulphide for the available iron, according to Equations (2-1) and (2-2). Interestingly, septicity did not impact performance of the ASP (Table 2-4), because sulphide was precipitated during the chemical phosphorus removal stage. Septicity in this case would result in a need to increase iron dosage in order to comply with the phosphorus discharge regulation, leading to an operational cost increase.

At WWTP 2, the sulphide present in the septic influent reached the ASP at a concentration of 8 mg S/L, inhibiting nitrification (Table 2-4), reducing the ammonia removal efficiency by 20% (Figure 2-4 B) and resulting in 14 mg NH_4-N/L in the effluent, which breached the 10 mg N/L ammonia consent. Furthermore, the combination of sulphide and VFAs in the ASP influent led to an increased SVI, due to the proliferation of filamentous bacteria (Table 2-4), and resulted in an increased TSS concentration of 48 mg/L, also breaching the 35 mg/L TSS consent. To avoid inhibition problems, the sulphide present in the septic wastewater should have been removed, for example by means of natural or forced aeration prior to the ASP (Gutierrez *et al.*, 2008). Depending on the method used to remove the sulphide prior to the ASP, it would result in an increased capital or operational cost.

At WWTP 3, the higher concentrations of VFAs of 80 mg/L in the septic influent reduced the effluent orthophosphate concentration to 0.3 mg P/L (Figure 2-4 C). However, 8 mg S/L of sulphide reached the ASP and inhibited the nitrifying population (Table 2-4) resulting in an ammonia concentration of 14 mg N/L in the effluent, which breached the 10 mg N/L ammonia consent. The presence of sulphide also resulted in the proliferation of sulphide oxidising bacteria, increasing the SVI and leading to an effluent TSS concentration of 48 mg/L, which breached the 35 mg /L TSS consent. Similar to WWTP 2, the removal method for sulphide would incur different cost increases; however, the external carbon dosage for the EBPR process is expected to be reduced.

2.9 Research gaps

The presence of sulphide at concentrations of 0.5-17 mg S/L in WWTPs (Table 2-1) was identified as problematic in the treatment of septic wastewater. Existing literature on the impact of sulphide on the EBPR process and nitrification is controversial, with a general agreement that sulphide concentrations higher than 50 mg S/L result inhibitory for PAO populations, but without a clear understanding of the impact at lower concentrations. Rubio-Rincon et al. (2017b) observed a reduction in the VFA uptake rate from 510 mg COD/(g VSS·h) to 430 mg COD/(g VSS·h) when the sulphide concentration increased from 0 mg S/L to 42 mg S/L, whereas Xue et al. (2017) reported an increase in the VFA uptake rate when sulphide increased from 0 mg S/L to 65 mg S/L. The increase in EBPR efficiency observed in Xue et al. (2017) was proved to result from the increased orthophosphate release in the anaerobic stage to generate energy for sulphide detoxification (transporting the sulphide from inside the cell to outside). Sulphide detoxification generated a higher proton driving force for VFA uptake in the anaerobic stage and therefore increased the orthophosphate uptake in the aerobic stage.

Regarding the impact of sulphide on nitrification, batch test studies have clearly proved that sulphide is inhibitory for nitrifying populations (Table 2-2). However, there are significant differences in the inhibition constants by up to two orders of magnitude. The study by Sekine et al. (2020) suggest that nitrifying populations are able to acclimatise to sulphide concentrations as high as 100 mg S/L. More research is needed on the impact of sulphide on EBPR, the inhibition of nitrification and subsequent acclimation, both with septic wastewater from impacted WWTPs and using activated sludge collected from full-scale activated sludge plants. Furthermore, the difference on nitrification inhibition by sulphide in suspended flocs and biofilms should be assessed, as there is a lack of information on literature.

Particle size is the governing parameter when removing solids by gravity. Anaerobic digestion was found to change the particle size distribution (PSD) of TSS (Elmitwalli *et al.*, 2001). Similarly, septicity development in sewers is expected to result in changes on the particle sizes of TSS, which would impact efficiency for their removal efficiency in the primary settler. However, there is no research on the transformation of TSS particle size during septicity development.

de Graaff et al. (2020) demonstrated that the AGS process is more resilient to the proliferation of sulphur accumulating filamentous bacteria than the ASP. However, there is opposing evidence that filamentous bulking is also a problem in AGS processes (Su *et al.*, 2012). Further research is required to define the optimal reactor operation at which filamentous bacteria do not cause bulking in AGS systems.

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3 DEVELOPMENT OF A SEPTICITY SCALE FOR RAW WASTEWATER

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Abstract

Hydrogen sulphide has been historically the main parameter of concern related to wastewater septicity. However, when wastewater septicity develops in sewerage systems, many other wastewater parameters are altered. To address septicity and promote septicity management options, capturing the different wastewater parameters that are altered during septicity is critical. To allow that, a septicity measure was developed in this study. The septicity measure is a combined measure derived from the measurement of key indicators of septicity. Potential key indicators were selected based on literature and were further refined with the data collected from wastewater incubation tests. Finally, 5 key indicators that form the septicity scale were derived, which are, sulphide, oxidation reduction potential (ORP), pH, soluble COD and ammonia. The importance of each indicator was defined by a consultation of 14 international experts on septicity and was of 39, 26, 18, 11 and 6 for sulphide, ORP, pH, soluble COD and ammonia, respectively.

The septicity measure was developed with two objectives in mind. The first objective was for the septicity scale to be used by researchers worldwide to provide a framework to compare different research in the field of wastewater septicity. The second use of the septicity scale was to develop a more holistic septicity warning and action tool for water utilities.

Keywords: Sulphide, Sewer processes, Anaerobic processes, Combined indicator

3.1 Introduction

Septicity is the condition of the wastewater after undergoing unintentional anaerobic transformations during wastewater conveyance or treatment (Boon, 1995; Hvitved-Jacobsen, Vollertsen and Nielsen, 2013). The anaerobic transformations consist of four steps, namely, hydrolysis, fermentation, sulphate reduction and methanogenesis. During those four steps, the biochemical characteristics of the wastewater change.

Many parameters have been related to septic conditions, such as redox potential (ORP), COD, sulphide, dissolved oxygen (DO) and volatile fatty acids (VFAs), but there are differing opinions in literature about the importance of each factor on septicity.

Historically, most of the attention has been put on sulphide. Sulphide is not present in urban wastewater but is generated during the sulphate reduction step, in which sulphate in the wastewater is reduced to sulphide by the metabolism of sulphate reducing bacteria (SRB) (Boon, Vincent and Boon, 1998). Sulphide dissociates into hydrogen sulphide (H_2S), bisulphide (HS^-) and sulphide (S^{-2}) in water. At common wastewater pHs (6-9) sulphide concentration is negligible and hydrogen sulphide and bisulphide are in equilibrium with a pKa of 7 (Sharma *et al.*, 2014). Hydrogen sulphide is toxic to humans at concentrations as low as 50 ppm and can cause death within minutes of exposure at 700 ppm (Park *et al.*, 2014). Furthermore, is the primary cause of microbial induced concrete corrosion (MICC), mainly in sewer crowns and manholes (Nielsen, Hvitved-Jacobsen and Vollertsen, 2012). The financial burden of H_2S related corrosion in sewer networks has been estimated at over €450 million per year in Germany and £85 million in the UK (Grengg *et al.*, 2018) and €5 million per year in Flanders (Belgium) (Vincke, 2008).

However, septic wastewater can also lead to problems at downstream wastewater treatment plants (WWTPs) that are not related to sulphide. Due to the biochemical transformations during the anaerobic processes, the soluble wastewater pollutants, like the soluble COD and ammonia, increase in their concentration, making the primary treatment less effective and thus, increasing the load of the secondary treatment (Ashley *et al.*, 2002). Furthermore, the compounds that are generated and accumulated in the wastewater like sulphide and volatile fatty acids (VFAs) can cause toxic or inhibitory effects on the microorganisms that treat the wastewater and lead to activated sludge bulking (Jenkins, Richard and Daigger, 2003).

To understand septicity and its impacts, it is clear that only measuring sulphide is not enough to see the complete picture of septicity and several wastewater parameters have to be measured. Those parameters can indicate if a wastewater is septic or not, but if they are used in combination, they can show a much more detailed and accurate picture of the septicity of that wastewater. To combine different parameters into a single one, different statistical methods can be used. Developing a regression model is a common method to do so, as it creates a predictive model on which measuring the model parameters would give a septicity measure for a given wastewater. However, to develop the regression model, the septicity level would have to be measured for the model training observations, and as the septicity level cannot be physically measured, it is not possible to use a regression model. Another method is developing a combined indicator based on the wastewater parameters that influence septicity. The combined indicator method is commonly used in socioeconomic studies, where countries or other entities are to be compared between them, for example, the Happy Planet Index (Abdallah *et al.*, 2012) or the Research Excellence Index (Vertesy, 2018). A combined indicator has also been successfully developed for benchmarking WWTP energy efficiency (Longo *et al.*, 2019).

Therefore, the objective of this study is to develop a septicity index, based on commonly measured wastewater parameters using the combined indicator method. This index can be used as a risk factor for the impacts septicity has on the overall wastewater treatment scheme.

3.2 Materials and Methods

3.2.1 Data gathering

Fresh raw wastewater samples were collected from five wastewater treatment plants (WWTPs) with different characteristics and quickly transported in a fridge to the laboratory. Wastewater samples were anaerobically incubated in 2L airtight reactors with a coupled GoDirect ORP probe (Vernier, US) for 4 days (Figure 3-1). Before starting the incubation, samples were sparged with nitrogen gas to displace the dissolved oxygen until a DO <0.05 mg/L was achieved, measured with an LDO101 probe (Hach, UK). Then, the bottles were closed using rubber stoppers with two tubes (a long one for liquid sampling and a short one for adding nitrogen) that were closed with pegs (Figure 3-1). The liquid was mixed at 200 rpm throughout the test using a magnetic stirrer and a magnetic plate. Liquid samples (25 ml) were collected twice a day and the same volume of nitrogen gas was injected to avoid vacuum in the reactor. 5 mL of liquid sample were immediately filtered through a 0.45 μm pore size syringe filter (Millipore, UK) and analysed for dissolved sulphide using the Merck sulphide test (114779). pH of the sample was measured using a PHC101 probe (Hach, UK). The rest of the liquid sample was filtered through a 0.7 μm pore size filter (Whatmann, UK) and analysed for soluble COD (sCOD), ammonia and sulphate using Standard Methods (APHA, 2005). Acetic and propionic acid were measured using an HPLC (Shimadzu VP) equipped with an AJ0-4287 SecurityGuard cartridge (Phenomenex, UK), a Rezex ROA-Organic Acid H^+ column (Phenomenex, UK) 300 x 7.8 mm maintained at 50 °C, and a UV-Vis detector at 208 nm. The mobile phase was 0.5 mM sulphuric acid at a flow rate of 1 ml/min.

Figure 3-1 Experimental set-up of the anaerobic incubation tests

3.2.2 Development of the septicity index

The septicity index is a combined indicator for a particular wastewater sample, which captures the influence of key indicators of septicity commonly measured on wastewater. Several steps have to be followed to calculate the septicity index, namely, selection of the key indicators, normalisation, weighting, aggregation and benchmarking. The procedure for the septicity index calculation is shown in Figure 3-2 and the steps are explained in detail in the next sections.

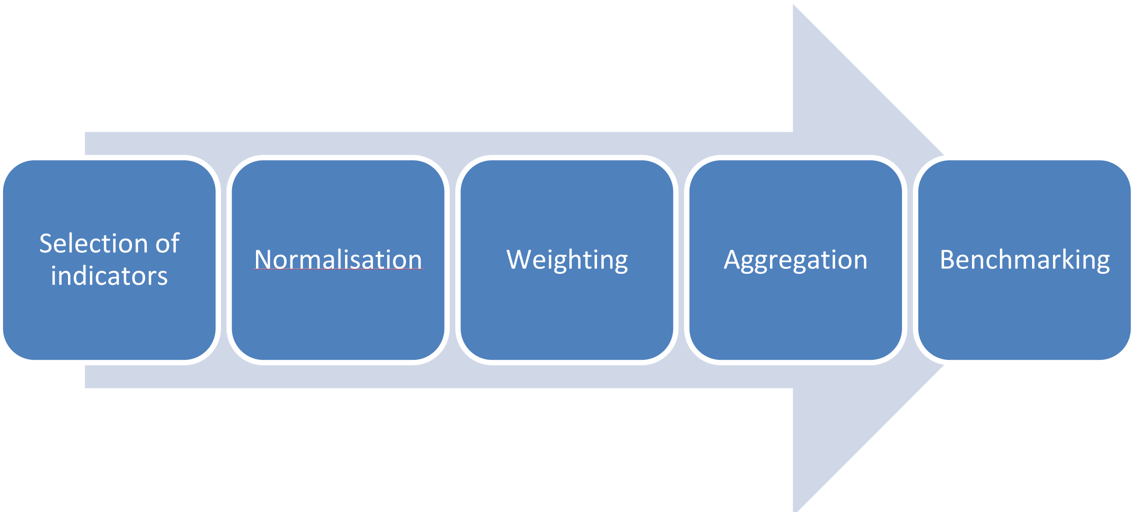

Figure 3-2 Workflow for the Septicity Index calculation

3.2.2.1 Selection of key indicators

The selection of the indicators to be included in the septicity index calculation was a key step, as it would influence the applicability and robustness of the septicity index. Eight potential indicators (pH, ORP, soluble COD, ammonia, sulphide, sulphate, acetic acid and propionic acid) were selected and monitored during the anaerobic incubation experiments, based on a literature review of the septicity indicators. To avoid duplicating the impact of some indicators, a correlation analysis was performed and some of the potential indicators were discarded due to their high correlation with others.

3.2.2.2 Normalisation

The key indicators are expressed in a variety of units and have different orders of magnitude, which would cause a problem when aggregating them. To bring them to a common basis, the min-max normalisation technique is used (Equation (3-1)), which converts all indicators to values between 0 to 1.

$$I_{norm} = \frac{I - \min{(I)}}{\max(I) - \min{(I)}} \tag{3-1}$$

3.2.2.3 Weighting

The weighting of each indicator emphasizes its contribution towards septicity identification. The weight selection was based on a questionnaire (8Appendix A) that was responded to by 14 subject matter experts in septicity from the UK, Denmark and Australia, most of them with over 10 years of experience working on septicity related topics. The questionnaire asked to assign points to different key indicators of septicity based on their importance in determining a wastewater sample's septicity. The results from all the completed surveys were analysed to calculate the weighting of each key indicator.

3.2.2.4 Aggregation

The aggregation consisted of the combination of the weighted indicators onto the Combined Septicity Index (CSI). The CSI was calculated through a weighted sum (Equation (3-2)).

$$CSI = 1 - \sum_{i=1}^{n} w_i I_{norm,i} \quad \textbf{(3-2)}$$

3.2.2.5 Benchmarking

The calculated CSI values for all samples were fitted to a Gumbel distribution, to allow benchmarking of future wastewater samples against the incubated samples. The Septicity Index (SI) is obtained by applying the following:

- If $CSI/p_{25} \leq 1$ use $SI = CSI/p_{25}$
- If $1 \leq CSI/p_{50}$ use $SI = 1 + CSI/p_{50}$
- For other cases use $SI = 1 + [(CSI - p_{25}) / (p_{50} - p_{25})]$

Finally, the septicity level is assigned based on the following ranking:

- Level 0 (no septicity) if $3 \leq SI$
- Level 1 if $2.25 \leq SI < 3$
- Level 2 if $1.5 \leq SI < 2.25$
- Level 3 if $0.75 \leq SI < 1.5$
- Level 4 (highly septic) if $SI < 0.75$

3.3 Results and Discussion

The potential indicators measured followed a similar trend during the incubation periods for all wastewater samples, regardless of their strength or origin. Sulphate, pH and ORP were reduced whereas soluble COD (sCOD), sulphide and acetic acid were generated (Figure 3-3). Similar behaviour of the wastewater parameters during anaerobic transport have been previously reported by several authors (Raunkjaer, Hvitved-Jacobsen and Nielsen, 1995; AEsoy *et al.*, 1997; Bachmann, Saul and Edyvean, 2007; Rudelle *et al.*, 2016).

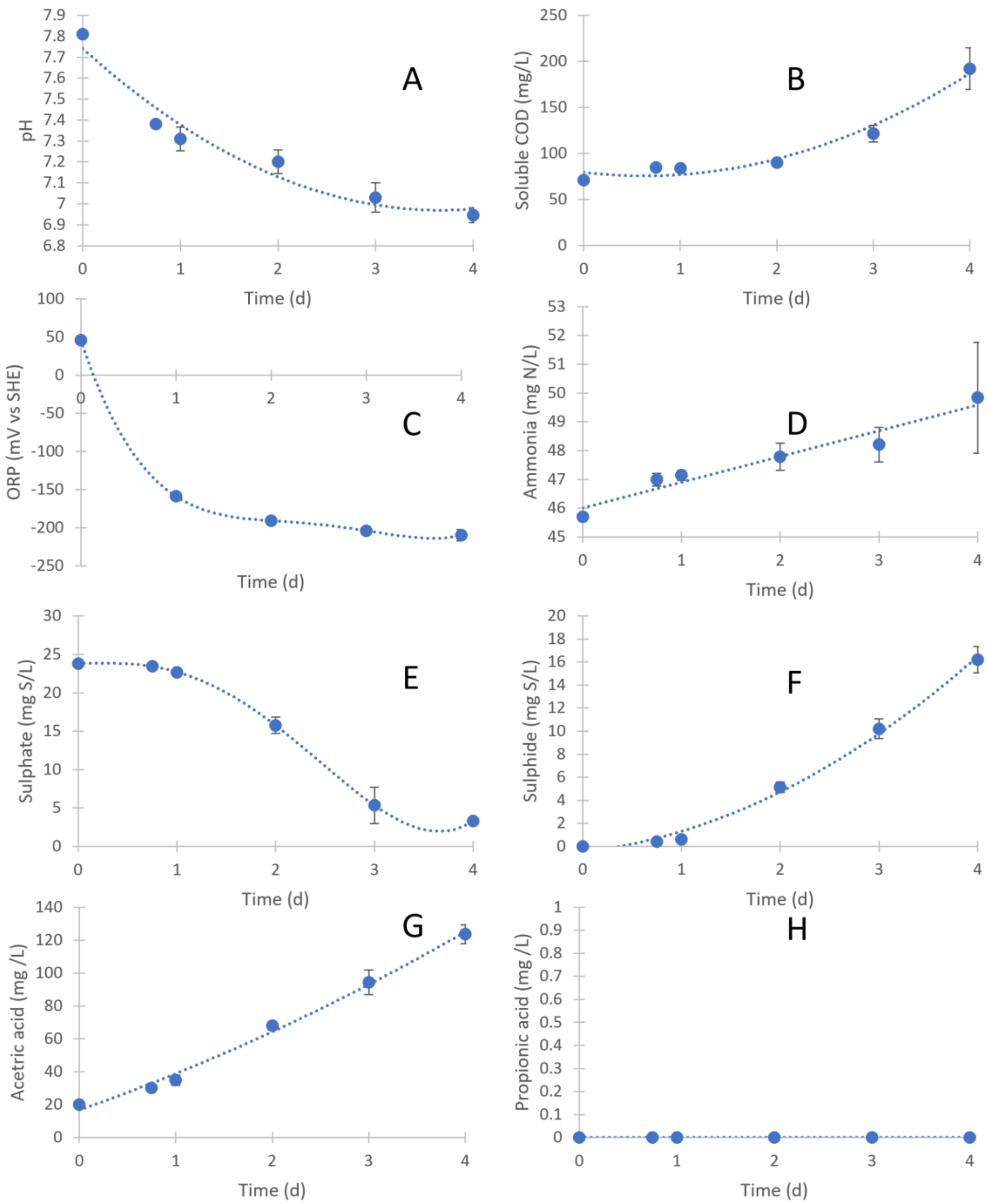

Figure 3-3 Development of key indicators during the incubation period of the raw wastewater samples. A: pH; B: Soluble COD; C: ORP; D: Ammonia; E: Sulphate; F: Sulphide; G: Acetic acid; H: Propionic acid

To form the dataset from which the septicity scale was going to be built, only one incubation period was selected for each sample. The selection was made by comparing the range of the septicity indicators each incubation period covered and what had been reported in literature as septic wastewater (Table 3-1). The incubation period selected as most representative of septic wastewater was the second day incubation period.

Table 3-1 Range of septicity indicator parameters during different incubation periods and literature ranges (Chapter 2) of the indicator parameters for septic wastewater

	Incubation Period				
Parameters	**Day 1**	**Day 2**	**Day 3**	**Day 4**	**Literature Values**
pH	6.97 – 7.72	6.93 – 7.58	6.75 – 7.46	6.61 – 7.36	6 - 8
ORP (mV vs SHE)	-109 – -243	-146 – -254	-169 – -275	-178 – -260	-180 – -310
Soluble COD (mg/L)	25 - 227	44 - 294	53 – 383	72 – 471	60 - 400
Ammonia (mg N/L)	21 – 67	24 - 68	24 – 69	24 – 69	25 - 80
Sulphide (mg S/L)	0 – 4.65	0 – 9.55	1.44 – 18.4	3.37 – 24.6	0.5 - 17
Acetic acid (mg/L)	0 - 84	0 - 115	31 - 178	45 - 168	40 -120

From the 8 potential indicators measured during the incubation periods, propionic acid was first discarded, as it was only detected in 30% of the septic samples. From a biochemical point of view, sulphide and sulphate were deemed to be representative of the same aspect of septicity, as sulphate is converted to sulphide during the sulphate reduction process (Sharma *et al.*, 2008; Hauduc *et al.*, 2017). Therefore, the decision to remove sulphate as an indicator was taken. Finally, the correlations between the 6 remaining potential indicators were analysed with a correlation matrix (Table 3-2). Soluble COD and acetic acid showed a high correlation coefficient of 0.76, which was not surprising as acetic acid is part of the soluble COD measurement and it is the main product of the fermentation process that happens during anaerobic incubation (Rudelle *et al.*, 2011). Therefore, acetic acid was removed from the septicity indicator list, as wastewater is more commonly monitored for soluble COD than acetic acid and thus, the resulting septicity scale would be less applicable.

Table 3-2 Correlation matrix for the potential key indicators of septicity

	pH	ORP	sCOD	Ammonia	Sulphide	Acetic acid
pH	1	0.16	-0.38	0.02	-0.08	-0.52
ORP	0.16	1	0.00	0.42	-0.1	-0.18
sCOD	-0.38	0.00	1	0.29	0.07	0.76
Ammonia	0.02	0.42	0.29	1	-0.01	0.05
Sulphide	-0.08	-0.1	0.07	-0.01	1	0.13
Acetic acid	-0.52	-0.18	0.76	0.05	0.13	1

Next, the 5 final indicators (pH, ORP, ammonia, sulphide and soluble COD) were normalised using the min-max normalisation method described in Equation (3-1). To do so, the descriptive statistics for the dataset shown in Table 3-3 were used.

Table 3-3 Descriptive statistics of the key indicator dataset used for the septicity scale

Indicator	Units	Average	SDEV	Min	Max
pH	-	7.22	0.19	6.93	7.58
ORP	mV vs SHE	-212.07	24.17	-254.43	-146.44
sCOD	mg/L	123.13	63.31	43.50	294.00
Ammonia	mg N/L	45.54	10.92	23.75	67.87
Sulphide	mg S/L	5.27	2.76	0.00	9.55

The weighting questionnaire responses were analysed and the average of each indicator weight from the valid responses (assessed as the questionnaire being completely filled) was used as the weighting for the septicity scale formation. The selected weights were 39 (range: 1 – 90, stdev: 19.5), 26 (range: 10 – 54, stdev: 13.3), 18 (range: 0 – 40, stdev: 10.6), 11 (range: 0 – 20, stdev: 7.6) and 6 (range: 0 – 25, stdev: 8.4) for sulphide, ORP, pH, soluble COD and ammonia, respectively. Sulphide was selected as the most important parameter to identify septicity in raw wastewater by all but one respondent. It is not surprising, as sulphide has been historically the focus of septicity research and is the biggest hazard of all indicators (Park *et al.*, 2014).

The normalised indicator values were aggregated using Equation (3-2) to calculate the combined septicity index (CSI) for each sample, and the CSIs were fitted to a Gumbel distribution (Table 3-4) to be used as the benchmarking distribution for new samples.

Table 3-4 Parameters of the Gumbel distribution for the septicity scale

	Value
μ	0.3880
β	0.1452
25th percentile (p_{25})	0.3406
50th percentile (p_{50})	0.4412

The use of the septicity scale is quite straightforward. The first step is measuring the septicity indicators on the sample to be assessed. The second step is normalising the individual indicators using Equation (3-1) and Table 3-3 values. The third step is aggregating the normalised indicators to calculate the CSI of the sample. Finally, the Septicity Index (SI) of the sample can be calculated following the procedure described in Section 3.2.2.5, utilising the Table 3-4 Gumbel distribution values.

The septicity scale was developed with two uses in mind. The first objective was for the septicity scale to be used by researchers worldwide to provide a framework to compare different research in the field of wastewater septicity. Nowadays, only a few publications monitor and report all the septicity indicators identified in this study. Utilising the septicity scale would first increase the publications where the septicity indicators are monitored and second, it would offer a valuable tool to compare research done at different locations and with different wastewater characteristics.

The second use of the septicity scale was to develop a more holistic septicity warning and action tool for water utilities. The septicity indicators would be monitored (preferably with online monitors) and the septicity level would be assessed based on those indicators. Depending on the septicity level, some consequences could be derived for the downstream wastewater treatment plant

(WWTP) and actions to prevent those consequences would be applied. A framework for the septicity warning tool utilising the septicity scale is shown in Figure 3-4.

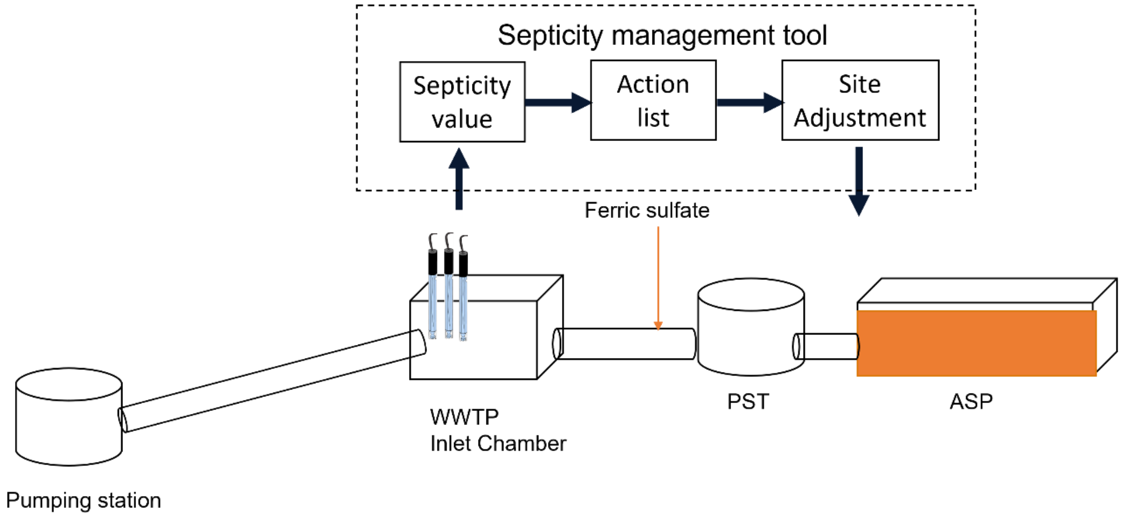

Figure 3-4 Septicity warning and action tool framework utilising the septicity scale

The main limitations of the septicity scale rely on the dataset used for its development. The datapoints were generated by incubating fresh raw wastewater samples in a laboratory setting, rather than measuring septic wastewater samples in-situ. This approach was taken as finding septic wastewater was unsuccessful. Although the dataset was compared to literature values on septic wastewater, it would be recommended for the scale to be validated with in-situ measurements of several septic wastewaters (preferably from different countries and climates).

3.4 Conclusions

This study describes the development of the septicity scale, specifically tailored to measure septicity in raw wastewater. Starting from a clear definition of the key indicators of septicity, the septicity scale offers a tool to researchers to compare their septicity related research formally. Furthermore, the septicity scale is of great use to water utilities for the management of their sewerage systems and wastewater treatment plants. By using commonly measured wastewater parameters such as pH, ORP, soluble COD, ammonia and sulphide, the development and change of the septicity level in their system can be monitored and acted accordingly. Alarms can be set-up at different septicity levels and reactive actions can be taken to reduce or prevent any consequences that could be derived from treating septic wastewater. Overall, the septicity scale provides the wastewater community with a tool to further improve septicity management and reduce the cost of remediation of septicity related problems.

3.5 Acknowledgements

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4 IMPACTS OF SEPTICITY ON MUNICIPAL WASTEWATER TREATMENT PLANTS

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Abstract

Wastewater septicity is becoming increasingly common due to global warming and reduction in the water usage. During wastewater transport, anaerobic conditions in sewers result in biochemical changes, with the most noticeable and problematic being sulphide production. Septic wastewater is known to cause concrete corrosion, odour nuisance and operational issues at downstream wastewater treatment plants (WWTPs). The aim of this study was to quantify the impact and identify operational issues created by treating septic wastewater in municipal wastewater treatment plants (MWWTP), particularly in primary settling, chemical phosphorus removal (CPR) and the activated sludge process. Septic wastewater was found to reduce the effectiveness of CPR starting at a 0.35 S:Fe molar ratio and only 10% phosphorus removal was measured at 1.4 S:Fe molar ratio. Septic wastewater with 6.4 mg/L of sulphide was found to impact activated sludge flocs, with significant proliferation of filamentous bacteria, resulting also in a reduced COD removal by 55% and nitrification by 44%. Furthermore, it was observed sludge bulking in the secondary settler and consequent biomass washout. Overall, this study quantified the difficulty and inefficiencies of a standard MWWTP to treat septic wastewater.

Keywords: Sulphide, Inhibition, Activated sludge, Nitrification, Phosphorus removal, Filamentous bacteria.

4.1 Introduction

Wastewater is transported from source to a central treatment plant through a sewerage system, that is often a long train of interconnecting pipes buried underground. Wastewater septicity develops naturally when the existing dissolved oxygen is consumed by microbes present in sewers, as organic matter is hydrolysed to short-chain organic compounds, such as volatile fatty acids (VFAs) (Hvitved-Jacobsen, Vollertsen and Nielsen, 2013). In anaerobic conditions, the VFAs and sulphate are reduced by sulphate reducing bacteria (SRB) generating sulphide as a by-product. As such, septic wastewater is characterised by low oxidation-reduction potential (ORP), presence of dissolved sulphide and an increase in VFAs (Boon, 1995; Rudelle et al., 2011). Wastewater septicity is expected to become more common in all sewerage systems due to a combination of several factors. i) Temperature increase due to global warming (Cintra Campos and Darch, 2015) ii) Water saving strategies being promoted (Sun et al., 2015) iii) Separate sewers being built instead of combined sewers iv) Centralisation of WWTPs. Furthermore, in some specific cases, huge in-sewer rainwater storage systems are being built, such as the Thames Tideway in London and Shieldhall Tunnel in Glasgow, that will further increase the risks associated with treating septic wastewater.

Most research on wastewater septicity has been traditionally focused on how septicity develops and the volatilisation of the dissolved sulphide into hydrogen sulphide gas. Hydrogen sulphide is the cause of microbially induced concrete corrosion (MICC), which was estimated to cost over €450 million per year in Germany and £85 million in the UK (Grengg *et al.*, 2018) and €5 million per year in the Flanders region of Belgium (Vincke, 2008). Hydrogen sulphide also causes odour nuisance and is a health and safety hazard, as it is life threatening at over 50 ppm (Park et al., 2014). However, there is a clear gap in research exploring the impacts septicity has in municipal wastewater treatment plants (WWTPs).

Septic wastewater has been observed to impact most of treatment processes in WWTPs; nevertheless, little research has been done on the impact of septicity on WWTPs (Chapter 2). The efficiency of chemical phosphorus removal (CPR) has been implied to be reduced due to wastewater septicity, although this has not been quantified yet. Some studies report competition between dissolved sulphide and orthophosphate to form precipitates with iron-based coagulants, in septic wastewaters (Nielsen et al., 2005; Firer, Friedler and Lahav, 2008). This suggests that phosphorus removal efficiency by iron-based coagulant is dependent on the sulphide concentration in the wastewater, for a given coagulant dose, but it is not clear what is this correlation. When investigating primary settling of septic wastewater, a lower total suspended solids (TSS) removal is expected, as the particulate matter is hydrolysed in the sewer, the particle size distribution (PSD) is shifted towards smaller particles sizes (Elmitwalli et al., 2001). Smaller particles have slower settling speeds and the settling tanks are not that effective in retaining them. Nevertheless, there is little information and studies that validate this hypothesis.

Secondary treatment is vastly dominated by activated sludge processes, applied for chemical oxygen demand (COD) and nitrogen removal in Europe.

Nitrification has been found to be partially inhibited by sulphide by several authors (Bejarano-Ortiz *et al.*, 2015; Kouba *et al.*, 2017; Sekine *et al.*, 2020). Both ammonia oxidizing bacteria (AOB) and nitrite oxidizing bacteria (NOB) were found to be inhibited by sulphide, with NOB being more sensitive (Delgado-Vela, Dick and Love, 2018). Sulphide concentrations as low as 1.43 mg/L have been reported to inhibit 50% of the AOB (Sekine et al., 2020). However, all of the nitrification inhibition studies to date were performed in small batch tests and using fresh wastewater with sulphide added to the matrix (Delgado-Vela, Dick and Love, 2018), which does not fully represent septic wastewater. Furthermore, septicity has been linked with the proliferation of filamentous bacteria in activated sludge flocs leading to sludge bulking in secondary settlers (Richard, 2003). The main filamentous bacteria related to septic wastewater treatment are sulphur oxidizing filamentous bacteria, such as *Thiothrix* spp., Type 021N and *Beggiatoa* spp. (Nielsen, de Muro and Nielsen, 2000).

Therefore, there is a need to assess the impact of treating septic wastewater on the treatment efficiency and operability of MWWTPs, focusing on nitrification and chemical phosphorus removal.

The main objective of the paper was to quantify the impacts of continuously treating septic wastewater on a MWWTP and to assess the operational issues that may arise from treating different sulphide concentrations in the septic wastewater.

4.2 Materials and Methods

Two identical pilot plants (Figure 4-1) consisting of a primary settler (4 L volume), an aerated tank (4 L volume) and a secondary settler (2 L volume) were used to assess the impact of septicity on the individual processes and effluent quality. The activated sludge reactor was seeded using nitrifying activated sludge from a nearby MWWTP collected from the RAS lane. The seed quantity was calculated to achieve a 2.5 g/L of MLSS in each reactor. The aeration was manually controlled to achieve a DO ~2 mg/L. The PST was manually desludged every 3-4 days to avoid rising sludge. Activated sludge was manually wasted to achieve a sludge retention time (SRT) of 12 days (Tchobanoglous *et al.*, 2014). The Return Activated Sludge (RAS) was pumped at 50% of the influent flow using a peristaltic pump.

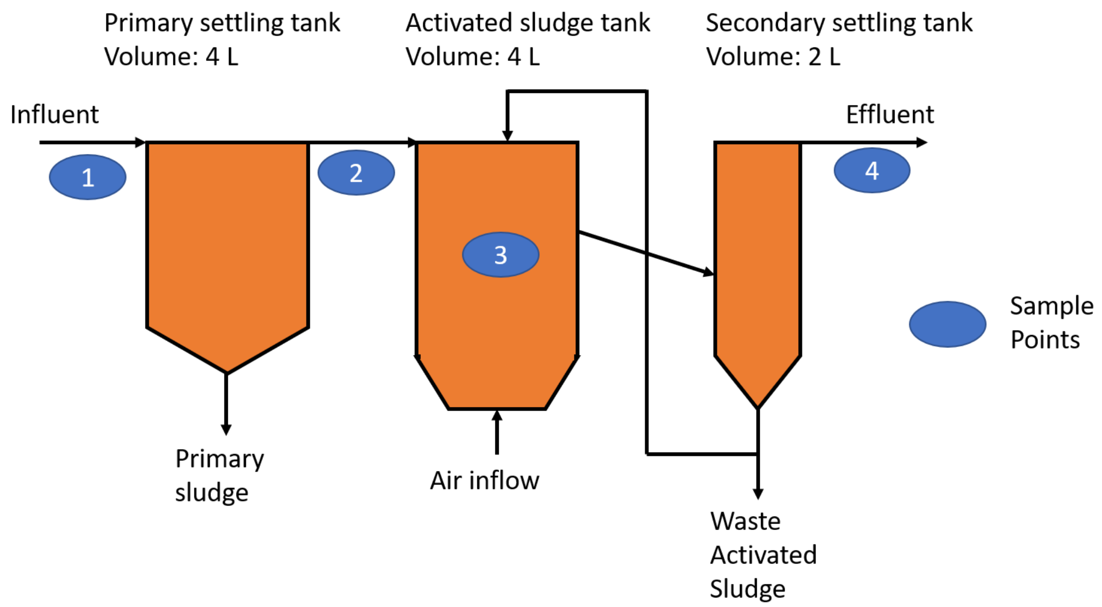

Figure 4-1 Schematic representation of WWTP pilot-plant used to study the impact of septic wastewater. Two pilot-plants with the same dimensions and design, were operated in parallel, a control plant (fed with fresh wastewater) and a test plant (fed with septic wastewater)

Wastewater samples were collected from the influent tank, primary settler effluent, activated sludge process and the pilot effluent (Figure 4-1) three times a week. At the end of each operational run and once during Run 3, activated sludge samples were collected to perform a sludge volume index (SVI) test and for microscopic observation, and immediately returned to the biological reactor. During Run 3, activated sludge samples were collected and used to perform respirometry tests.

4.2.1 Respirometry tests

Oxygen consumption was measured as a surrogate for biomass activity for the activated sludge on Run 3 using an aerobic electrolytic respirometer (Environmental Services Ltd, Cornwall, UK) to understand the nature of the inhibition observed in the pilot plant. Thirty millilitres of activated sludge sample were mixed with 30 mL of a synthetic solution: 150 and 300 mg/L COD as starch in the tests investigating heterotrophic bacteria activity; and 40 and 60 mg-N/L of ammonium sulphate with 320 and 480 mg-$CaCO_3$/L, in the tests investigating nitrifying bacteria activity. Respirometry tests were run for 1 day and they were done in triplicate. The oxygen consumed was measured automatically by the respirometer and the oxygen data was used to calculate the biomass activity.

4.2.2 Chemical phosphorus removal tests

Jar tests were performed on a programmable jar tester (Phipps & Bird PB-900) to assess the impact of wastewater septicity on chemical phosphorus (P) removal. For this, septic wastewater was prepared in batches, by storing the fresh raw municipal wastewater for up to 3 days and sulphide concentration was adjusted by adding sodium sulphide. The coagulant used was ferric chloride at a fixed dose of 2.5 mg Fe/ mg total P. The jar test procedure was as followed: 250 rpm for 1 minute (ferric dosed at this time), 25 rpm for 15 minutes and 0 rpm for 30 minutes. Overall, phosphorus removal efficiency was investigated for 5 different sulphide to iron ratios from 0.5-1.8 S:Fe molar ratio. Samples were collected from the supernatant and the jar tests for each S:Fe ratio were done in triplicate.

4.2.3 Analytical procedures

Total suspended solids (TSS) and volatile suspended solids (VSS) were measured following Standard Methods (APHA, 2005). Ammonia (114558), total and soluble phosphorus (114543), nitrate (114764), total nitrogen (TN) (114763), total and soluble COD (114540) and dissolved sulphide (114779) were measured using cell tests and measured in the Spectroquant (Merk, UK). pH, ORP, DO and temperature were measured in-situ at the pilot plant using a HQD meter with Intellical probes (PHC101, MTC101 and LDO101, respectively) (Hach, UK).

4.2.4 Statistical analysis

The MWWTP pilot plant results for the fresh and septic fed pilots were statistically compared using the Student's t-test assuming unequal variances method integrated in Microsoft Excel. The analysis returns a p value (significance) which defines if the means for the two groups (in this case the fresh fed pilot and the septic fed pilot) are statistically different or not. For the means to be statistically different a p value <0.05 was selected.

4.3 Results and Discussion

One of the pilots was fed with fresh raw municipal wastewater from a full-scale WWTP and the other one was fed with septic wastewater. The septic wastewater was prepared in batches, by storing the fresh raw municipal wastewater for up to 3 days at room temperature in a closed container. The characterisation of the fresh and septic wastewater is presented in Table 4-1. The pilot plants were operated under three different conditions (Run 1, Run 2 and Run 3) for six weeks after they had reached stable conditions. The flowrate was 10 L/day in Run 1 and maintained at 20 L/day in Run 2 and 3. The sulphide concentration in the septic wastewater was also increased to 6.4 mg/L in Run 3 by adding sodium sulphide to the septic wastewater (Table 4-1). The operational conditions of the activated sludge processes were the same for both pilot plants (Table 4-2).

Table 4-1 Fresh and septic wastewater influent feed characterisation for Run 1, 2 and 3

		Run 1			Run 2			Run 3		
Parameter	**Unit**	**Fresh**	**Septic**	**Significance**	**Fresh**	**Septic**	**Significance**	**Fresh**	**Septic**	**Significance**
pH	-	8.0 ± 0.2	7.9 ± 0.1	No ($p = 0.12$)	8.25 ± 0.2	8.1 ± 0.2	Yes ($p = 0.01$)	8.0 ± 0.1	8.2 ± 0.2	Yes ($p = 0.01$)
Redox Potential	mV vs SHE	163 ± 81	38 ± 89	Yes ($p = 0.00006$)	181 ± 31	22 ± 28	Yes ($p = 10^{-25}$)	193 ± 8	-3 ± 11	Yes ($p = 10^{-28}$)
TSS	mg/L	189 ± 88	71 ± 34	Yes ($p = 0.0007$)	110 ± 36	75 ± 34	Yes ($p = 0.0005$)	136 ± 44	73 ± 28	Yes ($p = 0.0001$)
Soluble COD	mg/L	60 ± 17	63 ± 12	No ($p = 0.57$)	82 ± 12	82 ± 15	No ($p = 0.89$)	44 ± 6	48 ± 8	No ($p = 0.12$)
Total Nitrogen	mg N/L	47 ± 9	49 ± 11	No ($p = 0.67$)	58 ± 7	55 ± 7	No ($p = 0.13$)	34 ± 5	32 ± 5	No ($p = 0.26$)
Ammonia	mg N/L	26 ± 7	33 ± 5	Yes ($p = 0.001$)	40 ± 5	42 ± 5	No ($p = 0.2$)	18 ± 4	20 ± 3	No ($p = 0.06$)
Sulphide	mg S/L	0 ± 0	1.9 ± 1.3	Yes ($p = 0.00001$)	0 ± 0	1.5 ± 0.8	Yes ($p = 10^{-10}$)	0 ± 0	6.4 ± 0.9	Yes ($p = 10^{-13}$)

Table 4-2 Operational conditions of the pilot plant for Run 1, 2 and 3

			Run 1			Run 2			Run 3		
	Parameter	**Unit**	**Fresh**	**Septic**	**Significance**	**Fresh**	**Septic**	**Significance**	**Fresh**	**Septic**	**Significance**
PST	**Solids loading rate**	kg $TSS/m^3.d$	0.55 ± 0.27	0.20 ± 0.09	Yes (p = 0.00003)	0.57 ± 0.20	0.37 ± 0.16	Yes (p = 0.0001)	0.68 ± 0.22	0.37 ± 0.14	Yes (p = 0.0001)
Activated Sludge Process	**Hydraulic retention time (HRT)**	hours	10	10	NA	5	5	NA	5	5	NA
	Mixed liquor suspended solids (MLSS)	mg/L	3292 ± 455	2830 ± 460	Yes (p = 0.003)	2535 ± 428	2337 ± 454	No (p = 0.06)	2937 ± 510	2314 ± 277	Yes (p = 0.0004)
	Temperature*	°C	9 ± 2	9 ± 2	No (p = 0.68)	13 ± 4	13 ± 4	No (p = 0.89)	8 ± 3	8 ± 3	No (p = 0.87)
	Dissolved oxygen (DO)	mg O_2/L	3.9 ± 2	3.9 ± 1	No (p = 0.92)	2.9 ± 2	2.2 ± 2	No (p = 0.2)	4.1 ± 3	4 ± 2	No (p = 0.52)
	Organic loading rate (OLR)	kg $COD/m^3.d$	0.28 ± 0.12	0.25 ± 0.09	No (p = 0.34)	0.56 ± 0.16	0.51 ± 0.08	No (p = 0.08)	0.38 ± 0.18	0.44 ± 0.15	No (p = 0.36)
	Nitrogen ammonia loading rate (NLR)	kg $N/m^3.d$	0.07 ± 0.02	0.08 ± 0.01	Yes (p = 0.001)	0.20 ± 0.03	0.21 ± 0.03	No (p = 0.19)	0.09 ± 0.02	0.10 ± 0.01	No (p = 0.06)
	Food to microorganism ratio (F:M)	kg COD/kg VSS.d	0.1 ± 0.05	0.11 ± 0.04	No (p = 0.67)	0.24 ± 0.09	0.24 ± 0.07	No (p = 0.98)	0.15 ± 0.07	0.2 ± 0.06	Yes (p = 0.03)

*Pilot-plants were operated during Autumn and Winter, and wastewater temperature was aligned and normal for the season of the year.

The influent wastewater (Table 4-2) showed typical low-medium strength municipal wastewater concentrations (Tchobanoglous *et al.*, 2014). The septic influent wastewater for Run 1 and 2, with no chemical addition, showed septicity levels that were low, which can be deduced by the low sulphide and near 0 mV ORP (Rudelle *et al.*, 2016). In Run 3, the sulphide level was increased to 6.4 mg/L by sodium sulphide addition to simulate an average septic wastewater (Chapter 2).

The fresh and septic wastewater were fed to the respective pilot plants and the solids removal was investigated in the primary settling tanks (PST) (Table 4-3). The PST fed with fresh wastewater achieved an average TSS removal of 64.7%, 67.4% and 71.9% for Run 1, 2 and 3, respectively, whereas the PST fed with septic wastewater achieved an average TSS removal of 36%, 62.3% and 44.2% for Run 1, 2 and 3, respectively. Although the TSS removal rates were lower for the PST fed with septic wastewater, the PST effluent TSS were also lower on the three runs (Table 4-3). Therefore, wastewater septicity did not show any conclusive impact on the PST performance, as the solids loading rates were not comparable for the fresh and septic wastewater (Table 4-2). The solids loading difference was likely due to the lower mixing speed applied to the septic wastewater feed tank in order to avoid reoxygenation of the wastewater.

Table 4-3 PST performance over the test period for Run 1, 2 and 3

	Run 1		**Run 2**		**Run 3**	
Parameters	**Fresh**	**Septic**	**Fresh**	**Septic**	**Fresh**	**Septic**
PST Effluent TSS (mg/L)	68.7 ± 23.6	50.4 ± 21.7	39.5 ± 26.9	24.6 ± 11.9	39.5 ± 28.1	37.2 ± 15.8
TSS Removal (%)	64.7 ± 15.6	36.0 ± 22.6	67.4 ± 19.3	62.3 ± 21.7	71.9 ± 15.6	44.2 ± 29.6

After the ASP was seeded, each pilot was fed with fresh and septic wastewater, respectively, until the process was determined to be stable (3 weeks minimum to allow for microbial changes), which was assessed based on the stabilisation of the operating MLSS.

The organic loading rates (OLR) applied were of 0.28 ± 0.12 and 0.25 ± 0.09 kg COD/m^3/d for the fresh and septic fed ASP in Run 1. The OLR were increased for Run 2 and 3 by doubling the influent flow from 10 L/d to 20 L/d. The OLR applied were of 0.56 ± 0.16 and 0.51 ± 0.08 kg COD/m^3/d for the fresh and septic fed ASP in Run 2 and 0.38 ± 0.18 and 0.44 ± 0.15 kg COD/m^3/d for the fresh and septic fed ASP in Run 3. The average total COD removal efficiency in Run 1 was comparable for both ASPs, at 72 ± 15 and 67 ± 8% for the fresh and septic fed ASPs, respectively. In Run 2, the average total COD removal efficiency was lowered to 33 ± 17 and 19 ± 11% for the fresh and septic fed ASP, respectively. The reduction in the COD removal efficiency is likely to be due to higher inert COD being present on the influent wastewater. In Run 3, the average total COD removal efficiency was significantly lower for the septic fed ASP, at 62 ± 14 and 28 ± 26% for the fresh and septic fed ASP, respectively. Overall, the COD removal efficiency was reduced when treating septic wastewater at high OLRs (Run 2 and Run 3). One of the reasons for the observed COD removal reduction in Run 3 was the reduced average MLSS of 2314 mg/L in the septic fed ASP compared to 2937 mg/L in the fresh fed ASP (Table 4-2), which resulted in a F:M ratio of 0.2 kg COD/kg VSS/d compared to 0.15 COD/kg VSS/d, respectively. However, the reduced COD removal cannot be explained only by the reduced MLSS concentration and it is likely that the heterotrophic bacteria were impacted by the ORP of -3 mV and the 6.4 mg/L of sulphide present in the septic wastewater.

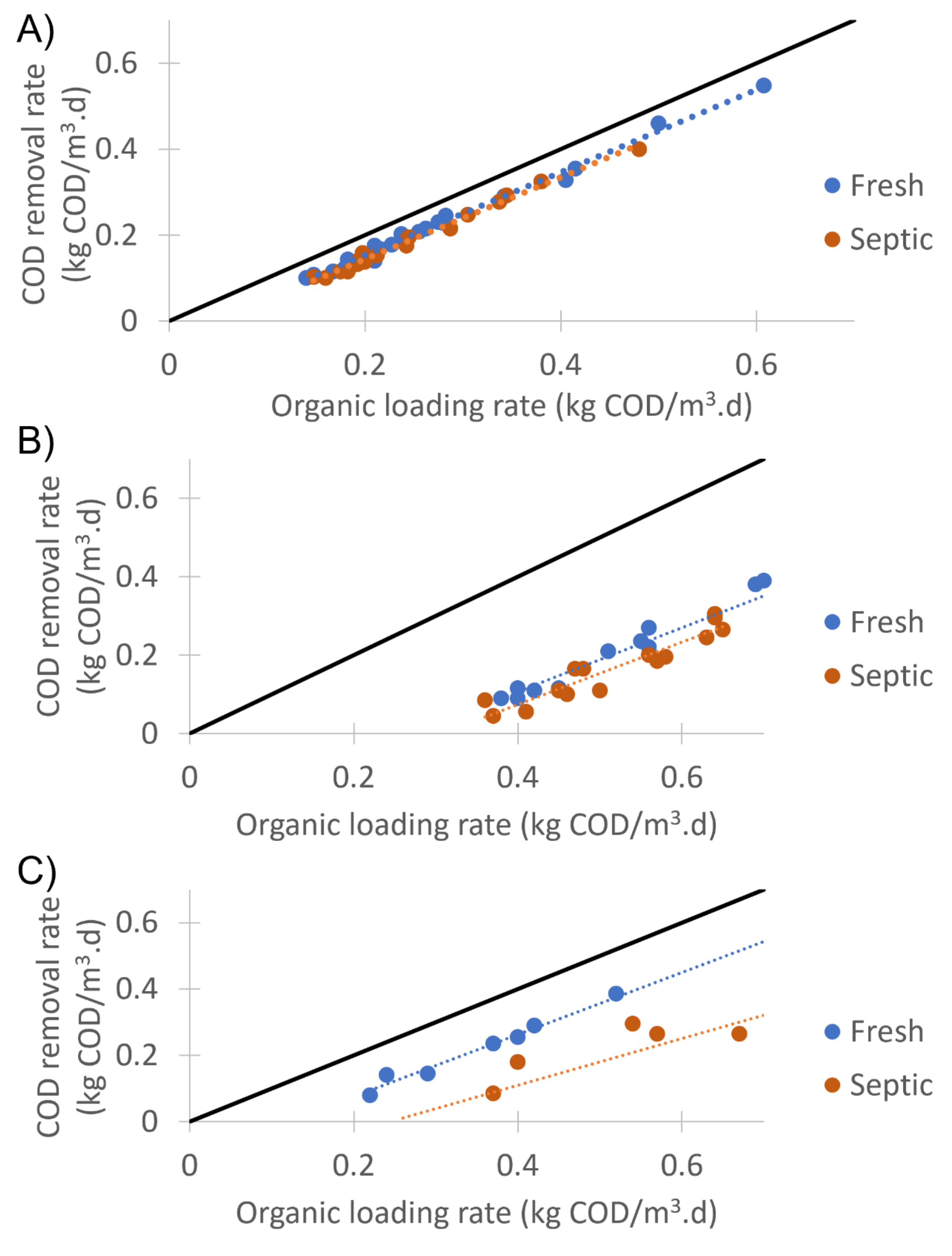

Figure 4-2 COD removal rate in the activated sludge process fed with fresh and septic wastewater for Run 1 (n = 20) (A), Run 2 (n = 15; double influent flow) (B) and Run 3 (n = 8; double influent flow and 6.4 mg/L sulphide) (C)

The average nitrification efficiency in Run 1 was of 98 ± 4 and 91 ± 18% for the fresh and septic fed ASPs, respectively. The nitrification efficiencies for Run 1 were assessed using a t-test and the efficiencies were statistically comparable for both ASPs ($p = 0.053$). The average nitrification efficiency in Run 2 was of 93 ± 8 and 80 ± 10% for the fresh and septic ASPs, respectively. The nitrification efficiencies for Run 2 were assessed using a t-test and the efficiencies were different ($p = 0.001$). The average nitrification efficiency in Run 3 was of 82 ± 16 and 46 ± 20% for the fresh and septic ASPs, respectively. The nitrification efficiencies for Run 3 were assessed using a t-test and the efficiencies were different ($p = 0.002$). Overall, increasing the OLR from 0.25 kg COD/m^3/d (Run 1) to 0.51 kg COD/m^3/d (Run 2) reduced the nitrification efficiency of the septic fed ASP from 91% to 80%, and was further reduced to 46% when the influent sulphide concentration was increased from 1.5 mg/L (Run 2) to 6.4 mg/L (Run 3). The respirometry tests for the activated sludge in Run 3 showed that with ammonia as a substrate, the oxygen consumption rate was of 200 ± 43 mg O_2/L/h/g MLVSS and 161 ± 4 mg O_2/L/h/g MLVSS for the activated sludge from the fresh and septic fed ASPs, respectively. The oxygen consumption rates were assessed using a t-test and the rates were comparable ($p = 0.256$). The respirometry tests showed that the difference observed in the nitrification efficiency in Run 3 between the fresh and septic fed ASPs was caused by the different influent characteristics (Table 4-1). The direct inhibition of nitrifying populations in the presence of sulphide had been previously reported (Bejarano-Ortiz *et al.*, 2015; Delgado-Vela, Dick and Love, 2018).

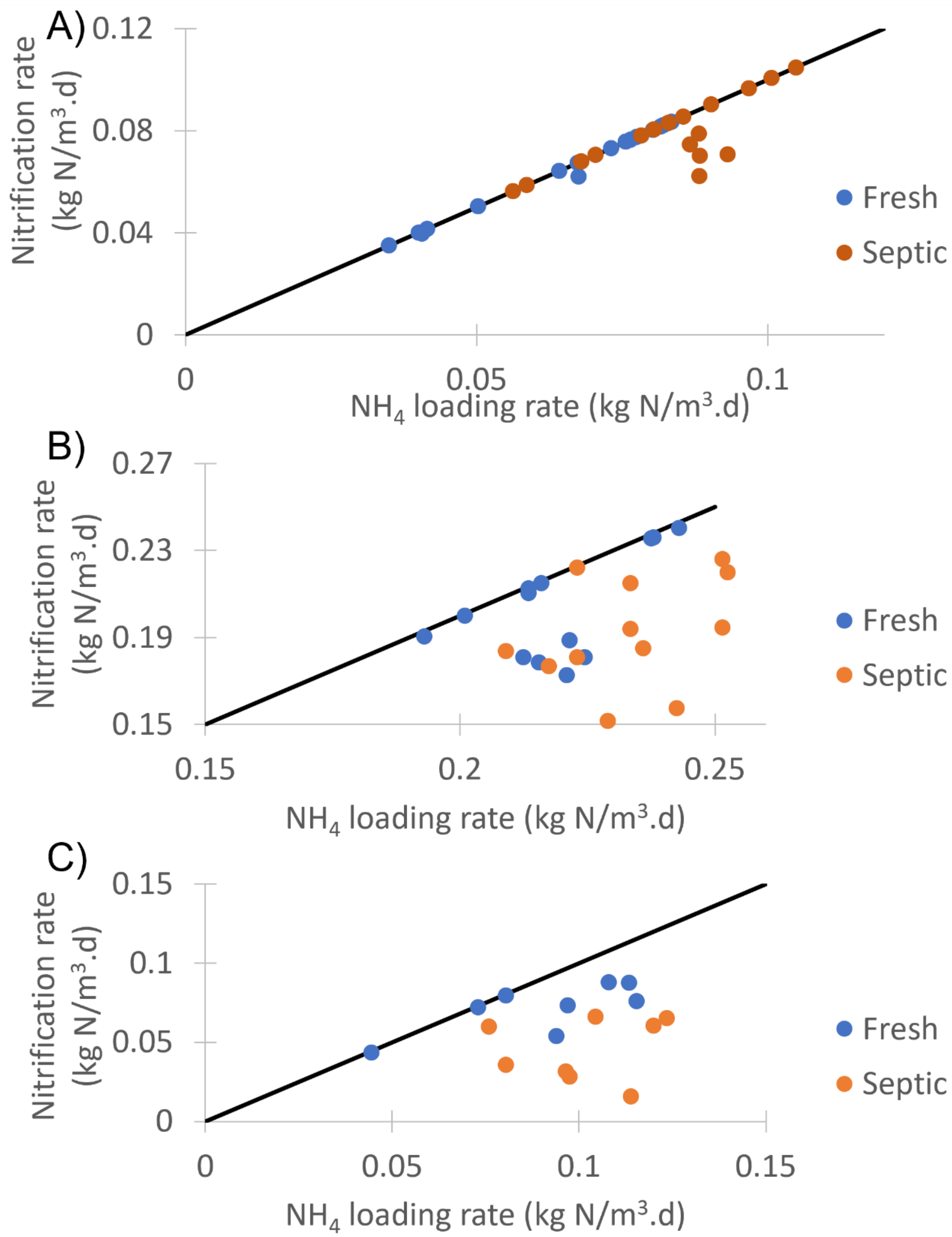

Figure 4-3 Nitrification removal rate for activated sludge fed with fresh and septic wastewater for Run 1 (n = 20) (A), Run 2 (n = 15; double influent flow) (B) and Run 3 (n = 8; double influent flow and 6.4 mg/L sulphide) (C)

The sludge volume index (SVI) of the activated sludge process at the end of Run 1 was of 144 and 69 mL/g for the fresh and septic fed processes, respectively.

Both of the ASPs were operating under acceptable SVIs <150 mL/g (Richard, 2003). At the end of Run 2, the SVI of the ASP was of 116 and 96 mL/g for the fresh and septic fed processes, respectively. The SVI of the activated sludge process treating septic wastewater in Run 3 increased to 253 mL/g in day 24 and 289 mL/g in day 41 (Figure 4-4), while the ASP treating fresh wastewater showed a SVI of 100 mL/g in day 24 and of 51 mL/g in day 41. The high SVIs measured in the septic fed ASP led to a reduction in MLSS during Run 3, from 2840 mg/L on day 24 to 1800 mg/L on day 40. By day 40 it was observed the washout of the activated sludge.

Microscopic observations of the activated sludge flocs at the end of Run 1 showed healthy looking flocs with ciliates and flagellates for both the fresh and septic fed ASPs. At the end of Run 2, the flocs had a high abundance of flagellates and ciliates for both the fresh and septic fed ASPs. In the microscopic observations of Run 3 (Figure 4-4), filamentous bacteria overgrowth was clearly visible in the activated sludge flocs treating septic wastewater, which was not observed in any other activated sludge samples of the ASPs treating septic wastewater in Run 1 and Run 2. The sulphide concentration being the only significant difference from Run 3 compared to Run 2, the filamentous bacteria overgrowth was determined to be caused by the sulphide concentration, at an average concentration of 6.4 mg/L. Although the filamentous bacteria morphotype was not identified, Nielsen, de Muro and Nielsen, (2000) have shown that sulphide oxidising filamentous bacteria, proliferate when treating sulphide rich wastewater.

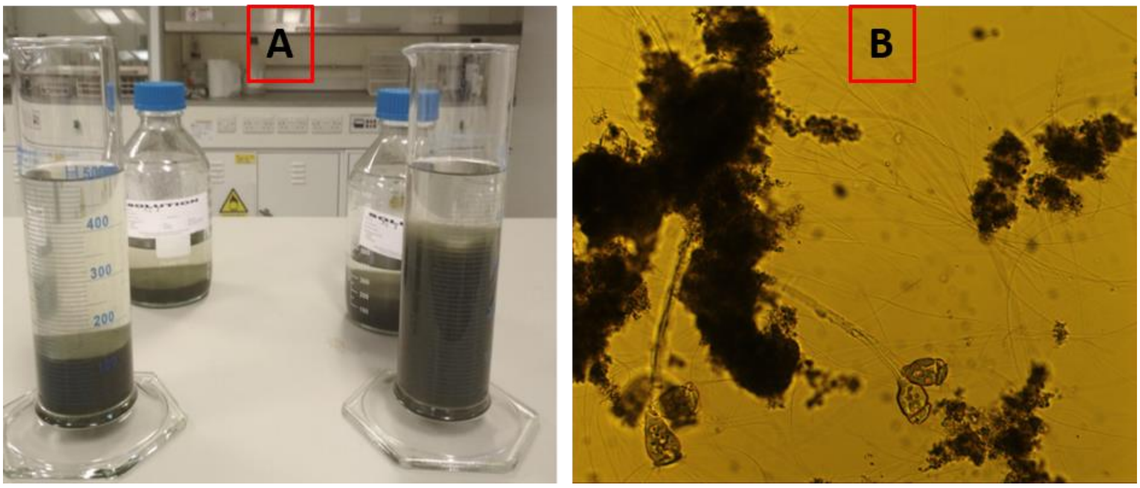

Figure 4-4 Sludge volume index (SVI) test for activated sludge solids during Run 3 fed with fresh (left in A) and fed with septic wastewater (right in A) and optical microscope 10x image of activated sludge flocs fed with septic wastewater in Run 3 (B)

There was not a noticeable impact in the effluent quality when feeding septic wastewater compared to feeding fresh wastewater in Run 1. The total COD and ammonia were of 27 ± 11 mg/L and 0.3 ± 0.7 mg N/L for the fresh fed pilot plant and 30 ± 6 mg/L and 3.8 ± 4.8 mg N/L for the septic fed pilot plant. When the OLR was doubled from 0.25-0.28 kg COD/m^3/d in Run 1 to 0.51-0.56 kg COD/m^3/d in Run 2, the difference in final effluent total COD and ammonia increased, with concentrations of 73 ± 5 mg/L and 3.2 ± 3.7 mg N/L for the fresh fed pilot plant and 81 ± 9 mg/L and 8.8 ± 4.6 mg N/L for the septic fed pilot plant. When the influent sulphide concentration was increased in Run 3, the final effluent quality difference increased even further, with total COD and ammonia concentrations of 27 ± 4 mg/L and 3.8 ± 3.3 mg N/L for the fresh fed pilot plant and 62 ± 16 mg/L and 11.3 ± 4.8 mg N/L for the septic fed pilot plant (Figure 4-5). The ammonia concentrations observed in the final effluent of the septic fed pilot plant in Run 3 highlight the severe impact septicity can have on the performance of a municipal wastewater treatment plant, which would have breached a 10 mg N/L ammonia permit. Furthermore, the final effluent TSS concentration nearly triplicated in Run 3 (average of 27 mg/L) compared with the TSS concentration in Run 1 and Run 2 (average of 12 and 10 mg/L, respectively). The final effluent TSS increase is linked to the proliferation of filamentous bacteria observed in the microscope.

In this study the impact of septic wastewater on chemical phosphorus removal was also investigated, which is the most commonly used process for removing phosphorus from wastewater (Wilfert *et al.*, 2015). Jar tests were performed (as described in Section 4.2.2) to quantify the impact of septicity on chemical phosphorus removal. The wastewater used had a range of 3.4-7.8 mg/L of total phosphorus, which is in the range of municipal wastewater ((Tchobanoglous *et al.*, 2014)). The S:Fe molar ratios used ranged from 0 to 2, which offered enough spread to quantify the impact of septicity on chemical phosphorus removal as the expected product was ferrous sulphide (FeS), which has a 1:1 S:Fe molar ratio (Firer, Friedler and Lahav, 2008).

Jar tests showed a reduction in the phosphorus removal by 90% with increasing sulphide to iron molar ratios. Up to 0.3-0.4 S:Fe molar ratio, there was a threshold and stable P removal was observed. At S:Fe molar ratios >0.35 there was significantly impact P removal and only a 10% removal was measured at a S:Fe molar ratio of 1.4 (Figure 4-6). The breakpoint at which the P removal deteriorated corresponded to a dissolved sulphide of 3 mg S/L and 5 mg P/L of total phosphorus. Sulphide in septic raw wastewater has been found to be in the range of 0.5-17 mg/L (Chapter 2). Therefore, the impact of septicity on chemical phosphorus removal is particularly relevant to raw wastewater dosing. Increasing levels of sulphide in the wastewater will require to increase the iron dose above the 2.5 mg Fe/mg total P used in this study to achieve the required phosphorus removal. Overall sulphide was found to significantly decrease the phosphorus removal efficiency for a fixed ferric dose.

The reduction in the phosphorus removal efficiency is thought to be due to the thermodynamic advantage of sulphide to precipitate with the iron dosed over the phosphate (Hu *et al.*, 2019). During the jar tests an intense dark colour was observed in the wastewater, which is characteristic of the ferrous sulphide precipitates. The formation of ferrous sulphide (FeS) when iron and sulphide are available in wastewater was observed by Firer, Friedler and Lahav (2008) when dosing ferric for sulphide removal and by Wilfert *et al.* (2020) when dosing sulphide to recover phosphate from iron rich wastewater sludge.

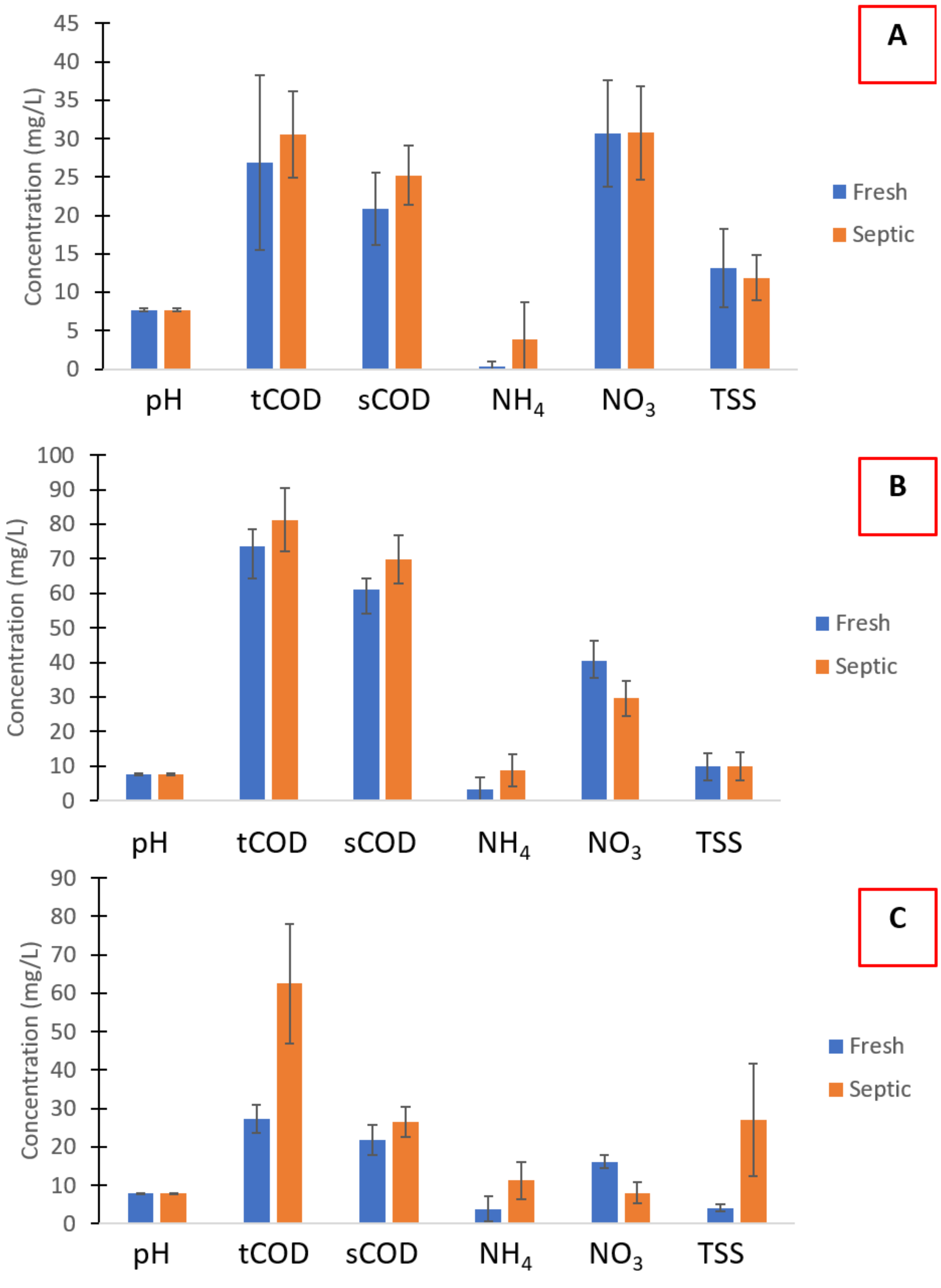

Figure 4-5 Effluent quality of the wastewater treatment plant pilots in Run 1 (A), Run 2 (B) and Run 3 (C)

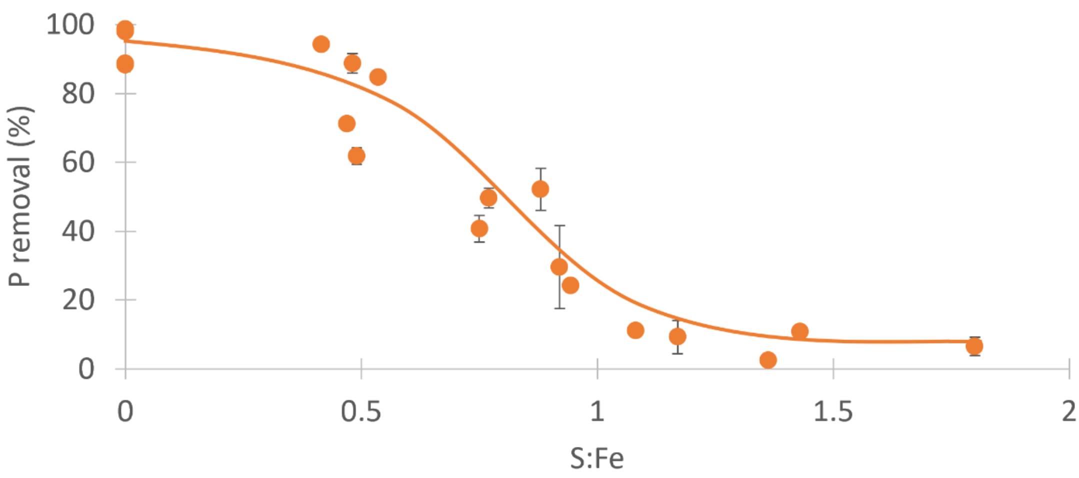

Figure 4-6 Phosphorus removal during chemical P removal at a dose of 2.5 mg Fe-$FeCl_3$/mg total-P at different sulphide to iron molar ratio

4.4 Conclusions

In this study, the implications on process efficiency and effluent quality when treating septic wastewater by a municipal wastewater treatment plant pilot were measured and the efficiency of chemical phosphorus removal on septic wastewater was quantified. The key findings were:

- In an activated sludge process, COD removal and nitrification were directly impacted by the septicity in the feed wastewater, with the COD and nitrification efficiency being reduced by 55% and 44% when the process was fed with septic wastewater containing 6.4 mg/L sulphide.
- Treating septic wastewater containing 6.4 mg/L of sulphide resulted in the proliferation of filamentous bacteria and activated sludge bulking that led to biomass washout.
- The efficiency of chemical phosphorus removal was negatively impacted at S:Fe molar ratios higher than 0.35, reaching a minimum 10% removal rate at a S:Fe molar ratio of 1.4.

4.5 Acknowledgements

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5 USE OF A NOVEL DISSOLVED SULPHIDE SENSOR TO MONITOR SULPHIDE AND BUILD A PREDICTION MODEL USING LONG SHORT-TERM MEMORY ARTIFICIAL NEURAL NETWORK

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Abstract

Hydrogen sulphide generated at long rising mains results in corrosion and odour nuisance at manholes and at wastewater treatment plant (WWTP) inlet structures. Hydrogen sulphide monitoring, particularly the dissolved hydrogen sulphide, is an underdeveloped area and is commonly achieved by grab sample analysis. In this study, the aim was to identify the source of the corrosion observed at an inlet chamber of a full-scale WWTP, which had 12 rising mains discharging into it. For that purpose, a novel dissolved hydrogen sulphide sensor was installed at the outlet of three rising mains (RM1 to RM3), as well as the combined inlet (CI). The sensor measurements, with a 5 minute resolution, showed a very distinct hydrogen sulphide daily pattern that was correlated with the inverse of the typical flowrate pattern to the WWTP. RM1 detected the highest hydrogen sulphide load at 3.6 kg/d during the cold months and 4.2 kg/d during the warm months. RM3 and RM2 detected loads of 2.96 kg/d and 0.98 kg/d, respectively, during the cold months. The RM1 outlet dissolved hydrogen sulphide concentrations were predicted using a long short-term memory (LSTM) artificial neural network (ANN) model. The model used as inputs the rising main flowrate, the wastewater temperature and the time of the day. The model achieved a root mean square error (RMSE) of 0.34 and a Nash-Sutcliffe efficiency (NSE) of 0.57, and was able to predict the distinct pattern observed in the monitored hydrogen sulphide data. The novel dissolved hydrogen sulphide sensor offered a great insight into hydrogen sulphide dynamics in the sewerage system and at WWTP and could be used as a septicity warning system or as part

of a feedforward control system for hydrogen sulphide treatment chemical dosing. Furthermore, the development of hydrogen sulphide prediction models allows for the repurposing of the dissolved hydrogen sulphide sensors, allowing for savings in both capital and operational costs.

Keywords: Long Short-Term Memory, Recurrent Neural Network, Monitoring, Rising Main, Corrosion.

5.1 Introduction

Wastewater delivery to centralised wastewater treatment plants (WWTPs) can result in wastewater becoming septic, particularly at large sewerage networks. Septicity is characteristically identified by wastewater having a dark colour and rotten eggs smell. However, septicity is not a risk due to its colour or smell, it is mainly a risk due to the dissolved hydrogen sulphide within the wastewater and its volatilisation to the air. The hydrogen sulphide contained in septic wastewater can get stripped to the air in gravity sewers and high turbulence areas, such as drops (Lahav, Sagiv and Friedler, 2006). The stripped hydrogen sulphide is known to cause corrosion to concrete and metal structures and poses a health and safety risk to humans. Even low concentrations of total dissolved sulphide, even <0.5 mg S/L, have been found to cause moderate concrete corrosion (Hvitved-Jacobsen, Vollertsen and Nielsen, 2013). The cost of microbial induced concrete corrosion (MICC) was found to be of £85 million per year in the UK and €450 million per year in Germany (Grengg *et al.*, 2018) and of €5 million per year in Flanders (Vincke, 2008). Hydrogen sulphide gas concentrations of 50 mg/L are already toxic to humans and 700 mg/L can cause death within minutes (Park *et al.*, 2014). Increasing population growth in cities and urban areas is and will result in larger sewerage networks, which will further increase septicity problems. Furthermore, the application of water saving strategies (Sun *et al.*, 2015) and the increase in global temperatures due to climate change (Cintra Campos and Darch, 2015) will further exacerbate the problem.

In vast sewerage networks, the first step to managing septicity is to identify septicity formation and hydrogen sulphide release hotspots. This is usually done by using one of the following methods: i) Measurement of hydrogen sulphide (either dissolved or in the gas phase) throughout the network or ii) Utilisation of a biochemical modelling tool such as WATS (Vollertsen *et al.*, 2015) or SEWEX (Sharma *et al.*, 2008). The first method consists of directly measuring the total dissolved sulphide by grab samples, which has the disadvantage of being labour intensive, not capturing the temporal variations and low precision due to the unstable nature of hydrogen sulphide (Sutherland-Stacey *et al.*, 2008). Hydrogen sulphide gas monitoring offers real-time monitoring opportunities utilising gas monitors, such as OdaLog. However, it has other disadvantages, such as not giving a direct measure of the septicity issue and high dependency on the positioning of the monitor (Pacheco Fernandez, Despot and Barjenbruch, 2021). The use of modelling tools also requires hydrogen sulphide measurement throughout the network when setting up the model. Therefore, practical and precise ways of measuring dissolved hydrogen sulphide in real-time are required.

Several dissolved hydrogen sulphide sensors can be found in literature, but most of them rely on stripping the dissolved hydrogen sulphide from the sample and measuring the hydrogen sulphide gas and then calculating the dissolved hydrogen sulphide based on Henry's law. These types of sensors are usually large and expensive, as they require a stripping chamber (Yavarinasab *et al.*, 2020). There are only a few sensors that can measure hydrogen sulphide in wastewater. The first use of a sensor to measure total dissolved sulphide was reported by Sutherland-Stacey et al. (2008), which relied on a UV/VIS spectrometer to measure the absorbance of the bisulphide ion and then combine it with a pH sensor to calculate the total dissolved sulphide. Recently, two more sensors have been developed, both based on electrochemically measuring hydrogen sulphide ions in wastewater. The first one was developed by Yavarinasab et al. (2020), but has been only reported to be trialled in aqueous solution and in a lab set-up and it is not commercially available. The second one is the commercially available SulfiLogger sensor (SulfiLogger, 2020), which has been shown in several case studies to be used for sewer management and optimisation of hydrogen sulphide mitigation chemical dosing (Despot, Pacheco Fernandez and Barjenbruch, 2021).

Although the total dissolved sulphide sensors offer high temporal resolution and precision, the cost of the sensors and maintenance required incur high costs for users. Therefore, the development of models to predict the total dissolved sulphide concentration at a single point would offer a great advantage, as they would allow for the sensors to be repurposed (moved to another location) once the model had been trained. Mechanistic models, such as WATS and SEWEX, offer great insights into the sewer processes and are widely applied for hydrogen sulphide prediction. However, they require extensive sampling campaigns measuring many parameters. On the other hand, data-driven models can work with fewer parameters and still provide accurate predictions, as has been shown for hydrogen sulphide formation prediction (El Brahmi and Abderafi, 2020; El Brahmi, Abderafi and Ellaia, 2021) and hydrogen sulphide emissions at WWTP inlets (Zounemat-Kermani, Stephan and Hinkelmann, 2019). Deep learning and particularly long short-term memory (LSTM) artificial neural network (ANN) has been shown to be great at sequential data prediction, as was the case for traffic prediction (Ma *et al.*, 2015) and sewer flow prediction (Zhang *et al.*, 2018).

Therefore, this study had two objectives: i) Determine the source of concrete corrosion at a WWTP inlet chamber where several rising mains were discharging using a novel dissolved hydrogen sulphide sensor (SulfiLogger), and ii) Develop an LSTM ANN model for one of the rising mains as proof of concept, to allow sensor repurposing.

5.2 Materials and Methods

5.2.1 Site description

The study WWTP is in the UK and treats wastewater for around 89,000 population equivalent (PE), mainly from domestic sources. The wastewater is collected at an inlet chamber, with 12 rising mains of different diameters discharging into it. The inlet chamber has severe signs of corrosion and it is due to be replaced, as it posed a structural risk. The source of the corrosion was unknown due to the high number of rising mains discharging into the same chamber and the objective of the work was to identify the key contributors to the overall corrosion.

Out of the 12 rising mains, 3 were selected to be monitored, due to their size, colour of the wastewater, anecdotical evidence and a previous catchment wide septicity potential study. For this study, these have been labelled as rising main 1, 2 and 3 (RM1, RM2, RM3) and their characteristics are defined in Table 5-1.

Rising main 1 (RM1) had calcium nitrate dosing at the pumping station wet well during the monitoring period. The calcium nitrate dosing was controlled based on temperature, wastewater flow and potentially more parameters (the dose was controlled by a contractor under a patented control philosophy). The average daily calcium nitrate dose was of 338.6 L/day.

Table 5-1 Characteristics of the rising mains to be monitored

Rising Main	Diameter (m)	Length (m)	Dry weather flow (m^3/day)	Nitrate dosing (Y/N)
1	0.6	6,800	6,001	Y
2	0.6	5,300	4,908	N
3	0.38	3,100	2,960	N

5.2.2 SulfiLogger sensor

SulfiLogger S1/X1-1020 (SulfiLogger A/S, Risskov, Denmark) were used in this study (hereby referred to as SulfiLogger). SulfiLogger measured H_2S electrochemically. The H_2S was measured by a current produced when the H_2S in the liquid or gas phase penetrates the silicone membrane at the sensor's tip and is subsequently electrochemically oxidised (Despot, Pacheco Fernandez and Barjenbruch, 2021). The sensor is made of stainless steel, it's lightweight (0.85 kg), compact and has a passive anti-fouling flush front (SulfiLogger, 2020). The sensor has a measuring range of 0-5 mg H_2S/L and can be set-up at a measuring frequency as low as 1 minute. The sensor was calibrated every 3 months, according to the manufacturer's instructions, by mounting the calibration cap to the sensor for a feed of calibration gas at 1,000 mg/L H_2S (SulfiLogger, 2020).

5.2.3 Sensor location

Six SulfiLogger sensors were acquired and installed, as seen in Figure 5-1. Two were installed at two terminal pumping stations, for RM1 and RM2, respectively. They were installed hanging with a chain into the wet well and connected to a PowerCom Box, which provided power to the sensor and sent the data to the collection platform. Three were installed at the outlet of the rising mains, within the inlet chamber. These were installed using scaffolding and were introduced into the respective bellmouth. The last one was installed at the combined inlet (CI) after the inlet chamber.

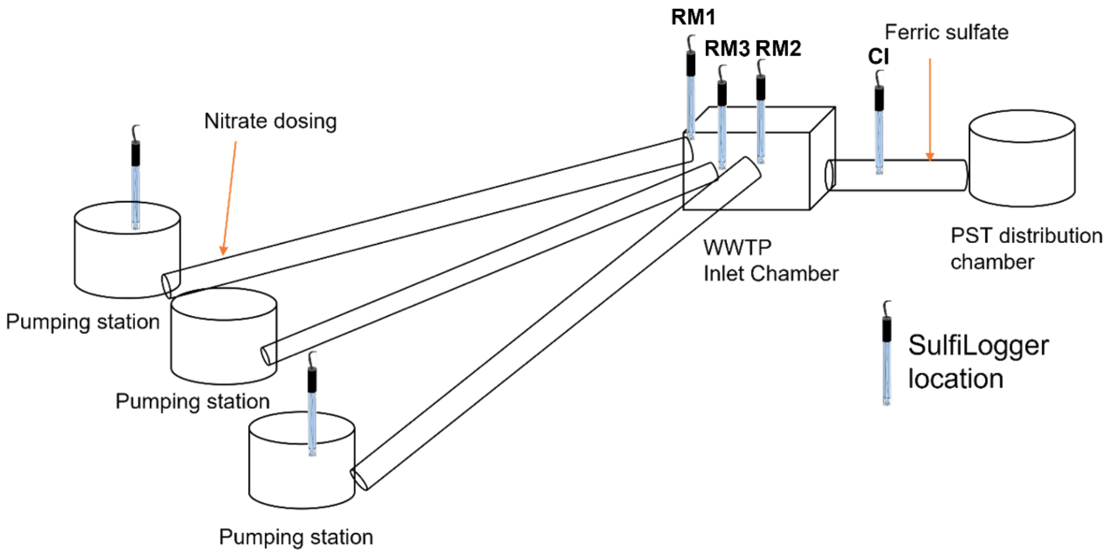

Figure 5-1 SulfiLogger sensor locations

5.2.4 Monitoring campaign

The sensors were installed and data was collected for 9 months (start of summer to next spring) at a measurement frequency of 5 minutes. The sensors data were further validated by grab sample analysis and comparison to the sensor measurement. The sensor measurements were stored in the PowerCom Box and were transmitted through a phone signal to the data collection webpage every half a day. The PowerCom Box batteries had to be replaced every 3 months, which was conveniently done when calibrating the sensors.

The monitoring campaign started on July 2020 with 3 sensors installed at the wet well of RM1, at the outlet of RM1 and at the combined inlet to the WWTP. After 3 months of monitoring, 3 more sensors were installed at the wet well of RM2, the outlet of RM2 and the outlet of RM3 (Figure 5-1).

5.2.5 Analytical measurements

Ammonia, total and soluble COD and total dissolved sulphide were measured using Spectroquant cell tests (Merk, UK). pH, ORP, DO and temperature were measured in-situ at the WWTP using a HQD meter with Intellical probes (PHC101, MTC101 and LDO101, respectively) (Hach, UK).

5.2.6 LSTM model set-up

An LSTM model was developed to predict the hydrogen sulphide concentration at the outlet of RM1, based on readily available input parameters. The input parameters selected were wastewater flowrate on the rising main, wastewater temperature and time of the day. The available data was cleaned by replacing missing values with the average between the two closest datapoints. Then, the data was normalised for each parameter using the min-max normalisation technique (Equation (5-1)), which converted all indicators to values between 0 to 1.

$$x_{norm} = \frac{x_i - \min(x)}{\max(x) - \min(x)} \quad \textbf{(5-1)}$$

In order to train the model, the dataset was split into an 80% training set and a 20% test set. The model parameters were fine-tunned using the mean squared error loss function. The optimal model was achieved using the following parameters: i) 1 LSTM hidden layer with 60 units, ii) optimiser: “Adam”, iii) epochs: 150, iv) batch size: 32.

The LSTM model was developed in Python 3, using the Keras deep learning library. Other Python libraries were also used, such as NumPy, Scikit-learn and matplotlib.

5.2.7 Model performance comparison

The performance of the LSTM model was assessed by comparing it to a linear and a polynomial regression model. The polynomial regression was fitted to a sixth-grade polynomial. The model performance criteria used in this study were the root mean square error (RMSE) and the Nash-Sutcliffe efficiency (NSE). The RMSE was calculated as Equation (5-2):

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n}\left(y_i^{obs} - y_i^{sim}\right)^2}{n}} \tag{5-2}$$

NSE is a normalized statistic that determines the relative magnitude of the residual variance ("noise") compared to the measured data variance ("information"). The NSE was calculated as Equation (5-3).

$$NSE = 1 - \left[\frac{\sum_{i=1}^{n}\left(y_i^{obs} - y_i^{sim}\right)^2}{\sum_{i=1}^{n}\left(y_i^{obs} - y^{mean}\right)^2}\right] \tag{5-3}$$

5.3 Results and Discussion

5.3.1 Monitoring results

The sensors RM1 and RM2 showed that no dissolved hydrogen sulphide was present at the wet wells, with concentrations below 0.01 mg/L hydrogen sulphide being measured consistently within the 9 and 6 months of monitoring, respectively. The hydrogen sulphide concentrations at the outlets of RM1, RM2 and RM3, as well as the CI concentrations, showed a very distinct daily pattern throughout the dry days of the monitoring period (Figure 5-2). The hydrogen sulphide concentration increased during the night time (00:00 to 8:00), was reduced during the morning peak (8:00 to 12:00) and was moderate during the afternoon and evening (12:00 to 00:00). The hydrogen sulphide concentration profile was the inverse of the daily flow pattern to a typical WWTP (Tchobanoglous *et al.*, 2014) and therefore, followed quite well the pattern of the hydraulic residence time (HRT) in the rising main. The dependence of hydrogen sulphide formation on HRT is well known, as had been shown by several authors when proposing empirical equations for hydrogen sulphide prediction that included the HRT term (Thistlethwayte, 1972; Pomeroy and Parkhurst, 1977; Nielsen *et al.*, 2008). The hydrogen sulphide was always measured as 0 mg/L, i.e. below the detection limit, in wet weather conditions, due to HRTs <4 hours in the rising mains and possibly also due to the diluted wastewater, as COD has also been shown to impact the hydrogen sulphide generation proportionally (Rudelle *et al.*, 2012).

Interestingly, the hydrogen sulphide pattern measured at the RM1 outlet was very similar to the pattern measured at the RM2 and RM3 outlets, which indicated that the nitrate dosing was not being as effective as intended. The nitrate dosing at the RM1 wet well was controlled based on wastewater temperature and RM1 flowrate. Therefore, it was expected to achieve a consistent hydrogen sulphide concentration at the RM1 outlet, which was originally designed to keep hydrogen sulphide below 0.1 mg/L. The fact that the hydrogen sulphide pattern was similar to RM2 and RM3, which were not dosed, suggests the nitrate is being consumed faster than what was originally expected. This phenomenon has been observed and reported by Jiang et al. (2013) at a rising main in Australia and was linked with the growth of denitrifying biofilms within the rising main once the nitrate dosing was started.

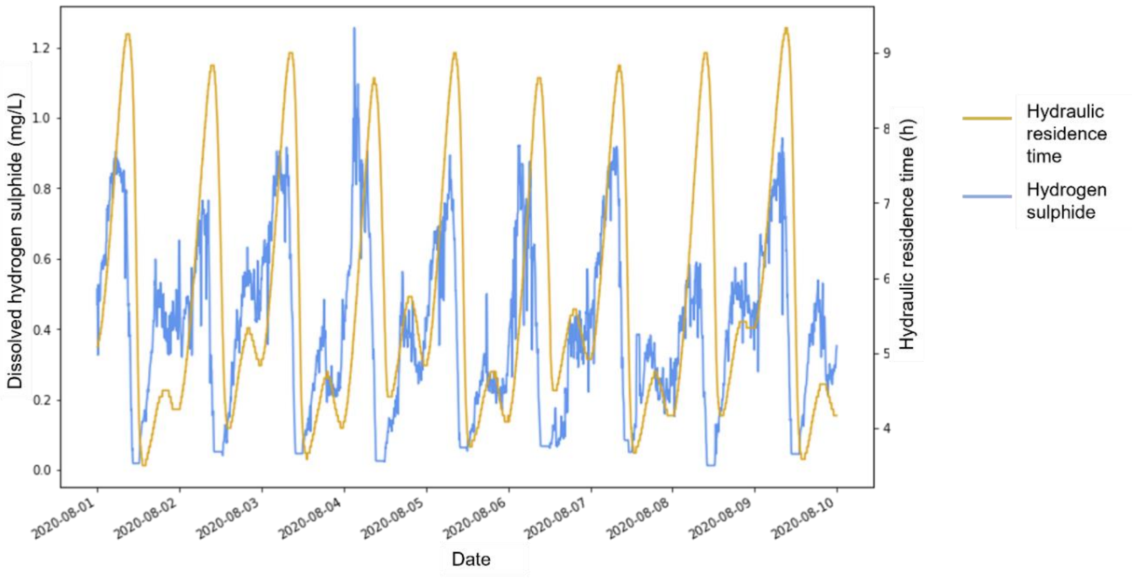

Figure 5-2 Hydrogen sulphide concentration at the outlet of RM1 and the hydraulic residence time of the wastewater at RM1 between 01-08-2020 and 10-08-2020

There were several issues with some of the sensors during the monitoring campaign. The sensor at the RM3 outlet flatlined at 0 mg/L from February onwards and was not possible to fix the issue by calibrating the sensor. The RM2 outlet sensor failed the calibration procedure in April and the data collected was therefore considered as not precise. The combined inlet sensor also stopped measuring and the signal was flatlining. Therefore, as can be seen in Figure 5-3, the data for these periods was discounted.

The average hydrogen sulphide concentrations for the RM1, RM2 and RM3 outlets and for the combined inlet of the WWTP are shown in Figure 5-3. The corrosion potential of the rising mains was assessed based on the dissolved hydrogen sulphide load, calculated as the average hydrogen sulphide concentration times the dry weather flow (DWF). As hydrogen sulphide usually shows seasonality, the assessment was divided into warm months (April – September) and cold months (November – January). In the cold months, the main source of corrosion potential was RM1, having a 3.6 kg/d hydrogen sulphide load, 21.6% higher than RM3, which had a 2.96 kg/d load and 266.8% higher than RM2, which had a 0.98 kg/d load. During the warm months, it was only possible to calculate the load of RM1, which was of 4.2 kg/d and 16% higher than in cold months. The slight increase in RM1 hydrogen sulphide from cold months to warm months suggests the nitrate dosing was definitely having an effect. Even more when considering that July and August, the warmest months, with 21.05 °C and 24.77 °C temperature respectively, had lower concentrations than the cold months. However, the mechanism of the dissolved hydrogen sulphide reduction could not be defined, as it was not possible to assess if the total dissolved sulphide was reduced or the pH of the wastewater was increased, as it has been proved that nitrate dosing results in higher wastewater pHs (Liang *et al.*, 2023). The increase in pH would justify the lower hydrogen sulphide concentrations, as the dissolved sulphide speciates in different species depending on the pH of the wastewater (Equation (5-4)) (Yongsiri *et al.*, 2004).

$$S^{2-} + 2H^{+} \xleftrightarrow{K_{a2} \approx 0.8 \cdot 10^{-17}} H^{+} + HS^{-} \xleftrightarrow{K_{a1} \approx 1.0 \cdot 10^{-7}} H_2S_{(aq)} \qquad \textbf{(5-4)}$$

Similarly, the mass balances to assess the highest sources of hydrogen sulphide to the WWTP were not possible to calculate. The pH of each rising main outlet would probably have been different and therefore, the total dissolved sulphide is unknown. When using dissolved sulphide sensors, which usually measure either the dissolved hydrogen sulphide or the dissolved bisulphide, a pH probe is also required if the total dissolved sulphide is to be calculated. This has also been recently highlighted by Despot et al. (2021) when comparing different dissolved sulphide sensors.

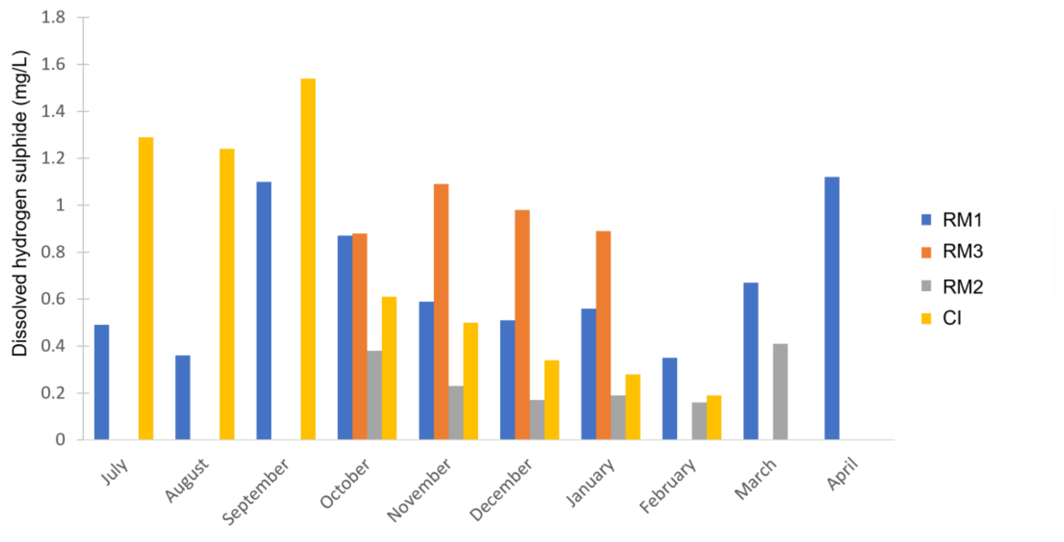

Figure 5-3 Hydrogen sulphide monthly averages at RM1, RM2 and RM3 outlets and at the combined inlet of the WWTP

5.3.2 LSTM model

As a proof of concept and to repurpose the SulfiLogger sensors to another location, a dissolved hydrogen sulphide prediction model was developed for the RM1 outlet. RM1 was selected, as it had continuous data for 9 months covering cold and warm seasons. Instead of using a mechanistic model, such as the WATS or SEWEX, it was decided to follow a data-driven approach using the LSTM model. The decision was taken as the LSTM model required the monitoring and availability of significantly fewer parameters and was expected to achieve high accuracy.

As a first step, the input parameters selected, and the dissolved hydrogen sulphide were analysed by using a correlation matrix (Figure 5-4). The parameters were shown not to have strong correlations, all being below the 0.5 correlation parameter. The parameters used for the model development had values between the ones shown in Table 5-2. The RM1 flowrate ranged from 0 to 936 m^3/hr, the temperature ranged from 0 to 29.75 °C, the time variable ranged from 0 to 1 days and the dissolved hydrogen sulphide ranged from 0 to 5 mg/L, the 5 mg/L being the upper limit of the SulfiLogger measurement range. As the model is built on the training data, it can only be applied within the limits the model development data covers.

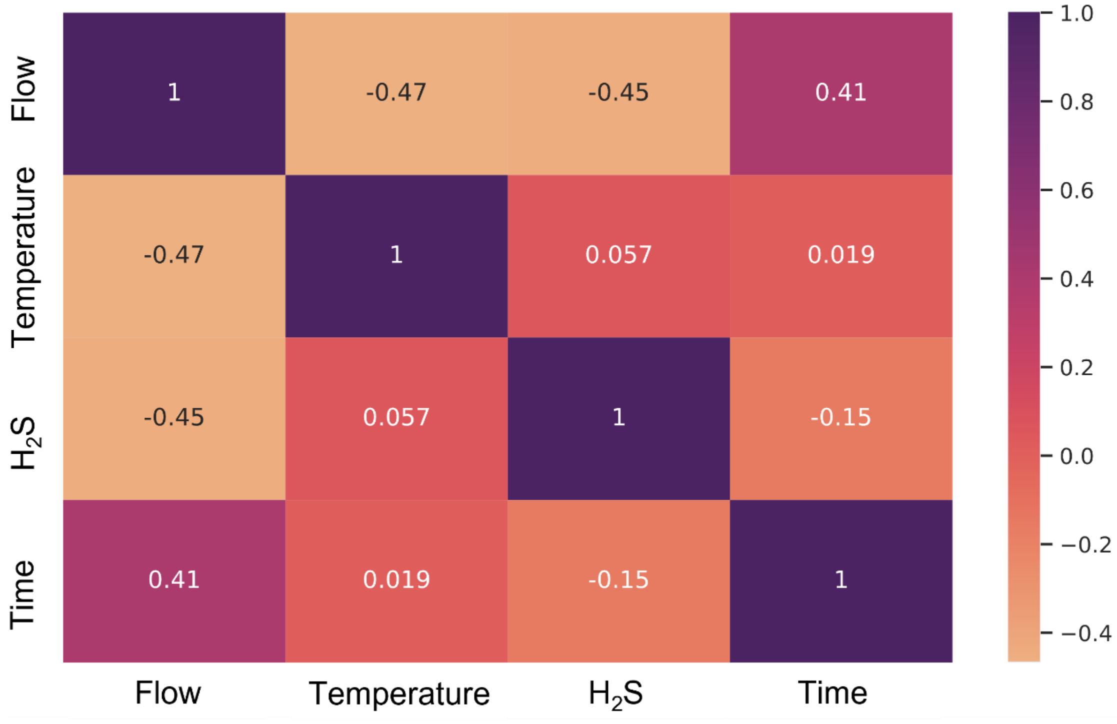

Figure 5-4 Correlation matrix of the LSTM model variables

Table 5-2 Descriptive statistics of the LSTM model variables

Parameter	Units	Average	SDEV	Min	Max
Flow	m^3/hr	514.29	204.01	0	936
Temperature	°C	13.51	6.24	0	29.75
Time	Day	0.5	0.29	0	1
H_2S	mg/L	0.65	0.53	0	5

The LSTM model was developed as described in Section 0, and the predicted against the measured dissolved hydrogen sulphide can be observed in Figure 5-5 for 4 days of the test set. As seen in Figure 5-5, the predicted hydrogen sulphide by the LSTM model follows the measured curve quite well, with the three characteristic daily variations. However, some of the high peaks are underestimated by the LSTM model and suggest these can't be well explained by the input parameters.

Also in Figure 5-5, it can be seen that the polynomial regression follows very closely the prediction of the dissolved hydrogen sulphide by the LSTM, whereas the linear regression has a worse performance. This is further confirmed by the model performance metrics in Table 5-3. The RMSE for the LSTM in the test set is the lowest at 0.34 and the NSE is the highest at 0.57, which indicates the LSTM model is the best performing model and achieves the overall closest predicted value to the measured dissolved hydrogen sulphide. The polynomial regression follows closely with an RMSE of 0.35 and an NSE of 0.54. On the other hand, the linear regression performs significantly worse with an RMSE of 0.46 and an NSE of 0.20. Adding additional LSTM layers to the LSTM model was trialled, up to 3 LSTM layers; however, although the train set RMSE was improved down to 0.30 compared to the 0.38 achieved by the final LSTM model, the test set RMSE was higher at 0.40 when using 3 LSTM layers. The phenomenon observed when increasing the LSTM layers is known as over-fitting and it is a common observation when developing data-driven models (Srivastava *et al.*, 2014).

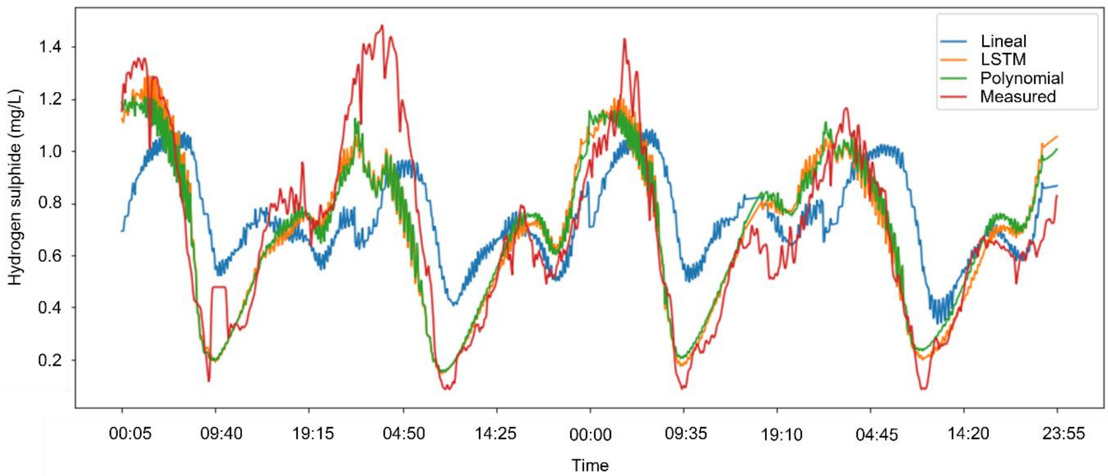

Figure 5-5 Hydrogen sulphide concentration at the outlet of RM1 and the predicted concentrations using linear regression, polynomial regression and LSTM models

Table 5-3 Model performance assessment for hydrogen sulphide prediction at the outlet of RM1 using linear regression, polynomial regression and LSTM model

	RMSE		NSE	
Model	Train	Test	Train	Test
LSTM	0.382	0.340	0.473	0.571
Linear	0.467	0.465	0.212	0.199
Polynomial	0.379	0.350	0.482	0.544

When comparing the model performance to other uses of the LSTM model, such as the one to predict flowrate to a WWTP developed by Zhang et al. (2018), achieved a much better performance with RMSE and NSE values of 0.04 and 0.94, respectively, for a 15 minute interval data. The performance difference is expected to be related to the complexity of the system being modelled. The flowrate to a WWTP relies on a few key factors that can be included in the prediction model. On the other hand, the hydrogen sulphide formation is dependant on many variables, such as the rising main physical properties, the wastewater composition and the hydraulic conditions, and in this specific case, the nitrate dose could also have been an important factor not directly considered in the model development. Overall, the LSTM model performance was deemed acceptable for the potential uses of the predictions.

Two main uses are suggested for the dissolved hydrogen sulphide prediction. The first one would be using the predicted dissolved hydrogen sulphide as an input to a septicity warning system, such as the one described in Chapter 3. The use of the warning system would be dependant on the specific WWTP, but more information is available in the paper itself. The second use would allow for a feedforward control of a hydrogen sulphide treatment chemical, such as nitrate (Mohanakrishnan *et al.*, 2009) or iron (Kiilerich *et al.*, 2017), at the prediction point, which has been shown in Australia to be more effective than dosing at the wet well of a rising main (Ganigue *et al.*, 2011).

Finally, the dissolved hydrogen sulphide monitoring is required to be of a minimum of 8-9 months, and has to capture the warmest and coldest months of the year, as it is key to cover all the range of temperatures in the training set.

Otherwise, the LSTM model would not be applicable on the extreme temperature months.

5.4 Conclusions

In this study, the dissolved hydrogen sulphide at 3 rising main outlets discharging at a WWTP inlet chamber were monitored using a novel dissolved hydrogen sulphide sensor (SulfiLogger) for 9 months. Furthermore, a hydrogen sulphide prediction LSTM model was developed for a rising main outlet using wastewater flowrate, temperature and the time of the day as input parameters.

In conclusion:

- The dissolved hydrogen sulphide profile followed a distinct daily pattern, which was the inverse of the typical daily flow pattern to the WWTP.
- The RM1 had the highest hydrogen sulphide load at 3.6 kg/d, which was 22% higher than the RM3 load and 2-fold higher than RM2 in the cold months.
- The LSTM model was able to predict the hydrogen sulphide daily pattern successfully and has the potential to be applied in sulphide treatment chemical control systems.

5.5 Acknowledgements

The authors gratefully acknowledge financial support from the Engineering and Physical Sciences Research Council (EPSRC) [grant number EP/R512515/1] through their funding of the STREAM Industrial Doctorate Centre, and from the project sponsor Thames Water.

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6 ECONOMIC ASSESSMENT OF SEPTICITY PREVENTION METHODS AT RISING MAINS DISCHARGING TO WASTEWATER TREATMENT PLANTS

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Abstract

Wastewater transport to treatment plants frequently results in septic wastewater. Septicity management is mainly achieved by chemical dosing into wet wells prior to rising mains. However, there are several management options and there are no clear guidelines on which to select. In this study, an economical assessment of septicity management was performed on a UK rising main discharging into a wastewater treatment plant (WWTP). The septicity management options assessed consisted of calcium nitrate, air and ferric sulphate dosing. The most economically favourable option was calcium nitrate dosing, with a net present value (NVP) 27% and 45% lower than for ferric sulphate and air dosing, respectively. Furthermore, the study highlighted the economic benefits of utilising dissolved hydrogen sulphide sensors for calcium nitrate dose optimisation. Finally, a further assessment was done on the dosing location for calcium nitrate and air. Dosing at a downstream section of the rising main resulted in an NPV reduction of 13% and 50% when compared to wet well dosing for calcium nitrate and air, respectively.

Keywords: Nutriox, Nitrate, SulfiLogger, Aeration, CAPEX, OPEX, NPV

6.1 Introduction

Wastewater septicity is undesirable and is directly linked with several issues at both the sewerage network and at wastewater treatment plants (WWTPs). The issues at the sewerage network are exclusively related to the generation and subsequent volatilisation of hydrogen sulphide when the wastewater is anaerobic, common at rising mains (Boon, 1995). The hydrogen sulphide gas can cause concrete corrosion and odour nuisance, as well as being toxic for humans at above 50 ppm (Park *et al.*, 2014). At the WWTPs, septicity has been shown to increase the chemical demand for chemical phosphorus removal and to increase the aeration demand due to the hydrolysis of particulate organic matter (Chapter 4). Therefore, preventing septicity from happening or treating the generated sulphide is desirable prior to the outfall of the rising mains (Zhang *et al.*, 2008).

Several septicity prevention or sulphide treatment methods are used worldwide, with chemical dosing being the most common (Ganigue *et al.*, 2011). Within chemical dosing for septicity management, calcium nitrate dosing at wet wells prior to the wastewater being pumped is the most common in the UK. However, nitrate dosing has a high associated cost, due to the high dose rates required to maintain a minimum nitrate concentration throughout the rising main, so that septicity does not develop at the final sections of the rising main (Jiang *et al.*, 2009). Other chemicals dosed to manage septicity worldwide include aeration of the wastewater and iron coagulant dosing, although these are not particularly popular in the UK.

Several studies have highlighted that nitrate dosing and aeration are more effective as sulphide treatment methods, rather than using them for maintaining anoxic or aerobic conditions, respectively (Gutierrez *et al.*, 2008; Mohanakrishnan *et al.*, 2009). These studies suggest dosing nitrate or oxygen at the final sections of the rising mains, just leaving enough time for sulphide oxidation. This sulphide control strategy had lower dose requirements while achieving similar sulphide control and therefore, reduced the operational costs (OPEX) of septicity management (Ganigue *et al.*, 2011; Vinck *et al.*, 2017). Iron coagulants, on the other hand, have been shown to partially inhibit sulphide production, up to 60% reduction, in rising main biofilms and therefore, the optimal dosing strategy is thought to be at the start of the rising main (Zhang, Keller and Yuan, 2009; Zhang *et al.*, 2012).

Owing to the several septicity management options and the lack of clear selection guidelines (Ganigue *et al.*, 2011; Vinck *et al.*, 2017), this economical assessment focused on analysing the costs and benefits of the different septicity management options at a rising main discharging at a conventional activated sludge WWTP with chemical phosphorus removal.

6.2 Materials and Methods

6.2.1 Business case scenarios

This study was based on a simplified version of an existing WWTP in the UK (Chapter 5). The WWTP had a concrete inlet chamber, which received wastewater from a 6.8 km rising main (0.6 m diameter). The dry weather flow (DWF) in the rising main was of 6,000 m^3/d with an average hydraulic retention time (HRT) of 6.4 hrs. The WWTP consisted of a conventional activated sludge (CAS) plant with ferric sulphate dosing prior to the primary settlement tanks (PSTs) for phosphorus removal (Figure 6-1).

Five scenarios were analysed, as illustrated in Figure 6-1, which were limited to dosing at the wet well, due to limitations in rising main access throughout the rising main. Scenario A was the baseline scenario, which didn't include any action for septicity control. Scenario B was the current scenario, as nitrate was being dosed at the rising main currently. Scenario C was the optimised nitrate dosing scenario, including a dissolved sulphide sensor measurement at the outlet of the rising main that informed the dosing controller. Scenario D consisted of aeration on the wet well. Scenario E consisted of dosing ferric sulphate at the wet well.

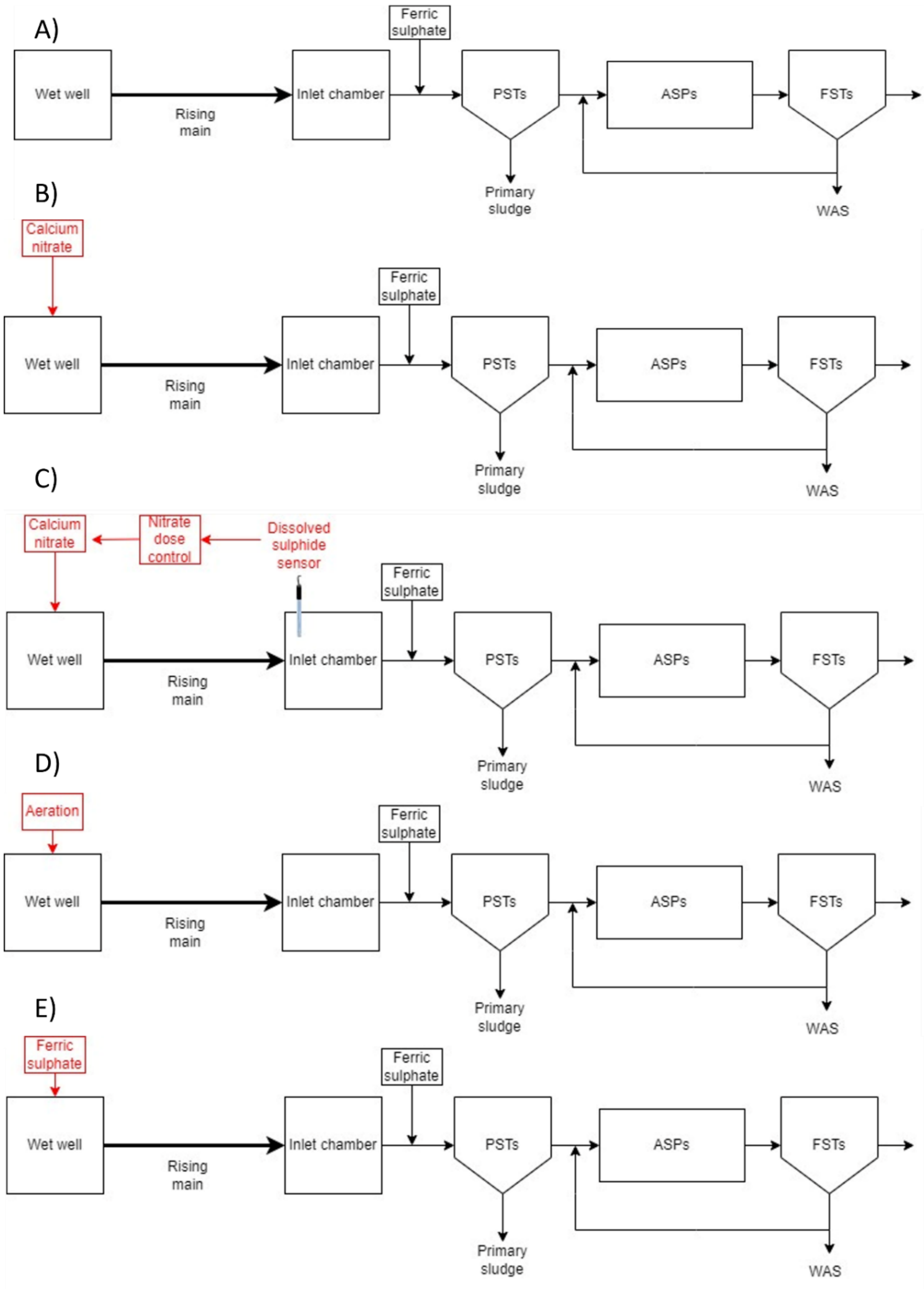

Figure 6-1 Business case scenarios, with the particular components in red

6.2.2 Design parameters

All scenarios have been validated using results within this thesis or existing literature values. Assumptions are listed in Table 6-1.

Table 6-1 Summary of assumptions

Scenario	Design assumption
General	• Chemical tankering costs were excluded, as the costs are site specific • Wet well sulphide was negligible, as measured in Chapter 5 • Influent wastewater quality had 600 mg/L COD and 8 mg/L total phosphorus • PSTs removed 40% of the influent COD • Septicity increased up to 20% the COD in the PST effluent, as a factor of the sulphide concentration (excluding iron dosing, as it doesn't prevent septicity) • The lowest sulphide concentration that can be achieved by chemical treatment is 0.2 mg S/L (Vinck *et al.*, 2017) • The ferric sulphate dosing rig for chemical phosphorus removal has enough capacity for additional dose requirements for sulphide precipitation • CAS aeration system has spare capacity for the additional COD load due to septicity • Desludging and sludge management costs excluded
Scenario C	• The dissolved sulphide sensor needs calibration every 3 months and has a lifespan of 5 years

For Scenario A (Table C-1), the sulphide concentration at the outlet of the rising main was calculated using Equation (6-1) proposed by Boon and Lister (1975). The inlet chamber replacement frequency was assumed to be of 10 years, as the sulphide concentration at the outlet of the rising main was of 5.1 mg S/L. The ferric sulphate required for phosphorus removal due to the competition with sulphide precipitation was calculated based on the results of Chapter 4, aiming to have an S:Fe molar ratio of <0.35 to have a high phosphorus removal efficiency. A standard dose of 2.5 mg Fe/mg P was used as the baseline (when no sulphide was present) (B. Martin, 2019, pers. comm, 9th May) and the additional ferric sulphate dose required was calculated as the difference. Finally, the additional COD load to be treated at the CAS was defined as 20% of the calculated PST effluent COD load (based on a 20% soluble COD increase due to septicity (Chapter 4)).

$$\boldsymbol{C_s = 0.228x10^{-3}(COD)1.07^{T-20}\frac{A}{V}t_h} \quad \textbf{(6-1)}$$

For Scenario B (Table C-2), the rising main outlet sulphide concentration was calculated using the measured dissolved hydrogen sulphide average between August 2020 and January 2021 (Chapter 5), to cover for both warm and cold months and was converted to total dissolved sulphide using Equation (5-4) with a wastewater pH of 8.39 (as the average measured in spot samples). The inlet chamber replacement frequency was calculated using a corrosion factor calculated by the dissolved sulphide difference in Scenario B and Scenario A of 2.3. The additional COD load to be treated at the CAS was defined using the same 2.3 factor between Scenario A and Scenario B dissolved sulphide. Finally, the calcium nitrate dose was defined using the average daily nutriox dose between August 2020 and January 2021.

For Scenario C (Table C-3), the rising main outlet sulphide concentration was assumed to be 0.2 mg S/L, as it is the minimum achievable by nitrate dosing (Vinck *et al.*, 2017). The inlet chamber replacement frequency was of 50 years, as it is its asset life and very low corrosion was expected. Finally, a 20% increase in the calcium nitrate dose was assumed to be enough for effective sulphide control using the dissolved hydrogen sulphide data to optimise the dosing regimen (Chapter 5).

For Scenario D (Table C-4), the time the wastewater in the rising main would remain aerobic was calculated using a maximum achievable DO when aerating of 5 mg/L (Ganigue *et al.*, 2011) and an oxygen uptake rate (OUR) of 5.94 mg/L/hr (Gutierrez *et al.*, 2008). The remaining HRT was assumed to be anaerobic and the rising main outlet sulphide concentration was calculated using Equation (6-1).

The inlet chamber replacement frequency was also calculated using the corrosion factor between Scenario A and Scenario D of 1.2. The additional ferric sulphate dose for phosphorus removal was calculated using the same method as for Scenario A. Finally, the additional COD load to be treated at the CAS was defined using the same 1.2 factor between Scenario A and Scenario D dissolved sulphide.

For Scenario E (Table C-5), the rising main outlet sulphide concentration was assumed to be 0.2 mg S/L, as for Scenario C. The ferric sulphate dose was calculated using a 0.9 Fe:S molar ratio defined by Firer, Friedler and Lahav (2008) for effective sulphide control using ferric salts. To calculate the sulphide concentration to define the ferric dose, Equation (6-1) was used. The inlet chamber replacement frequency was set at 50 years, as low corrosion was expected. Finally, the additional COD load to be treated at the CAS was defined the same as for Scenario A, due to the fact that ferric dosing does not prevent septicity (Nielsen *et al.*, 2005).

6.2.3 Economic evaluation

6.2.3.1 Capital and operational costs

The capital and operational costs (Table 6-2) were obtained from literature and water company data and were then converted to British Pound Sterling (£) and converted to 2022 prices using the Consumer Price Index (CPI) for the 2010-2022 period.

Table 6-2 Summary of capital and operational cost estimates

Parameter	Value	Unit	Notes	Reference
Capital				
Calcium nitrate dosing rig and ancillaries	78,053	£		(de Haas *et al.*, 2008)
Wet well aeration system and ancillaries	78,053	£		(de Haas *et al.*, 2008)
Ferric sulphate dosing rig and ancillaries	142,168	£		(de Haas *et al.*, 2008)
Concrete inlet chamber	500,000	£		(D. Vernon, 2019, pers. comm, 16th Oct)
Operational				
Calcium Nitrate	0.28	£/L		(D. Attwood, 2019, pers. comm, 16th Oct)
Wet well aeration	14.33	£/ML		(Ganigue *et al.*, 2011)
Ferric sulphate	0.25	£/kg		
Energy cost	0.2	£/kWh		
Dissolved sulphide sensor	6000	£	Based on SulfiLogger	(SulfiLogger, 2020)
Sensor calibration	300	£		

6.2.3.2 Net present value estimate

The Net Present Value (NPV) was calculated for a period of 20 years assuming a 7% discount rate and using Equation (6-2). The calculation was modified to show the costs over a 20 year period, as there are no revenues in the economic assessment. The NPV includes initial CAPEX and ongoing OPEX associated with chemical dosing for sulphide control, annualised inlet chamber corrosion costs (calculated using the chamber cost and the replacement frequency), the additional ferric sulphate for phosphorus removal, additional aeration for COD removal and the dissolved sulphide sensor replacement and calibration.

$$NPV = CAPEX + \sum_{t=0}^{20} \frac{OPEX}{(1+i)^t} \tag{6-2}$$

6.3 Results and Discussion

Capital costs were the same for the calcium nitrate dosing rig and the aeration equipment (Scenario B and D). On the other hand, the ferric sulphate dosing rig had an 82% higher cost due to the safety equipment (such as safety showers, eye wash station) associated. Nevertheless, the CAPEX contribution was relatively small at around 10% when compared to the contribution of the OPEX of around 90% on the NPV calculation (Table 6-3).

The lowest OPEX was achieved by Scenario C, of which 79% was related to the calcium nitrate dose. Septicity management proofed to be beneficial in all scenarios but Scenario D. The wet well aeration couldn’t manage to reduce the septicity significantly (due to the fast DO consumption at the rising main) and overall had higher OPEX than Scenario A (no septicity management). However, the OPEX did not include any odour complaint costs (the WWTP is located far from the public), which could have meant higher costs for not managing septicity at another WWTP.

Regarding the NPV, calcium nitrate dosing (Scenario B and C) has the lowest cost over the 20 year period (Table 6-3). The optimisation of the nitrate dosing using dissolved sulphide sensors has proven to be more economical, with the sensor purchase cost and calibration costs, as well as the increased calcium nitrate consumption, being offset by the reduced corrosion costs and aeration demand costs. However, the calcium nitrate optimisation (Scenario C) is only economically favourable if the nitrate dose increase required for an effective septicity control is lower than 150% of the current dose (in the analysis it was assumed it would be 120%). A dosing optimisation trial would be required to determine the dose increase requirement.

The potential co-benefits of calcium nitrate dosing, ferric dosing and aeration were also not included in the economic assessment. Nitrate dosing and aeration are known to reduce the organic load due to anoxic and aerobic respiration, respectively (Zhang *et al.*, 2008). Furthermore, ferric salts are also known to co-precipitate phosphorus, which could lead to a lower than standard ferric sulphate dose requirement at the WWTP (Gutierrez *et al.*, 2010). These factors further highlight the benefits of septicity management.

Table 6-3 Economic summary of the evaluated scenarios

Scenario	CAPEX (£k)	OPEX (£k/year)	NPV (£M)
A	0	97.3	1.1
B	78.1	63.3	0.8
C	84.1	52.7	0.69
D	78.1	102.9	1.24
E	142.2	70.7	0.94

6.3.1 Downstream sulphide control

The use of downstream sulphide control strategies has the potential to be further favourable than wet well dosing. Particularly aeration and calcium nitrate dosing at the downstream section of the rising mains has been suggested in literature (Ganigue *et al.*, 2011). The sulphide control mechanisms are very similar for both, with oxygen being able to react chemically and biologically (Gutierrez *et al.*, 2008) and nitrate only biologically (Mohanakrishnan *et al.*, 2009; Jiang, Sharma and Yuan, 2013). It is important to optimise the dosing location to avoid additional chemical dosage but also to allow enough time for the sulphide oxidation to happen (Despot, Reinhold and Barjenbruch, 2022).

A further economical assessment was done to enable a comparison of wet well dosing versus downstream dosing for nitrate and aeration (Table 6-4). The nitrate dose was calculated based on the reported dose requirement of 0.045 L/m^3 by Despot, Reinhold and Barjenbruch (2022) (Table C-6). They found a 5-fold reduction in nitrate dosing required when comparing wet well dosing with downstream dosing. For the rising main in this study, the dose reduction was lower at 80% of the optimised dose at the wet well. This is likely due to the diameter of the rising main, as in this study the diameter was of 0.6 m and on Despot, Reinhold and Barjenbruch (2022) was of 0.2 m, and it has been demonstrated that nitrate consumption mainly happens in the biofilms (Jiang, Sharma and Yuan, 2013). The aeration requirements and installation costs were assumed to be the same for the downstream assessment as for the wet well assessment (Table C-7).

The assessment clearly shows that downstream dosing (if access and land are available) is more economically feasible, with cost reductions of 13% and 50% for nitrate and aeration, respectively.

Table 6-4 Economic comparison of wet well dosing and downstream dosing using air and calcium nitrate

Location/chemical	CAPEX (£k)	OPEX (£k/year)	NPV (£M)
Wet well nitrate	84.1	52.7	0.69
Downstream nitrate	84.1	44.3	0.6
Wet well aeration	78.1	102.9	1.24
Downstream aeration	78.1	47.7	0.62

6.4 Conclusions

In this economic assessment, three septicity management methods were assessed to be applied on the wet well of the pumping station discharging to a WWTP. The management methods included ferric sulphate dosing, calcium nitrate dosing and aeration. Furthermore, the dosing location of nitrate and air was compared including wet well dosing and downstream dosing.

The findings were:

- Septicity management was cost-effective and the most feasible chemical for wet well dosing was nitrate

- Dissolved sulphide sensors for dosing optimisation offer an economical benefit
- Downstream dosing of both nitrate and air is more cost-efficient than wet well dosing, with cost reductions of 13% and 50%, respectively

6.5 Acknowledgements

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6.6 References

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7 DISCUSSION

The overall aim of the thesis was to understand the mechanisms governing septicity in wastewater and mitigate the impacts both in sewers and at the wastewater treatment plant.

7.1 Impacts of septicity on wastewater treatment plants

Chapter 2 highlighted the main gaps in knowledge on the impacts of septicity on WWTPs. The focus on WWTP impact of septicity was exclusively on the impact of sulphide on the different processes, such as CAS, EBPR or AGS. Furthermore, the research available was mainly lab-scale experiments, utilising synthetic wastewater and adding sulphide as a chemical (Chapter 2).

The experiments in Chapter 4 utilised real municipal wastewater to see if the impacts reported in literature using synthetic wastewater were applicable. For chemical phosphorus removal, competition between sulphide and phosphate with ferric for precipitation was observed at sulphide levels relevant at WWTPs (3 mg S/L sulphide). At an S:Fe molar ratio >0.35 a reduction in phosphorus removal efficiency was observed, which would require a higher chemical dose to achieve the required phosphorus permit levels. The dissolved sulphide was suspected to be precipitated with ferric forming ferrous sulphide (FeS) as an intense black colour formed immediately after adding the ferric to the sulphide-containing wastewater. The formation of FeS when iron and sulphide are available in wastewater was observed by Firer, Friedler and Lahav (2008) when dosing ferric for sulphide removal and by Wilfert *et al.* (2020) when dosing sulphide to recover phosphate from iron-rich wastewater sludge.

The WWTP pilot plant experiments also showed that low levels of septicity at low OLRs do not pose a risk to final effluent quality or process operation. However, increasing the OLR from 0.25 kg COD/m^3/d to 0.51 kg COD/m^3/d reduced the nitrification efficiency of the septic fed ASP from 91% to 80%, and was further reduced to 46% when the influent sulphide concentration was increased from 1.5 mg/L to 6.4 mg/L. The respirometry tests showed that directly due to the septic wastewater. Similar results of direct inhibition of nitrification had been previously reported (Bejarano-Ortiz *et al.*, 2015; Delgado-Vela, Dick and Love, 2018).

Most of the literature research focused on a specific treatment process rather than understanding the interactions between the treatment processes and the septicity in the raw wastewater. Chapter 2 further highlighted differences on the potential impacts of septic raw wastewater depending on the WWTP configuration and processes. To date, no study or approach has been found that investigates the interaction between processes for septic wastewater. Two approaches are thought feasible; i) full scale sampling and assessment of a WWTP with septicity issues and comparison with a "standard" performing WWTP with the same configuration. ii) Mechanistic modelling of the different septicity related interactions and integration to a WWTP modelling package (such as BioWin or GPS-X).

7.2 Septicity monitoring

Septicity has been traditionally only related to sulphide and it was likely due to the focus on corrosion and odour that is exclusive to sulphide (Boon, Vincent and Boon, 1998). However, with the shift in focus to a more holistic system where sewerage networks and WWTPs are integrated, covering the septicity state with only sulphide is not feasible. The aim of Chapter 3 was to develop a more inclusive and real parameter referring to septicity that could be practical to use. Using common wastewater parameters, a wastewater septicity value ranging from 0 to 4 was developed. However, some of the parameters are not easy or practical to measure, as is the case of sulphide, sCOD and ammonia. Ammonia and sCOD have the lowest weights on the septicity value, 6 and 11, respectively. Therefore, it is possible to take both measurements out of the septicity measure requirements by recalculating the scale using only sulphide, ORP and pH. On the other hand, sulphide is the most important parameter in the septicity value with a weight of 39, meaning that it needs to be used.

The more advanced use of the septicity value would be using it in real-time to inform WWTP site managers and operators on septicity levels coming into the WWTP and setting up actions or alarms accordingly. For that, the septicity indicators that form the septicity value need to be measured in real time. ORP and pH sensors are available and well developed for wastewater measurement. On the other hand, sulphide sensors are far from established, with only very few commercial dissolved sulphide sensors in the market (Sutherland-Stacey *et al.*, 2008; Despot, Pacheco Fernandez and Barjenbruch, 2021). Furthermore, the reliability and accuracy, as well as the installation requirements and the maintenance regime are not well established. Chapter 5 assessed the usefulness of a novel dissolved sulphide sensor at wet wells, at the outlet of rising mains and at an inlet to the WWTP. The data gathered gave great insights into septicity dynamics, and nitrate dosing efficiency and showed the potential to be integrated into an online system. However, the sensor reliability was far from optimal, with 50% of the sensors failing during the 9-month trial. Therefore, further improvements are required in the dissolved sulphide sensors for a useful and economically feasible large-scale deployment.

7.3 Septicity management

Chapter 6 assessed the economic viability of the most popular chemical control methods for septicity. Nitrate dosing at the wet well with optimised control using a dissolved sulphide sensor showed to have the lowest NPV. However, moving the dosing point to the downstream section of the rising main showed to have an even lower cost, as shown in other studies as well (Gutierrez, Sutherland-Stacey and Yuan, 2010; Ganigue *et al.*, 2011; Despot, Reinhold and Barjenbruch, 2022). This showed that assessing the dosing location is also key, not only the potential chemicals to be used.

Optimisation of the doses by utilising a dissolved sulphide sensor was shown to be preferable, as the cost of the sensor and the maintenance were significantly lower than the cost of the chemical savings. This approach was recently followed by Despot, Reinhold and Barjenbruch (2022) when designing nitrate dosing at a rising main.

Other methods of septicity management have been suggested in literature and could be interesting for specific applications (Zhang *et al.*, 2008; Talaiekhozani *et al.*, 2016). The use of corrosion-resistant material could be applied in sewers, although septicity would still develop. This approach could well be used for EBPR plants, as the VFA accumulation during septicity could be beneficial (Ashley *et al.*, 2002), although odour and toxicity problems would still occur. Another suggested method is inhibiting the SRB activity in the biofilms by dosing free nitrous acid (FNA) (Jiang, Gutierrez and Yuan, 2011; Gao *et al.*, 2016). This method would be even better than corrosion-resistant materials for EBPR WWTPs, as it would allow for VFA accumulation without significant sulphide generation.

Clearly, there is no "magical" solution for septicity management, with most solutions having to be selected based on specific site conditions and costs. Trials to generalise management solutions were made by Vinck *et al.* (2017), but there is still uncertainty on whether those would apply on a case by case basis.

7.4 Contribution to knowledge

Overall, this thesis has contributed to the integration of the sewerage network and the WWTP, with the development of a septicity indicator and the quantification of the downstream impacts at a conventional WWTP. Furthermore, the septicity dynamics at a rising main have been measured using a novel dissolved sulphide sensor and the ability to predict sulphide concentrations at the outlet of a rising main based on readily available data has been demonstrated. The contribution to knowledge of this EngD is summarised in Table 7-1.

Table 7-1 Contribution to knowledge of the EngD thesis

	What has been confirmed?	What has advanced knowledge?
Theoretical knowledge	• Filamentous microorganisms proliferate when a septic influent is fed to an activated sludge plant, leading to high SVIs and biomass loss (Chapter 4) • The development of septicity in the liquid phase was demonstrated in the incubation experiments and lower rates than those reported for sewers containing biofilms were measured (Chapter 3)	• The main indicators of septicity have been defined, along with their contribution to the overall septicity parameter (Chapter 3)
Empirical evidence	• Septic raw wastewater reduces the efficiency of nitrification in activated sludge plants (Chapter 4)	• Chemical phosphorus removal efficiency using ferric chloride is reduced when the wastewater is septic at S:Fe molar ratios higher than 0.35, requiring increased doses (Chapter 4) • Moderate septicity at activated sludge WWTPs with low organic loadings of 0.25 kg COD/m^3/d did not have any noticeable impacts on performance
Methodology	• Dissolved sulphide measurement is complicated and not very precise. The sample has to be measured immediately and avoid contact with air (Chapter 3)	• A novel methodology to assess wastewater septicity has been developed that would allow for international research to be more comparable in the septicity literature (Chapter 2)

		• The use of data-driven models is able to predict dissolved sulphide at the outlet of rising mains using readily available data (wastewater flow, temperature and time of day), with only dissolved sulphide monitoring required to develop the model (Chapter 5) • Coupling a pH sensor when measuring dissolved sulphide with any of the commercially available sensors is critical, as the total dissolved sulphide is the key parameter to be able to calculate mass balances and track the source of sulphide (Chapter 5) • When performing economic assessments of septicity management options, it is critical to include downstream impacts at the WWTP and not only focus on corrosion issues (Chapter 6)

7.5 Future work

- Integration of septicity related impacts, such as sulphide/phosphate competition for precipitation with ferric, nitrification inhibition by sulphide and proliferation of filamentous bacteria into a WWTP model (BioWin, GPS-X)
- Assessment of the impacts of septicity on an EBPR plant using a pilot scale plant and then potentially applying at a full-scale WWTP if the conditions promote EBPR (the expected impact would be similar to the installation of a sidestream fermenter)
- The study of particle size distribution change during septicity to assess the impact septicity has on primary settlement.
- The development of an automated action framework for WWTP management using online monitoring of the key indicators of septicity (Chapter 3) based on the impacts observed at WWTPs (Chapters 2 and 4)
- Further expansion of the septicity measurement proposed in Chapter 2 with samples collected at rising mains, to increase the range of the indicator parameters and increase the confidence in the validity of the scale
- Development of a nitrate dose controller based on a machine learning algorithm, using the dissolved sulphide measurements at the outlet of the rising main and trialling it at full-scale to measure the cost-benefits of the application of an advanced control mechanism
- Assessment of the sulphide mitigation capacity and economic viability of nitrate dosing at downstream sections of the rising main and comparison with wet well dosing

7.6 References

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8 CONCLUSIONS

The overall conclusions in reference to the original objectives presented in Chapter 1 are detailed below.

Objective 1. To provide a state of the art review on the impacts of septicity on wastewater treatment processes and identify gaps in knowledge

- Raw wastewater septicity was found to impact most of the processes in a WWTP.
- The impact on activated sludge as a single process unit had been extensively studied but only at lab scale and using synthetic sewage.
- The impact of wastewater septicity on a WWTP was found to be highly dependent on the WWTP configuration and the treatment processes involved.
- Beneficial impacts of wastewater septicity were found for BNR processes.
- Several research gaps were found, including the lack of understanding of the change of particle size distribution during septicity development and the impact it has on PST performance, the impact of sulphide on the EBPR cycle at high sulphide concentrations and acclimatation of activated sludge to septic wastewaters.

Objective 2. To identify the key indicators of septicity and develop a scale of septicity for raw wastewater

- The key indicators of septicity were found to be sulphide, ORP, pH, soluble COD and ammonia, in order of importance.
- Wastewater septicity was also found to develop in the bulk liquid without the need of biofilm, although the rates were lower than those reported for rising mains with biofilms.
- The septicity measurement had 4 levels, which could be used as an alarm system for WWTP site managers and operators.
- The most important indicators, i.e. sulphide, ORP and pH can be measured in real-time, allowing for an automated framework of actions to be taken depending on the septicity level coming into a WWTP.

Objective 3. To investigate the impacts of septicity on wastewater treatment processes, including primary settling, chemical phosphorus removal and activated sludge processes and identify risks to utilities and operators

- The impact of septicity on primary settling performance was inconclusive due to the feed wastewater mixing arrangements.
- In an activated sludge process, COD removal and nitrification were directly impacted by the septicity in the feed wastewater, with the COD and nitrification efficiency being reduced by 55% and 44% when the process was fed with septic wastewater containing 6.4 mg/L sulphide.
- Treating septic wastewater containing 6.4 mg/L of sulphide resulted in the proliferation of filamentous bacteria and activated sludge bulking that led to biomass washout.
- The efficiency of chemical phosphorus removal was negatively impacted at S:Fe molar ratios higher than 0.35, reaching a minimum 10% removal rate at an S:Fe molar ratio of 1.4.
- High sulphide concentrations of over 5 mg/L have been observed at a WWTP influent (Chapter 5). The performance of the phosphorus removal using ferric dosing would be reduced, and if the dose was not automatically adjusted (not common), the WWTP has the potential to have a sample breach.

Objective 4. To understand the potential of dissolved sulphide sensors to monitor septicity in real-time and evaluate septicity management options such as dosing of nitrate in sewers

- The SulfiLogger sensor provided continuous measurement of dissolved sulphide, which provided daily profiles previously unknown at the site.
- Dissolved sulphide measurement needs to have a pH sensor coupled to allow for total dissolved sulphide calculations and a mass balance calculation to track the sulphide source at a site with many rising mains discharging.
- The SulfiLogger sensor has the potential to help improve and optimise chemical dosing for septicity management.

- The calcium nitrate dosing was found relatively ineffective at the design dose applied for the rising main and it was highlighted that adjusting the dose profile and dose rate according to the dissolved sulphide measurements would be beneficial for both a better septicity control and for lowering the chemical dose.

Objective 5. To develop a sulphide prediction model based on readily available data for a rising main

- Readily available and inexpensive to gather data was possible to use for the sulphide prediction using data-driven models, rather than using a mechanistic model, which would require a much more expensive and intense sampling campaign to calibrate and validate the model.
- The LSTM model was able to predict the hydrogen sulphide daily pattern successfully and has the potential to be applied in sulphide treatment chemical control systems.
- For a data-driven model development, a monitoring period for the calibration dataset of at least 6 months is required to capture the seasonality of septicity development, both capturing the warm months and the cold months.
- The prediction model would allow for the dissolved sulphide sensor to be reused at another location while still providing sulphide concentration estimates coming into the WWTP.

Objective 6. To develop an economical assessment of the potential septicity management options, quantifying the cost-benefits against a base case

- Septicity management was cost-effective and the most feasible chemical for wet well dosing was nitrate.
- Dissolved sulphide sensors for dosing optimisation offer an economical benefit.
- Downstream dosing of both nitrate and air is more cost efficient than wet well dosing, with cost reductions of 13% and 50%, respectively.

- Considering not only the corrosion costs, but also the WWTP inefficiencies is critical when assessing the economic viability of potential septicity management options.

APPENDICES

Appendix A Septicity scale questionnaire

1. Indicate the years of experience you have working with septicity related issues:

Years of experience:	

2. Based on your experience and knowledge on the matter of wastewater septicity, assign points to the indicators shown below considering how important each of them is when determining if a wastewater sample is septic or not (*you have a total of 100 points and you must use all of them*)

Indicator	**Points** (100 in total)
Soluble Chemical Oxygen Demand (sCOD)	
Oxidation-Reduction Potential (ORP)	
pH	
Sulfide	
Ammonia	

3. Based on the indicator values shown, assign a septicity level from 1 to 4, 1 being low septicity and 4 being very septic, to the following wastewater samples.

Sample	**ORP (mV)**	**pH**	**Soluble COD (mg/L)**	**Ammonia (mg N/L)**	**Total sulfide (mg S/L)**	**Septicity level**
1	-162	7.3	236	71	0.4	

2	-254	7.0	101	40	8.4	
3	-191	7.2	90	48	5.1	
4	-222	7.7	107	47	5.2	
5	-258	7.1	50	36	7.5	

4. Indicate how confident you are on the answers provided in this questionnaire:

Not confident	
Somewhat confident	
Confident	
Very confident	

Appendix B Septicity scale questionnaire results

Response	Experience (years)	Country	Weighting					Septicity level					Confidence
			sCOD	ORP	pH	Sulphide	Ammonia	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	
1	3-5	UK	11	54	24	1	10	1	2	3	2	4	Confident
2	>10	UK	15	20	10	30	25	1	3	2	3	4	Somewhat confident
3	5-10	UK	10	30	20	30	10	1	4	3	3	4	Somewhat confident
4	>10	UK	20	30	20	30	0	1	4	2	3	3	Somewhat confident
5	>10	UK	20	25	15	40	0	1	4	2	2	3	Confident
6	>10	UK	20	20	20	25	15	1	4	3	2	3	Confident
7	>10	UK	5	30	30	30	5	1	4	3	3	3	Somewhat confident
8	>10	UK	10	30	15	40	5	1	4	2	3	4	Confident

9	5-10	UK	10	10	10	50	20	2	4	3	3	4	Somewhat confident
10	>10	Australia	0	20	40	40	0	1	4	2	1	3	Confident
11	>10	Australia	20	10	20	50	0	1	4	2	1	3	Confident
12	>10	Denmark	0	10	0	90	0	1	4	4	4	4	Very confident
13	5-10	UK	0	50	0	50	0	1	3	2	2	3	Confident
14	>10	Australia	10	30	20	40	0	2	4	4	3	4	Confident

Appendix C Design parameters for economic assessment of different septicity control scenarios

Table C-1 Main design parameters for Scenario A

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	5.1	mg S/L	Calculated using empirical equation	(Boon and Lister, 1975)
Inlet chamber replacement frequency	10	years	High concrete corrosion	
Additional ferric sulphate for P removal				
Fe:P	0.7	mg Fe/mg Total P	Calculated to achieve a S:Fe molar ratio <0.35	Chapter 4
Ferric sulphate strength	12.5	%Fe		
CAS aeration for COD removal				
Additional COD load	518	kg/d	Calculated assuming 600 mg/L influent COD and 40% removal at PST. Additional COD of 20% in the PST effluent due to septicity	Chapter 2
COD aeration requirement	0.49	kWh/kg COD		(Longo *et al.*, 2019)

Table C-2 Main design parameters for Scenario B

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Nitrate dosing rig				
Calcium nitrate dose	338.6	L/d	Current dosage	Chapter 5
Storage tank	10	m^3	30 days storage	
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	2.15	mg S/L	Calculated using pH of 7.39 (spot samples average)	Chapter 5 and (Hvitved-Jacobsen, Vollertsen and Nielsen, 2013)
Inlet chamber replacement frequency	23	years	Calculated using dissolved sulphide factor to Scenario A	
CAS aeration for COD removal				
Additional COD load	219	kg/d	Calculated assuming 600 mg/L influent COD and 40% removal at PST. Additional COD of 8.3% in the PST effluent due to septicity	Chapter 2
COD aeration requirement	0.49	kWh/kg COD		(Longo *et al.*, 2019)

Table C-3 Main design parameters for Scenario C

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Nitrate dosing rig				
Calcium nitrate dose	406.3	L/d	Assumed current dosage + 20%	
Storage tank	12	m^3	30 days storage	
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	0.2	mg S/L	Minimum achievable	(Vinck *et al.*, 2017)
Inlet chamber replacement frequency	50	years	Design life of asset	
Dissolved sulphide sensor				
Lifetime	5	year	SulfiLogger information	(SulfiLogger, 2020)
Calibration frequency	4	$year^{-1}$	SulfiLogger information	(SulfiLogger, 2020)

Table C-4 Main design parameters for Scenario D

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Wet well aeration				
Achievable dissolved oxygen	5	mg/L		(Ganigue *et al.*, 2011)
Rising main oxygen uptake rate	5.94	mg/L/hr	Calculated for A/V 6.6 m^{-1}	(Gutierrez *et al.*, 2008)
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	4.3	mg S/L	Empirical equation using anaerobic HRT	(Boon and Lister, 1975)
Inlet chamber replacement frequency	12	years	Calculated using dissolved sulphide factor to Scenario A	
Additional ferric sulphate for P removal				
Fe:P	0.19	mg Fe/mg Total P	Calculated to achieve a S:Fe molar ratio <0.35	Chapter 4
Ferric sulphate strength	12.5	%Fe		
CAS aeration for COD removal				
Additional COD load	438	kg/d	Calculated assuming 600 mg/L influent COD and 40% removal at PST. Additional COD up to 17% in the PST effluent due to septicity	Chapter 2
COD aeration requirement	0.49	kWh/kg COD		(Longo *et al.*, 2019)

Table C-5 Main design parameters for Scenario E

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Wet well ferric dose				
Fe:S	0.9		Molar ratio for sulphide control	(Firer, Friedler and Lahav, 2008)
Storage tank	9	m^3	Storage for 30 days	
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	0.2	mg S/L	Minimum achievable	(Vinck *et al.*, 2017)
Inlet chamber replacement frequency	50	years	Design life of asset	
CAS aeration for COD removal				
Additional COD load	518	kg/d	Calculated assuming 600 mg/L influent COD and 40% removal at PST. Additional COD of 20% in the PST effluent due to septicity	Chapter 2
COD aeration requirement	0.49	kWh/kg COD		(Longo *et al.*, 2019)

Table C-6 Main design parameters for downstream calcium nitrate dosing

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Nitrate dosing rig				
Calcium nitrate dose	0.045	L/m^3		(Despot, Reinhold and Barjenbruch, 2022)
Storage tank	10	m^3	30 days storage	
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	0.2	mg S/L	Minimum achievable	(Vinck *et al.*, 2017)
Inlet chamber replacement frequency	50	years	Design life of asset	
Dissolved sulphide sensor				
Lifetime	5	year	SulfiLogger information	(SulfiLogger, 2020)
Calibration frequency	4	$year^{-1}$	SulfiLogger information	(SulfiLogger, 2020)

Table C-7 Main design parameters for downstream aeration

Design parameter	Value	Units	Notes	Reference
Average flow	7,200	m^3/d	1.2 factor to DWF	
Downstream aeration				
Achievable dissolved oxygen	5	mg/L		(Ganigue *et al.*, 2011)
Rising main oxygen uptake rate	5.94	mg/L/hr	Calculated for A/V 6.6 m^{-1}	(Gutierrez *et al.*, 2008)
Inlet chamber corrosion				
Rising main outlet dissolved sulphide	0.2	mg S/L	Minimum achievable	(Vinck *et al.*, 2017)
Inlet chamber replacement frequency	50	years	Design life of asset	