Cranfield University

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# Tertiary Nitrification of Wastewater

# in Trickling Filters

# Cranfield Biotechnology Centre

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## TERTIARY NITRIFICATION OF WASTEWATER

# IN TRICKLING FILTERS

Supervisors: Dr A.D. Wheatley (1989-1991)

Prof. D.E. Brown (1991 onwards)

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# Abstract

This study was designed to investigate, at laboratory and pilot scale, the effects of various loading and climatic factors on the nitrification performance of four media, and to undertake a comparative assessment of the media. The media used were blast furnace slag and three random plastic media: Flocor RS, Etapak 160 and a new medium, Etapak



Laboratory experiments using pure cultures of *Nitrosomonas europaea* have determined the effect on nitrification of temperature, pH and substrate, BOD and inhibitor concentrations. Optimal values have been resolved for the temperature and pH and halfsaturation constants for the substrate and inhibition are calculated for pure culture conditions. The presence of glucose and glutamic acid has been shown to have a beneficial effect on nitrification, although this observation could not be adequately explained.

representation of a nitrifying trickling filter by using effluent from Cranfield STW in filters exposed to the full climatic variation. Data obtained from these filters have indicated the superior performance of the blast furnace slag media due mostly to its ability to maintain a large active bacterial population without excessive accumulation. Results have been used to support or challenge previous publications, and to generate a set of design curves.

Conclusions from this research have been used in the design of a full scale nitrifying trickling filter which has been constructed at Cranfield STW, early data from which are

**i** 

Pilot scale research, conducted over a two year period, has provided an accurate



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# Nomenclature,

A Specific Surface Area  $(m^2.m^{-3})$ 

- C Constant relating retention time to hydraulic (dimensionless) loading
- $C'$  Constant relating hydraulic load and specific  $(m^{-1})$ surface area to retention time  $(= C.A<sup>m</sup>)$





 $k$  Reaction rate constant (time<sup>-1</sup>)





- $k_T$  Temperature coefficient (°C<sup>-1</sup>)
	-
- Ammonia volumetric loading rate  $(g.m^{-3}.d^{-1})$

<u> 1989 - Andrew Marie Barbert e Barbert e</u>

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- 
- m Constant relating surface area to retention time (dimensionless)

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- N Volumetric rate of nitrification  $(g.m^{-3}.d^{-1})$
- $N$  Substrate flux  $(mg.cm^{-3}.sec^{-1})$
- n Constant relating hydraulic load to retention time (dimensionless)
- $Q$  Hydraulic loading rate (m<sup>3</sup>.m<sup>-3</sup>.d<sup>-1</sup>)

 $Q \qquad \qquad \text{Flow} \qquad \qquad (m^3.d^{-1})$ 



- t Time or Retention time (time)
	-
- V Voidage (volume of spaces as a percentage of total  $(\%)$ volume)
- $V_{\text{Max}}$  Maximum specific reaction rate (Michaelis (time<sup>-1</sup>) kinetics)
- $\nu$  Specific reaction rate (Michaelis kinetics) (time<sup>-1</sup>)
- $x$  Biofilm thickness ( $\mu$ m)
	- Y Yield coefficient for cell growth (dimensionless)

 $\mu$  Specific growth rate (Monod kinetics) (time<sup>-1</sup>)  $\mu_{\text{Max}}$  Maximum specific growth rate (Monod kinetics) (time<sup>-1</sup>) 0 Temperature correction coefficient (Schultze, 1960) (dimensionless)

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# Abbreviations

- ATU Allylthiourea (a toxic inhibitor of nitrification)
- BOD<sub>5</sub> Biochemical oxygen demand (measured over 5 days with ATU as nitrification inhibitor)
- DO Dissolved oxygen concentration
- MVF Moisture volume fraction (% v/v of water measured by neutron probe)
- NH<sub>4</sub>-N Ammoniacal nitrogen (nitrogen present as ammonia or ammonium)
- $NO<sub>3</sub>-N$  Nitrate nitrogen (nitrogen present as nitrate)
- NTU Nephelometric turbidity unit (Section 3.6.5)
- SS Suspended solids (Section 3.6.4)
- STW Sewage treatment works
- TS Total solids (Section 3.7.2)

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# Chapter 1: Introduction

During the last century, considerable improvements have been made in river water quality throughout the UK. The initial improvements were made in the BOD and solids discharges from wastewater treatment works, which had previously turned many rivers dark, odourous and incapable of supporting aquatic life. As the quality has improved, attention has switched to the control of ammonia discharges. Pollution by ammonia is

not as visually evident as BOD and solids pollution, but ammonia is toxic to aquatic life and its presence was preventing the recolonisation of rivers.

The major source of ammonia in wastewater is the cleavage of urea, a nitrogen-rich excretory product of animals, by the enzyme urease, which is produced by many of the micro-organisms present in sewage. Ammonia is also produced by the anaerobic degradation of proteins, which may occur when sewage turns septic and during anaerobic wastewater treatment processes. Other sources of ammonia include various industries: petrochemicals, pharmaceuticals, agrochemicals and paper.

Nitrifying trickling filters are not a new process; indeed many under-loaded wastewater treatment works constructed at the beginning of the century still achieve very effective

Most wastewater treatment works were constructed in the early part of the 20th century and were designed principally to remove BOD and solids. Ammonia removal, or nitrification, occurs subsequent to BOD removal in the treatment process. Consequently, additional treatment stages are required to achieve nitrification. There are a large number of small wastewater treatment works, most of which are based on the trickling filter process, which now require uprating for nitrification.

nitrification in the BOD-removal filters. However, the need has arisen to uprate a large number of filter works. A need was perceived for further research into the process of nitrification in trickling filters. The research was to encompass the effects of the hydraulic and pollution loading, the media type and configuration, the filter design and  $\frac{1}{2}$  $\frac{1}{\sqrt{2}}$ climatic effects on the performance of nitrifying filters. The data thus obtained may then

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be used in the design of full scale nitrifying trickling filters for other small wastewater treatment works.

Three fields of research are carried out:

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- Laboratory scale to study the pure-culture behaviour of nitrifying organisms under controlled conditions.
- Pilot scale, utilising a real wastewater under truly representative conditions to

determine the combined effects of loading, climate and support media on the mixed-culture behaviour of nitrifiers.

Full scale to validate the pilot scale results and to evaluate the performance of a full scale nitrifying trickling filter.

Plastic filter media were first produced in the 1960's and have since found uses for carbonaceous oxidation, air saturation and odour scrubbing, where their low density, high specific surface area and high voidage confer advantages over the more traditional mineral media. Manufacturers of plastic media are now proposing their products as media for nitrification, where the high specific surface area is perceived as a particular

advantage. A secondary objective of this research is to evaluate three random plastic media: Etapak 160 and 210, and Flocor RS.

# Chapter 2: Survey of the Literature

#### 2.1 Trickling filters.

Trickling filters, or percolating filters, are the most common fixed-film wastewater treatment process. The filter contains a solid support medium, through which the applied waste water flows. Bacteria and fungi develop within the filter and attach themselves to

The ecology of trickling filters has been extensively studied (Curds & Hawkes, 1975; Hawkes, 1963; Pike, 1978; Särner, 1980; Solbé, 1975; Tomlinson & Hall, 1955;

the support medium using an extracellular polysaccharide matrix and so form a biofilm. The open nature of the support medium enables the diffusion or convection of air through the filter to provide oxygen.

Nutrients are removed from the waste water by the bacteria and fungi in the biofilm, and the system is maintained at equilibrium by the loss of biomass through periodic sloughing of the biofilm and grazing by higher organisms.

Wheatley, 1976). In addition to the microorganisms previously mentioned, photosynthetic bacteria and algae may be found on the surface of filters. Ciliated protozoa feed on freely suspended bacteria and particles in the waste, thereby clarifying the effluent. Grazing fauna are represented successively by rotifers, nematodes, annelids, and by the larvae and adults of various insect species including Psychoda, Chironomida, Anisopus, beetles and springtails. It is believed that the development of a stable ecological habitat within a filter takes at least two years, although the maximal nutrient removal may be achieved much earlier (Wheatley, 1976).

#### , 2.1.1 The development of trickling filters

The earliest known method of wastewater treatment involved the channelling of effluent

onto the land during Roman times, where the soil acted as a simple trickling filter.

However this approach requires large areas of land, and as civilisation developed it became impractical to provide the sanitary standards demanded in the cities by this method. Work was therefore carried out at the end of the  $19<sup>th</sup>$  century to improve the efficiency of the process and to determine which type of soil is most suitable. Research at the St. Lawrence Experimental Station, Massachusetts in 1890 (Stanbridge, 1954) concluded that gravel and stone beds could accept higher rates of wastewater application than any soil type. This led to the construction of the first purpose-built large scale

Conventional trickling filters are single pass units containing rock medium of 50-100 mm diameter to a depth of 1.8 m. Filter beds can be circular or rectangular, with either rotating or travelling arms for the distribution of settled wastewater onto the surface. Variations on this basic design are described later.

trickling filter beds at Salford sewage works in 1893. The process featured the application of wastewater by spraying onto the medium surface. The drainage system and medium voidage permitted efficient ventilation of the bed.

# $2.1.2$  Trickling filter design

Trickling filters have been most widely used at small to medium-sized sewage treatment works due to their low operating costs, minimal maintenance requirements and ability to cope with highly variable loadings. The disadvantages of trickling filters are that they have a greater space requirement than some processes (notably rotating biological contactors and submerged aerated biofilters), and they can cause nuisance due to the large numbers of insects which can emerge from filters during spring and autumn (Southgate, 1964).

Trickling filters can be operated at low, intermediate or high rate, or as a roughing filter,

4

according to the loadings tabulated below.

# Table 2.1: Typical design parameters for trickling filters.



The removal of BOD from wastewater in trickling filters is usually modelled using first-

order kinetics (Schultze, 1960):

$$
S_e = S_0 e^{-kt}
$$
 (2.1)

where  $S_e$  and  $S_0$  represent the effluent and influent BOD concentrations respectively, k is the reaction rate constant, and t is the retention time within the filter. The retention time can be related to the depth, d, specific surface area of the medium, A, and the hydraulic loading rate, Q, using the semi-empirical relationship (Howland, 1958; Sinkoff

#### et al.,  $1959$ :

$$
t = \frac{CA^m d}{Q^n} \tag{2.2}
$$

where C, m and n are constants. The value of m is between 0.75 and 1.5, and a value of 1 is often used (Barnes *et al.*, 1983).

The value of n is constant for a given medium and is dependent on the medium geometry. It has values between 0.3 and I for different media, and is reported to be inversely proportional to the specific surface area, A (Atkinson & Ali, 1976; Jiménez et al., 1988).

The rate of BOD removal is affected to a small degree by the temperature; this can be included in the above equation by multiplying by the factor  $\Theta$ , where  $\Theta = 1.035^{(T-20)}$ (Schultze, 1960). Efficient BOD removal is reported at temperatures as low as 5'C and at a loading rate of 0.5 kg BOD. $m<sup>-3</sup>$ .d<sup>-1</sup> (Okey & Albertson, 1989; Solbé *et al.*, 1974).

## 2.1.3 Wastewater distribution systems

If it is assumed that the values of k, m, n, and A remain constant for the whole filter, the above equations can be combined as below:

$$
S_e = S_0 e^{-\frac{KA^2d}{Q^2}}
$$
 (2.3)

$$
(2.3)
$$

#### where the constants k and C are combined into a single constant,  $K$ .

Various methods have been devised for the application of wastewater onto the surface of filters. Large scale systems are usually based on either rotating or travelling distributor arms which can be dosed continuously or periodically using a siphon or a tipping trough. Alternatively, wastewater can be applied to the surface of high-rate

trickling filters using a fixed distribution network of pipes fitted with either nozzles or splash-plates.

The periodicity of wastewater application to a trickling filter affects the thickness of the developing biofilm and consequently the numbers of higher organisms, particularly flies, which may cause nuisance (Albertson, 1989; Hawkes & Jenkins, 1955; Lumb & Barnes, 1948; Tomlinson & Hall, 1955). By reducing the frequency of application, the rate of

flow over the biofilm is increased, thereby increasing the shear stress. This can be used to reduce excessive biofilm build-up on a filter, although a long-term increase in the hydraulic load may also reduce the treatment efficiency of the filter.

and 4:1, although higher ratios have been used, particularly for smaller plants. The effects are a decrease in the initial substrate concentration,  $S_0$ , and an increase in the flow, Q, with a consequent reduction in the residence time, t, in Equation 2.1.

Since the value of the exponential, n, in Equation 2.3 is less than 1, the net result is a reduction in the effluent substrate concentration,  $S_{\alpha}$ .

Total blockage of the medium surface is referred to as ponding, and can also be prevented by periodic raking of the medium surface, replacement with a higher voidage medium, recycling filter effluent (to increase the hydraulic load) or by the use of alternating double filtration (ADF).

Recirculation rates are usually at a ratio of effluent to settled wastewater of between 1:1

The other benefits of recirculation are that the higher flows ensure a more even distribution of water over the filter, thereby increasing the wetted area of the medium,

and also inhibiting the emergence of flies from the filter (Bruce & Merkens, 1973; Gray

& Learner,, 1984; Lumb, 1969; Sorrels & Zeller, 1955).

The overall beneficial effects of recirculation must be balanced against the addition costs

of pumping incurred.

 $\int$ 

The design loadings for both recirculation filters and ADF are in the range 0.25 1.21  $\overline{\phantom{a}}$  $kg$  BOD. m<sup>-</sup> of medium per day (Barnes *et al.*, 1981).

2.1.4 Support media

The development of plastic filter media in the late 1950's greatly increased the choice of media available for trickling filters. In general, plastic media have a far greater

ADF involves the periodic reversal of the sequence of two trickling filters with intermediate settlement, and has been reported to give improved performance in a number of cases (Gray & Learner, 1984; Lumb, 1969; Mills, 1945; Sorrels & Zeller, 1955; Wishart *et al.*, 1941). The first filter in the sequence receives a higher organic load, and biofilm development is consequently more rapid than in the second filter. When the order of the filters is reversed, the thick biofilm is starved of nutrients and will lose biomass through sloughing, thereby controlling biomass build-up. A possible

disadvantage with ADF is that heat-loss during the intermediate settlement stage may decrease the process efficiency (Bruce et al., 1975).

voidage than conventional mineral media, and are light enough to permit taller filter structures (Hutchinson, 1975).

There are two basic types of plastic support media: random and ordered types. Random media consist of small shaped pieces (usually sized between 20 and 200 mm) which are tipped at random into the filter. In contrast, ordered (or structured) media are constructed in larger bales, which must be carefully stacked in the filter. There are a multitude of different flow patterns which can be achieved using ordered media. Initial designs centred on horizontal and vertical flow media, but in recent years these patterns have

been almost entirely replaced by crossflow media, particularly at angles of 45<sup>°</sup> and 60<sup>°</sup>

to the horizontal which have been shown to possess superior performance (Harrison  $\&$ 

Daigger, 1987; Parker & Merrill, 1984; Richards & Reinhart, 1986).

Plastic media can be therefore be designed to achieve the required surface area, voidage

and, within reasonable limits, the retention time characteristics. They are generally of more consistent quality, but plastic media, particularly ordered media, are more expensive than mineral media. The low density of plastic media often enables savings to be made in the construction of filters.

The support medium in a trickling filter has two purposes: to provide an attachment surface for the microbial population and to facilitate the passage of water and air

The time required for the removal of nutrients from waste water is significantly less than the doubling time of the organisms responsible. It therefore follows that, if the bacteria were not in some way retained within the system, the hydraulic retention time for wastewater treatment would have to be in the order of days rather than hours.

#### through the filter.

While achieving these requirements, a medium must also satisfy several other criteria:

## 2.1.4a Microbial attachment

There are two methods by which bacteria may be retained: by the formation of flocs which can easily be settled out, as in activated sludge, or by attachment to solid objects which are either fixed in place or may be easily retained.

Bacteria adhere to solid surfaces by the formation of an extracellular acidic polysaccharide which adsorbs to the surface and forms an envelope around the bacteria (Fletcher & Floodgate, 1973). The products of subsequent cell divisions remain inside the polysaccharide envelope, such that a continuous film of biological matter known as

a biofilm eventually covers the surface.

The attachment of bacteria not only enables them to maintain a population in a system with a low retention time, but as early as 1936, it was reported that attachment appears to give the bacteria a kinetic advantage, particularly at low nutrient concentrations

(ZoBell & Anderson, 1936). This observed phenomenon has not been fully explained, but it is believed to be due either to the adsorption of nutrients to solid surfaces, or to the more effective use of exoenzymes by attached cells.

Nutrient diffusion limitations and grazing are thought to be responsible for the spring sloughing observed in trickling filters. During the winter, the activity of grazing macrofauna in the filter slows down, allowing a gradual thickening of the biofilm. As

The specific surface area of a solid, rounded medium, A in  $m^2.m^{-3}$ , can be approximated using the equation for the specific surface area of packed spheres in a cubic structure:

temperatures increase again during spring, the metabolism of the bacteria in the outer layers of the biofilm accelerates and they extract more nutrients from the effluent as it diffuses through the biofilm. The bacteria in the lower layers of the biofilm are therefore starved of nutrients and will die and detach from the support medium, taking the rest of the overlying biofilm with them. This process is therefore an inherent feature of all trickling filters and is essential to ensure the continuation of an active bacterial population in the biofilm.

#### 2.1.4b Mass transfer

A trickling filter support medium must allow water and air to permeate freely through the filter. The distribution characteristics are determined by the size, shape and packing geometry of the medium.

$$
A = \frac{\pi}{D}
$$
 (2.4)

where D is the diameter of the medium particles (in metres). However, the specific surface area of irregularly shaped media, such as slate or many plastic media, must be calculated using a variation on the paint-dipping technique (Porter  $\&$  Boon, 1972) and measurements of the packing density.

While the path of liquid flowing down through the filter is determined by the geometric shape and the packing characteristics of the particles, the rate of air convection is related to the voidage, the temperature difference between the medium and the air, and the ease of entry and exit from the filter.

The medium voidage is related to the size of the particles and to their geometry, but for near-spherical particles the voidage,  $\varepsilon$ , can be approximated by the equation (Barnes *et* 

Whether trickling filters are sited below or above ground, provision must be made for air to enter at the base of the filter. Conventional design practice recommends a ventilation area of about 5% of the filter surface area for BOD removal, or  $1-2\%$  for nitrification. This is easily achieved in filters built above ground level, by providing ports in the sides of the filter around the base. Filters below ground require either a

al., 1983):

$$
\epsilon = 1 - \frac{AD}{6} \tag{2.5}
$$

A support medium must have sufficient mechanical strength to withstand the weight of the layers of medium above it in the filter.

thereby greatly increasing their weight. The wet bulk density (ie the density of the medium when wet and covered with biofilm) of plastic media is typically 200 300  $kg.m<sup>-3</sup>$ , compared to  $1400 - 2000 kg.m<sup>-3</sup>$  for mineral media (Pike, 1978). ,  $2000$  kg.m

large central well, or a series of smaller vertical shafts spaced throughout the filter, to

provide ventilation.

#### 2.1.4c Structural strength

It is important, when calculating the required strength, to consider that the upper layers of medium may become clogged with biomass or even completely flooded with effluent,

The medium should also be sufficiently durable to withstand many years of use without

being adversely affected by freezing or by any substances likely to be found in wastewater. The generally accepted test for durability is the British Standard Sodium Sulphite Soundness method (BS1438,1971). However, it is thought that this test is too severe because many filter media which have failed the test have been shown to be very durable in practice. This is acknowledged in the wording of the standard.



Trickling filter media are characterised according to their specific surface area, voidage, shape and composition.

High voidage media are used when the loading applied to the filter is liable to result in excessive biofilm development and subsequent ponding. The specific surface area gives an indication of the amount of medium surface available for microbial attachment, although the shape of the medium particles and the hydraulic loading will determine how much of this surface is actually wetted. The composition of media can be divided between two basic types: plastic and mineral.

- ability to remove more BOD per unit volume at the same hydraulic loading, while maintaining effluent quality.

The category of mineral media includes broken rock, gravel, limestone, ceramics and slate although the most commonly used are the porous types: metallurgical coke, clinker and blast furnace slag (Wilkinson, 1958). The choice between the mineral media at a particular site is usually governed to some extent by local availability since transport costs are generally higher than the cost of the medium.

Mineral media were used in all trickling filters until plastic media became available in the 1950's. Mineral media typically have a low voidage  $(20-50\%)$  (Lo & Hung, 1988) and are consequently restricted in the hydraulic and organic loadings possible. Plastic media were introduced to overcome these problems. The ideal characteristics of a plastic medium compared with mineral media are: (Chipperfield, 1968)

- ability to operate at higher hydraulic loadings.
- adequate voidage to avoid blockage and ensure good ventilation.
- sufficient mechanical strength to support overlying layers.
- low density to enable significant savings to be made in filter construction.
- biological inertness.
- chemical stability.
- economical competitiveness with other types of media.

Some early synthetic polymers, such as some phenol-formaldehyde and ureaformaldehyde resins lacked chemical stability (Chipperfield, 1964) but modern plastic media made from unplasticised PVC or polypropylene have been shown to resist degradation under a wide variety of conditions (I.C.I. Ltd., 1963)

To reach the biofilm, nutrients must therefore pass from the fast-flowing bulk liquid into the slower liquid film covering the biofilm, and then into the biofilm itself by diffusion. Since the biofilm is composed of multiple layers of cells, the nutrient profile within the biofilm will be affected not only by diffusion, but also by the activity of the biofilm itself (Siegrist & Gujer, 1987; Suschka, 1987). When the biofilm becomes too thick, or the bacterial activity too rapid to allow nutrients to reach the lower levels of the biofilm, the bacteria there may die, lyse and detach from the support, causing sloughing of the  $$ whole affected biofilm. The equilibrium state, at which the net rate of biomass growth  $\overline{\phantom{a}}$ 

## 2.2 Biofilm kinetics

To comprehend the kinetics of waste water treatment, it is important to be aware of the processes occurring at the level of the biofilm.

Trickling filters, in common with most other waste water treatment processes, are three phase systems. They contain biofilm attached to a solid support, over which the liquid phase flows. The gaseous phase, providing oxygen and carbon dioxide, fills the remainder of the filter.

equals the rate of sloughing, is governed by factors such as the nutrient concentrations, biofilm activity and the shear stress (Rao Bhamidimarri & See, 1992).

The nutrient concentration profiles can be represented as a mass balance equation (Harremoës, 1978):

where N is the nutrient flux (mg.cm<sup>-3</sup>.s<sup>-1</sup>), x is the biofilm thickness, and r<sub>s</sub> is the rate of nutrient removal by the biomass  $(mg.cm<sup>-3</sup>. s<sup>-1</sup>)$ . Using Fick's second law, the nutrient flux can be related to the nutrient concentration gradient by:

$$
\frac{\delta N}{\delta x} = -r_s
$$



The diffusion processes present in a biofilm are represented schematically in Figure 2.1 (Harremoës, 1978). The relative rates of these processes, and the thicknesses of the liquid films and the biofilm, will determine the precise nutrient and reaction product concentration profiles, but simplified examples of these profiles are shown in Figure 2.2.

$$
N = D_s \frac{\delta^2 C_s}{\delta x^2} - r_s \tag{2.7}
$$

where  $C_s$  is the nutrient concentration in the biomass (mg.dm<sup>-3</sup>), and  $D_s$  is the nutrient diffusion coefficient in the biomass  $\text{cm}^2 \text{.} \text{s}^{-1}$ ). Similar equations can be written for all

substrates and products of biofilm reactions (Harremoës, 1982).

hydraulic loading rates (Maier, 1967; Williamson & McCarty, 1976). This results in a curved nutrient profile since transport is more rapid in the outer layers of water. Laminar flow has been apparently ignored by some researchers (Gujer & Boller, 1986), presumably in order to simplify their models, yet it has a significant effect on the

In Figure 2.2, the liquid film is shown exhibiting laminar flow, with the liquid adjacent to the biofilm moving very slowly compared to the outer layers (Hinton & Stensel, 1991). Laminar flow has been shown to exist over the typical range of trickling filter





transport of nutrients through the liquid film.

100 150 µm only (Chen & Bungay, 1981). Gullicks and Cleasby concluded that, for bulk-liquid ammonia-nitrogen concentrations above 4mg.dm<sup>-3</sup>, the diffusion of oxygen through the biofilm is the limiting factor for nitrification (Gullicks  $\&$  Cleasby, 1986).

As the nutrient reaches the biofilm, the profile is affected not only by the rate of diffusion, but also by the activity of the biofilm (Siegrist & Gujer, 1987). In the example shown, the oxygen is fully depleted before reaching the innermost layers of the biofilm, so this could lead to biofilm sloughing if maintained for a prolonged period. Research has shown that oxygen diffusion through a typical active biofilm is limited to the outer

This clearly demonstrates that measurements of the concentration of nutrients in the bulk liquid do not generally represent the true concentration as experienced by the bacteria. This difference becomes particularly important when comparing the effects of factors such as pH, dissolved oxygen and nutrient concentration between systems with different biofilm and liquid film characteristics (Siegrist & Gujer, 1987; Szwerinski *et al.*, 1986).

In a similar way, the products of microbial decomposition of the waste must diffuse out of the biofilm to the bulk liquid. There is therefore a significantly higher product concentration within the biofilm than is apparent in the bulk liquid which can, under certain circumstances, lead to product inhibition.

The study of the complex kinetics of transport, cell growth and nutrient removal in biofilms is a whole field of research in itself. This study is intended to concentrate on research into nitrification in trickling filters as a whole, so the kinetics within the biofilm will not be explored further. However, an excellent review of the subject was written

by Harremoës (Szwerinski et al., 1986).

# 2.3 Biological nitrification

# 2.3.1 Nitrifying bacteria

Microbial nitrification occurs in two sequential steps, involving the oxidation of ammonia first to nitrite, and then to nitrate.

$$
NH_{4}^{+} + 1\frac{1}{2}O_{2}^{-} + NO_{2}^{-} + H_{2}O + 2H^{+} \qquad \Delta E = -271kJ.(mole NH_{3}^{-}N)^{-1} \qquad (2.8)
$$
  

$$
NO_{2}^{-} + \frac{1}{2}O_{2}^{-} + NO_{3}^{-} \qquad \Delta E = -78kJ.(mole NH_{3}^{-}N)^{-1} \qquad (2.9)
$$

Both reactions are exothermic, and provide the energy required for the growth of the chemoautotrophic bacteria involved. To distinguish between the two reactions, they have been given the names nitritification and nitratification respectively (Prakasam & Loehr, 1972).

Nitritification of waste water is performed mainly by bacteria of the genus Nitrosomonas (notably N. europaea but also N. monocella). Nitrosococcus species and two species isolated from soil (Nitrosospira briensis and Nitrosolobulus multiformis) have also been shown to nitritify, but their activity is considered insignificant in the context of waste water treatment (Painter, 1977).

Nitrosomonas species are gram-negative obligate autotrophs (Hooper, 1969), gaining energy from the oxidation of ammonia and assimilating carbon from carbon dioxide. Carbon dioxide has been shown to be a non-limiting nutrient in waste water (Jones & Paskins, 1982). The bacteria are ovoid or rod-shaped of diameter approximately 1  $\mu$ m (Lewis & Pramer, 1958). They may be motile, although in trickling filters the bacteria

are attached to solid supports (Engel, 1961)

Nitratification in waste water is achieved predominantly by the species Nitrobacter agilis

and N. winogradskii, although bacteria from the genus Nitrosocystis and two marine species (Nitrosococcus mobilis and Nitrosospira gracilis) are also capable of performing the reaction (Painter, 1977).

Nitrobacter species are gram-negative facultative autotrophs (they can grow on an organic carbon source, although at a slower rate (Delwiche & Finstein, 1965)). They are also ovoid or rod-shaped, but smaller than Nitrosomonas at a diameter of 0.5  $\mu$ m

For the purposes of this work, the nitrifying organisms will be referred to under the general names of Nitrosomonas and Nitrobacter respectively.

(Zavarin & Legunkova, 1959) and may sometimes be motile (Pan, 1971).

It should be noted that a proton is liberated by each of the above reactions, resulting in a net acidification of the nitrifying culture.

However, the oxygen incorporated during nitratification has been shown to be derived from water, while molecular oxygen is again involved in the generation of energy via the cytochrome system (Aleem, 1970; Aleem *et al.*, 1965).

Nitritification can be separated into two distinct reactions, although the intermediate, hydroxylamine, is not normally seen. The first reaction can be summarised as (Painter, 1977):

 $NH_{4}^{+}$ +1/<sub>2</sub>O<sub>2</sub> -  $NH_{2}OH + H^{+}$  (2.10)



This first reaction is thought to release little or no energy, but the subsequent oxidation of hydroxylamine is thought to be exothermic, involving the cytochrome system with molecular oxygen acting as an electron acceptor (Aleem, 1970):

 $NH_2OH + O_2 \rightarrow H^+ + NO_2^- + H_2O$  (2.11)

No reaction intermediates have been found in the oxidation of nitrite (nitratification).
This dependence on molecular oxygen means that nitrification requires aerobic conditions, although the organisms can tolerate prolonged periods under anaerobic conditions (Kiff, 1972; Painter, 1970).

## 2.3.2 Nitrifier growth characteristics

The low energy yields from the nitrification reactions result in growth rates which are significantly slower than the growth rates of other aerobic bacterial species in waste water treatment, in particular the heterotrophic organisms. Therefore, to ensure the establishment and maintenance of a nitrifying population, it is necessary to limit the growth of competing species to the same as the growth rate of the nitrifiers. If the growth rate of competing bacteria exceeds that of nitrifiers, or if the mean cell residence time falls below the doubling time for the nitrifying population, then nitrifying activity will eventually be lost in a process known as "washout" (Downing *et al.*, 1964).

Typical values for the maximum specific growth rate  $(\mu_{\text{Max}})$  of *Nitrosomonas* are 0.5-2  $d<sup>-1</sup>$  (Buswell, 1954; Engel & Alexander, 1958; Hall & Murphy, 1980; Jones & Paskins,

1982; Loveless & Painter, 1968). The U.S. Environmental Protection Agency uses a figure of 0.47  $d^{-1}$  for river water and pure culture, but only 0.18  $d^{-1}$  for activated sludge (U. S. Environmental Protection Agency, 1975). This latter figure is now considered to be too low and the value of  $0.47$  d<sup>-1</sup> is often used instead for kinetic calculations in activated sludge systems.

The maximum specific growth rate of *Nitrobacter* is in the range  $0.3$ -1.4 d<sup>-1</sup> (Boon & Laudelout, 1962; Hall & Murphy, 1980). The slower growth rate of Nitrosomonas relative to Nitrobacter measured in laboratory studies explains the fact that nitrite, the product of nitritification, is very rarely observed in the effluent of sewage treatment works except under certain conditions of temperature and ammonia concentration where Nitrobacter is preferentially inhibited (Anthonisen et al., 1976; Balmelle et al., 1992). This is discussed in greater depth in Section 2.3.3e.

In practice, the Monod kinetics are often disguised by complex inter-reactions due to the heterogeneous nature of wastewater and to the effects of diffusion of nutrients either through bacterial flocs or through a biofilm. These are perhaps the reasons for some reports of first order or zero order kinetics (Bovendeur et al., 1990; Gonenc & Harremoës, 1990; Gujer & Boller, 1983; Shin & Polprasert, 1988; Vogelpohl et al.,

The growth of nitrifying bacteria follows Monod kinetics, where the specific growth rate is dependent upon the concentration of growth-limiting substrate in contact with the microbial culture, according to the relationship (Monod, 1949; Castens & Rozich, 1986; Chen et al., 1989; Downing & Knowles, 1966; Knowles et al., 1965; Quinlan, 1984):

$$
\mu = \mu_{\text{Max}} \frac{S}{K_{\text{SN}} + S} \tag{2.12}
$$

where  $\mu_{\text{Max}}$  is the maximum value of the specific growth rate,  $\mu$ , when substrate concentration, S, is not limiting.  $K_{SN}$  represents the half-saturation constant, the substrate concentration at which  $\mu$  is equal to half  $\mu_{\text{Max}}$ .

1990; Wild *et al.*, 1971). Other workers conduct experiments at extremely high loadings, hence the kinetics are within the pseudo-zero order range of the Monod curve (Gupta et al., 1994; Srinath et al., 1976).

The value of  $K_{SN}$  (in mg  $NH_4-N$ .dm<sup>-3</sup>) for the growth of *Nitrosomonas* is thought to be related to the liquid temperature by the relationship:

$$
K_{SN} = 10^{(0.051T - 1.158)}
$$
 (2.13)

or

Agency, 1975). For temperatures lower than  $23^{\circ}$ C, these relationships give values for

$$
K_{SN} = 0.4 e^{-0.118(T-15)}
$$
 (2.14)

where T is the temperature (in  $^{\circ}$ C) (Knowles *et al.*, 1965; U.S. Environmental Protection

 $K_{SN}$  below 1 mg.dm<sup>-3</sup>, which is significantly lower than the ammoniacal nitrogen concentration typically found in waste water (Knowles et al., 1965; Painter, 1977; Sharma & Ahlert, 1977). Under conditions of higher ammonia concentrations, Equation 2.12 can be approximated by  $\mu = \mu_{\text{Max}}$ , which represents zero order kinetics with respect to S. These are therefore the growth kinetics usually observed in sewage treatment processes where incomplete nitrification is occurring.

The low energy yield available to nitrifying organisms means that the biomass yield for nitrification is less than a quarter that of heterotrophic organisms. The yield coefficient is often quoted to be 0.15 mg biomass (as volatile solids) per mg ammoniacal nitrogen. In lightly loaded nitrifying systems, where endogenous respiration is more significant, the yield coefficient can fall to approximately  $0.10$  mg.mg<sup>-1</sup>.

### 2.3.3 Factors affecting the growth of nitrifying organisms

The growth rate of nitrifying organisms  $(\mu)$  is related exponentially to the temperature, T, by a form of the van't Hoff-Arrhenius equation (Metcalf & Eddy, 1973):

where  $k_T$  is the temperature coefficient (units  ${}^{\circ}C^{-1}$ ). The equation applies for temperatures between 8' and 30'C.

The reported values for the temperature coefficient,  $k_T$ , lie within the range 0.095  $^{\circ}C^{-1}$ (Ekama & Marais, 1984; Knowles et al., 1965) to 0.12  $^{\circ}$ C<sup>-1</sup> (Downing, 1968; Downing

& Knowles, 1966; Jenkins et al., 1984) although a low value of  $0.064$  °C<sup>-1</sup> has been recently reported (Sen et al., 1992).



$$
\mu = \mu_{15} e^{k_T (T-15)} \tag{2.15}
$$

Growth of nitrifiers effectively stops below 4'C (Buswell, 1954; Painter, 1970). The opulnum growth rate for *Nitrosomonas* has been reported to lie in the range 30  $30^{\circ}C$ (Buswell, 1954), above which the activity drops sharply. The thermal death point of a pure culture of *Nitrosomonas* is between 54<sup>°</sup> and 58<sup>°</sup>C (Barritt, 1933).

It has been shown that the nitrifying bacteria are capable of adapting to different temperatures so that the short-term response to temperature changes is not as favourable

as the long-term response (Gujer & Erni, 1978; Tendaj et al., 1992) (Figure 2.3). For this reason, the temperature of nitrification processes should be maintained as stable as possible.

Increasing temperature also exerts a negative influence on nitrification kinetics by affecting the values of the half-saturation constants for both ammonia and dissolved oxygen ( $K_{SN}$  and  $K_{SO}$  respectively). An increase in temperature causes an increase in both of these constants, thereby reducing the rate of nitrification (Knowles *et al.*, 1965;





Temperature ('C)

 $5 \t\t 10 \t\t 15 \t\t 20$ 

 $-$  Long Term Adaptation  $\cdots$  Varied from  $5^{\circ}$ C  $-$  Varied from 20 $^{\circ}$ C

Figure 2.3: The effects of short-term and long-term temperature changes on the growth of nitrifiers. (Gujer 1978)

Painter, 1970; Painter, 1977; Sharma & Ahlert, 1977; U. S. Environmental Protection Agency, 1975). The relationships between these constants and the temperature are given in Sections 2.3.2 and 2.3.3b respectively.

2.3.3b Dissolved oxygen concentration

The effect of dissolved oxygen concentration on the growth kinetics of nitrifying

bacteria has been shown to follow Monod kinetics (Boon & Laudelout, 1962; Chen et al., 1989; Downing et al., 1964; Knowles et al., 1965; Loveless & Painter, 1968):

$$
\mu = \mu_{Max} \frac{C_o}{K_{so} + C_o}
$$
 (2.16)

where  $\mu$  represents the growth rate,  $\mu_{\text{Max}}$  is the maximum growth rate,  $C_{\Omega}$  is the dissolved oxygen concentration (DO) and  $K_{so}$  is the half-saturation constant for oxygen.

 $O_2$ . dm<sup>-3</sup> (Loveless & Painter, 1968; Stankewich, 1972), although Stankewich's analysis of other researchers' data (Boon & Laudelout, 1962; Downing et al., 1964; Knowles et al., 1965; Loveless & Painter, 1968) suggests that the half-saturation constant may be dependent on temperature (Stankewich, 1972):

 $K_{S O (1)} = K_{S O (15)} e^{0.069 (T-15)}$  (2.17)

where the oxygen half-saturation constant at  $15^{\circ}$ C, K<sub>so(15)</sub>, is 0.21 mg.dm<sup>-3</sup>.

The half-saturation constant has been shown to have a value of between 0.2 and 0.4 mg

Southgate obtained a value for  $K_{SO}$  of 1.3 mg.dm<sup>-3</sup> and concluded that operation of activated sludge plants above a DO concentration of  $2 \text{ mg.dim}^3$  would not justify the extra costs of aeration involved (Southgate, 1964). The wide range of values for  $K_{s0}$ reported in the literature is suggested to be due to differences in measurement techniques, double substrate- limiting kinetics, or differences in floc size affecting the

## diffusion (Stenstrom & Poduska, 1980)

The stoichiometric oxygen requirements for nitritification and nitratification are respectively 3.43 mg  $O_2$  per mg NH<sub>4</sub>-N and 1.14 mg  $O_2$  per mg NO<sub>2</sub>-N. Adding these values gives a theoretical nitrogenous oxygen demand (NOD) of 4.57 mg  $O<sub>2</sub>$  per mg NH4-N. In practice, the basal metabolism of the bacteria must also be taken into account,

which results in a net reduction in the oxygen demand to 4.3 mg  $O_2$  per mg NH<sub>4</sub>-N due

to a small amount of oxygen which is generated by cell metabolism (Wezernak & Gannon, 1967).

An apparent inhibition of nitrification by high dissolved oxygen concentrations (as encountered when using pure oxygen aeration) (Charley *et al.*, 1980; Jones & Paskins, 1982; Painter & King, 1976) has been attributed instead to lack of carbon dioxide (Painter, 1986).

Anoxic conditions, such as the denitrification stage of an activated sludge plant, are tolerated by nitrifiers for the periods normally encountered in treatment processes. No loss of activity was reported for anoxic periods of up to 8 hours (Downing *et al.*, 1964; Kiff, 1972), although an oxidation ditch liquor lost  $50\%$  activity following 2 days of anaerobic conditions, and 90% after 9 days (Srinath et al., 1976).

Low dissolved oxygen concentrations have been reported to be a contributory factor in

the accumulation of nitrite, partly because *Nitrobacter* has a higher  $K_{so}$  than Nitrosomonas (Alleman, 1985; Balmelle et al., 1992; Yang & Alleman, 1992).



The optimum pH for the growth of nitrifying organisms is generally agreed to be in the range 7 - 9 (Boon & Laudelout, 1962; Hofman & Lees, 1953; Jones & Paskins, 1982; Loveless & Painter, 1968; Meyerhof, 1916; Painter, 1970; Sharma & Ahlert, 1977), although there is considerable evidence to suggest that cultures are able to adapt to

different pH values over time (Engel & Alexander, 1958; Hofman & Lees, 1953; Meyerhof, 1916). The rate of nitrification becomes very slow at pH values below 6.5 or above 10 (Downing et al., 1964; Srinath et al., 1976; Wild et al., 1971), although a plant treating poultry manure achieved 50% nitrification at pH 4.9 (Prakasam & Loehr, 1972).

Nitrosomonas was modelled by Downing and Knowles using the following relationship (Downing & Knowles, 1966):

It is proposed that the effects of pH on nitrification and nitrifier growth are due to the equilibria between ammonia-ammonium and nitrite-nitrous acid (Anthonisen *et al.*,

Using acclimatised activated sludge, the effect of the pH on the growth rate of

$$
\mu = \mu_{Max} (1 - 0.83(7.2 - pH))
$$
 (2.18)

### For pH values between 7.2 and 8, Equation 2.18 can be simplified to:

$$
\mu = \mu_{\text{Max}} \tag{2.19}
$$

At high pH, the rate of nitritification can be made to exceed the rate of nitratification, resulting in an accumulation of nitrite (Alleman, 1985; Balmelle *et al.*, 1992). It has

1976). It is suggested that the unionised forms (ammonia and nitrous acid) are able to permeate the cell membrane, causing inhibition, when a pH gradient exists across the cell membrane. This aspect of nitrifier inhibition will be discussed at greater length in Section 2.3.3e.

The buffer capacity or alkalinity of most waste waters, particularly in hard water areas, is usually sufficient to ensure that the hydrogen ions produced during nitrification do not cause an excessive drop in pH. However, in soft waters, or when a concentrated ammonia waste is being treated, nitrification may be limited by the available alkalinity. Stoichiometrically, nitrification consumes 7 mg of carbonate alkalinity per mg of

#### ammoniacal nitrogen oxidised (U.S. Environmental Protection Agency, 1975).

recently been suggested that this method could be used to induce direct denitrification of nitrite, thereby cutting out the nitratification step altogether and improving process efficiency (Abeling & Seyfried, 1992; Balmelle et al., 1992) as hypothesised by Turk et al (Turk & Mavinic, 1989). The potential advantages of this process would be a reduction in plant volume (due to an increase in denitrification efficiency), lower aeration demand, and a reduction in the carbon requirements for denitrification. However, the conditions required for direct coupling of nitritification and denitritification

would necessitate very precise control of the treatment process and the system would be particularly susceptible to breakdown in the event of microbial inhibition. Further research is therefore needed before this technology can be applied at full scale.

A theoretical study of the effects of pH produced a predicted optimum pH which decreased almost linearly with the logarithm of the ammonia-N concentration over the range  $0 - 50$  mg.dm<sup>-3</sup> (Quinlan, 1984). The prediction assumes that the substrate for nitrification is unionised ammonia, and that the subsequent ionisation of the enzymesubstrate complex is the rate-limiting step of nitrification. This shifting optimum implies

that there is no set pH for maximising nitrification.

The nitrification reaction liberates two protons per molecule of ammonia oxidised to nitrate, one during nitritification and one during nitratification. This equates to the theoretical consumption of 7.14 mg of alkalinity per mg of ammoniacal nitrogen. Therefore there is a tendency for the pH of the waste water to become gradually more acidic as nitrification proceeds. In most cases, the alkalinity of the waste water is sufficient to buffer the protons and prevent a significant drop in pH. However, problems can arise when the alkalinity or hardness of the waste water is low, or when an ammonia-rich waste is being treated. In such cases, the alkalinity can be supplemented

by the use of lime, caustic soda or limestone.

#### 2.3.3d Overall nitrifier growth equation

The above equations defining the effects of temperature, dissolved oxygen, substrate

concentration, and dissolved oxygen concentration can be combined to give an overall equation for growth of nitrifying organisms:

$$
\mu_{TpH,S,DO} = \mu_{Max}e^{0.098(T-15)}[1-0.833(7.2-pH)]\frac{S}{S+10^{0.051T-1.158}}\frac{DO}{DO+K_{SO}}^{(2.20)}
$$

2.3.3e Substrate and product inhibition

Both Nitrosomonas and Nitrobacter species are reported to be sensitive to high concentrations of their own substrate, but they are more sensitive to the substrate of the other (Painter, 1970). This theory was developed by Prakasam and others, who concluded that the unionised forms of the substrates (ammonia and nitrous acid) are inhibitory. The equilibrium between the ionised forms and the unionised forms are dependent on the pH (Appendix 1). Hence substrate and product inhibition is related to the pH (Anthonisen *et al.*, 1976; Prakasam & Loehr, 1972). This relationship is illustrated in Figure 2.4 (Anthonisen et al., 1976).

In zone 2, the free ammonia concentration has dropped sufficiently from zone I to allow Nitrosomonas to metabolise normally, although Nitrobacter is still inhibited. Similarly, zone 4 represents product-inhibition of Nitrobacter by free nitrous acid. Nitrosomonas

Figure 2.4 illustrates four operating zones (1-4) separated by three boundary areas (A-C) which represent the transition from partial to complete inhibition of the organism concerned. Data used in the generation of the figure were obtained in a variety of ways and at a number of different temperatures, yet they still exhibit a remarkable degree of convergence. The most serious weakness of the research is the lack of acclimatisation allowed, so the actual inhibition boundaries could be at higher concentrations.

Zone 1 represents the set of conditions where a combination of high pH and a high total ammonia-nitrogen concentration result in the ionisation equilibrium shifting towards unionised free ammonia, causing the inhibition of both Nitrosomonas and Nitrobacter.



is apparently unaffected by the presence of nitrous acid at the concentrations used. Under both these sets of conditions, nitrite will be seen to accumulate in the system.

Optimal nitrification is represented by zone 3, where both free ammonia and free nitrous acid are below the inhibition threshold values for both bacterial species.

Anthonisen's conclusions are disputed by Yang & Alleman, who propose that it is the accumulation of free hydroxylamine, in addition to free ammonia, which causes nitrite accumulation (Yang & Alleman, 1992).

Product inhibition (inhibition of nitrification by nitrate) has also been reported at high nitrate concentrations (Boon & Laudelout, 1962).

At the concentrations typically found in domestic wastewater, neither product nor substrate inhibition is usually significant. The majority of inhibition problems are due

# to the elevated concentrations of nitrogen compounds which can be found in certain industrial wastes.

## 2.3.3f Competition from heterotrophs

The growth rate of heterotrophic organisms is reported to be at least an order of magnitude higher than that of nitrifying organisms (Barnes et al., 1983). Therefore under conditions of a constant high BOD, heterotrophs will dominate the system at steady state and nitrifiers will be washed out. However, the short-term effects of other organisms on the activity of nitrifying organisms has not been fully explained.

be sustained over long periods, is often quoted to be as low as 15  $20$  mg. dm<sup>-</sup> (Harremoës, 1982; Parker & Richards, 1986; Paulson, 1989; Wanner, 1984). However, shock loads of up to  $110 \text{ mg.dim}^3$  BOD have been shown to have negligible effects on the rate of nitrification (Wild *et al.*, 1971).

Several researchers report a reduction in nitrification when organic matter is added to nitrifying trickling filters and activated sludge, and suggest that this is due to an increase in the growth of heterotrophs (Chen *et al.*, 1989; Stover *et al.*, 1976). It has been suggested that oxygen is often the limiting substrate for nitrification (Gonenc  $\&$ Harremoës, 1985; Painter, 1970), and the presence of heterotrophs will cause competition for the available oxygen. The critical BOD, above which nitrification cannot

Some workers report either small increases (Hockenbury *et al.*, 1977) or large increases (Jones & Hood, 1980) in the rate of nitrification when heterotrophic organisms are added to pure cultures. These findings are contrary to expectations, and the increased activity could be due to trace elements, which were omitted from the pure culture

medium, being added with the heterotrophs.

## 2.3.3g Other toxic and inhibitory substances

Nitrifying organisms, particularly *Nitrosomonas*, are subject to inhibition by a very wide

variety of inorganic and organic substances. The degree of inhibition exerted by a particular inhibitor depends on (Tomlinson et al., 1966):

- the presence of microorganisms other than nitrifiers
- the concentration of the inhibitor
- the concentration of the nitrifiers
- the pattern and duration of exposure to the inhibitor
- the mixing regime in which exposure occurs
- the presence of other inhibitors

In the past, several researchers suggested that organic matter was inhibitory to nitrification (Barritt, 1933; Downing *et al.*, 1964). It is now known that organic matter is not itself toxic, but it's presence promotes the development of a heterotrophic

The duration of exposure is important because nitrifiers have been shown to become acclimatised to many organic chemicals under conditions of long-term or repeated exposure (Tomlinson et al., 1966).

There are four major groups of compounds which have been shown to be particularly toxic to nitrifiers: isothiocyanates, cyclical N-containing compounds, amines and phenols. The most powerful of these are the isothiocyanates, which contain the S-C-N grouping, and are exemplified by thiourea which has been shown to be 75% inhibitory at a concentration of only 1  $\mu$ M (0.08 mg.dm<sup>-3</sup>) (Wood *et al.*, 1981). The inhibition by these compounds is believed to be effected at the level of the copper electrocouple in the cytochrome chain of *Nitrosomonas*, and is therefore quite specific to nitritification

Various metals, including copper, cadmium, nickel, zinc, chromium and mercury, have been shown to inhibit the growth of nitrifiers, and in most cases of heterotrophs also (Braam & Klapwijk, 1981; Martin & Richard, 1982; Painter, 1970; Tomlinson *et al.*,

population which can cause oxygen deficiency or competition for attachment sites (Hockenbury et al., 1977; Kiff, 1972; Painter, 1977; Sharma & Ahlert, 1977).

(Painter & Jones, 1963; Wood et al., 1981).

ammonia (Sato *et al.*, 1988). On the other hand, a metal may bind to an inhibitor with chelating properties and thereby reduce the inhibitory action of both species (Tomlinson et al., 1966).

Comprehensive lists of chemicals shown to influence the growth or activity of nitrifiers are given in reviews by Hockenbury and Richardson (Hockenbury *et al.*, 1977;

Richardson, 1985).

The duration of an inhibitory effect on nitrification is often of the order of a few hours (Hockenbury et al., 1977; Randall & Buth, 1984). However, severe pollution incidents or inhibition during cold weather can result in delays of several months for the reestablishment of nitrification (Tendaj et al., 1992).

## 2.3.3h Nitrifying population size

The observed rate of nitrification in a system is related to both the number and activity

of the nitrifying organisms (Downing & Knowles, 1970; Metcalf & Eddy, 1973). The concentration range of nitrifiers in wastewater has been estimated to be 1,000-10,000 organisms per cm' (Strom et aL, 1976).

At low concentrations of microorganisms, the rate of nitrification is directly proportional to the inoculum size. As the inoculum size increases, the effects of diffusion and substrate limitation exhibit more of an influence so the relationship is no longer linear (Srinath et al., 1976).

Typical values for nitrification activity, expressed as mg nitrogen oxidised per g cells

per hour at 20-30 $\degree$ C are within the range 0.5-0.6 mg.g<sup>-1</sup> for activated sludge or 110-250

mg.g<sup>-1</sup> for pure cultures (Srinath et al., 1976; U.S. E.P.A., 1975).

## $2.3.3i$  Other factors affecting growth of nitrifiers

Attachment to solid surfaces was once thought to be essential for the growth of nitrifying organisms (Lees, 1955). However, this has been shown to be untrue since nitrifiers can be cultured in clear media (Engel & Alexander, 1958; Painter, 1970; Painter, 1977; Sharma & Ahlert, 1977).

Light has been shown by some researchers to have an inhibitory effect on the growth of both Nitrosomonas and Nitrobacter (Painter, 1970; Sharma & Ahlert, 1977). However, this effect is unlikely to be significant in either activated sludge or trickling filters, although non-enclosed rotating biological contactors may exhibit some inhibition.

comparison with autotrophic nitrification, its effects are relatively insignificant (Focht  $&$  Chang, 1975).

It has also been suggested that an unidentified micro-organism, possibly a *Nitrosomonas* 1-D species, can produce nitrous oxide or nitrogen from nitrite under anoxic conditions, with

There are a number of micronutrients (vitamins, amino acids and essential trace metals) which are reported to influence the growth of nitrifying bacteria. Their effects are summarised in a review by Sharma & Ahlert (1977). However, it is unlikely that any of the metals would be absent from domestic wastewater.

In addition to the autotrophic nitrifiers described above, nitrification has been shown to occur both in aerobic heterotrophic and anoxic autotrophic populations.

Aerobic heterotrophic nitrification has been shown to occur in the stationary phase of the cell cycle. This contrasts with autotrophic nitrification, which occurs during the growth phase (Focht & Chang, 1975). Heterotrophic nitrification has been demonstrated  $\frac{2}{10}$ , 1715). Henrich propr in rivers, lakes and soil as well as wastewater treatment (Painter, 1977), although, in

the simultaneous oxidation of ammonia for use as an energy source, although there are too few data available to confirm this (Poth, 1986; van der Graaf et al., 1991; van der Graaf et al., 1990).

## 2.4 Nitrifying trickling filters

In the past, many sewage treatment works have been designed without any specific requirement for nitrification. More recently however, the initiative towards cleaner rivers and the general acceptance of a minimum Class 11 standard, enforced by the National Rivers Authority (NRA), has resulted in more stringent ammonia discharge consents. Whereas only a few years ago, permissible ammonia concentrations were as high as 20 or 30 mg. dm<sup>-3</sup>, many sewage works are now having consents of  $5$ -10 mg. dm<sup>-3</sup> imposed upon them.

Nitrifying trickling filters offer a relatively cheap method of tertiary biological nitrification. They are easy to design and construct, have been well researched and characterized in the context of large sewage treatment works (Upton  $\&$  Cartwright, 1984) and offer simple and reliable operation. The space requirements, while not as small as submerged biological filters, are nevertheless within reason for most sites. However, less information is available on the performance of plastic medium nitrifying filters or on nitrifying filters following high rate filtration.

Heterotrophic bacteria involved in carbonaceous BOD removal have a much higher growth rate than the autotrophic bacteria involved in ammonia removal (Barnes *et al.*, 1983; Sharma & Ahlert, 1977). Therefore, in a fixed film process where attachment space is limited, the heterotrophic population will inevitably dominate the system whenever the BOD is high enough. Trickling filters are effectively plug flow reactors, so in a sewage treatment process the waste water will initially have a high BOD. As the waste trickles down through the filter, or a series of filters, the BOD will eventually reach a point where autotrophic bacteria can compete. Thus BOD removal and nitrification are separated into two distinct phases of the treatment process. This

contrasts with completely mixed activated sludge systems, where nitrification and BOD removal can occur simultaneously.

It has been reported recently that nitrification can be coupled with denitrification in the aerobic biofilm of a rotating biological contactor if the biofilm is allowed to accumulate to a sufficient depth. Nitrification occurs in the outer layers of biofilm (Nishidome  $\&$ Kusuda, 1991) and the nitrate thus formed is denitrified in the anaerobic inner layers

(Masuda et al., 1987). However, there are as yet little data to confirm these findings, and the methods to maintain and control a biofilm of sufficient thickness are not fully understood.

Conventionally designed trickling filters can, if under-loaded, achieve quite a high degree of nitrification. Bruce reported a single pass filter containing 50 mm grade stone medium, loaded at  $0.093$  kg BOD.m<sup>-3</sup>.d<sup>-1</sup> and  $0.3$  m<sup>3</sup> water.m<sup>-3</sup>.d<sup>-1</sup>, which provided almost complete nitrification throughout the year at temperatures of 12  $18^{\circ}$ C (Bruce *et al.*, 1975).

The biomass yield and bacterial growth rate on trickling filters for nitrification are much lower than for BOD removal, leading to the development of thinner biofilms. Support media with narrower void spaces can therefore be used for nitrifying trickling filters.

## 2.4.1 Design of nitrifying trickling filters

To optimise the performance of nitrifying trickling filters, certain changes should be made to the design of conventional filters in order to optimise the conditions for a stable nitrifying population.

The nitrification reaction occurs more slowly than other reactions in waste water treatment, therefore the retention time within the filter should be increased. According to Equation 2.2, the retention time is directly proportional to the depth, and approximately proportional to the specific surface area of the medium.

Although nitrifying biofilms have a lower specific activity than heterotrophic biofilms, the higher oxygen demand for nitrification implies that the specific oxygen demand for a nitrifying filter is approximately equal to that for an intermediate-rate filter for BOD removal. However, heat retention is more important in a nitrifying filter, so designs must achieve a compromise between excessive heat loss through convection and inadequate aeration.

The optimal hydraulic loading rate for nitrification has been reported to be at about 6 m<br>111  $\mathbf{3}_{\geq 3}$ m.". $a$ , although higher rates are possible (Hill *et al.*, 1990). The maximum loading rate for efficient ammonia removal is about 0.1 kg  $NH_4-N.m^{-3}.d^{-1}$  (Hill *et al.*, 1992; Upton & Cartwright, 1984). Combining these two figures, the maximum ammoniacal nitrogen concentration for effective treatment at a hydraulic loading of 6  $m^3.m^{-3}.d^{-1}$  is 16.7 mg.dm<sup>-3</sup>.

This can be achieved by building tall filters containing low voidage medium, but heat loss through convection can also be reduced by limiting the aeration area to 1 - 2% of the plan area of the filter (Hill *et al.*, 1990). Nitrifying trickling filters are typically 2.5 to 4 metres deep for stone media down to 25 mm diameter, or up to 10 m tall using plastic media (Hutchinson, 1975).

It is worth noting here that the current British Standard governing the design of trickling filters in small sewage treatment works, permits a maximum depth of mineral medium filters of only 2.5m (B.S. 6297, 1983). While this may apply to filters for BOD removal,

Nitrifying trickling filters have been reported by some researchers to be sensitive to periodic dosing (Parker et al., 1990; Upton & Cartwright, 1984). Consequently, many nitrifying filters are equipped with fixed continuous dosing systems, usually by means of pipes laid on the filter surface fitted with upward spraying nozzles. This system minimises the maintenance since there are no moving parts and the nozzles are designed

the standard should be modified to reflect the fact that nitrifying filters can be built to greater depths.

to prevent blockage, and it ensures that the distribution is even and continuous.

This method of continuous application of wastewater also helps to minimise the numbers of filter flies emerging from the filter, which can cause a nuisance, particularly during spring and early autumn (Parker et al., 1990; Southgate, 1964).

When recirculation is employed on a nitrifying filter, the effluent should not be settled

channel widths through the media can be reduced, allowing a greater specific surface area over which the biofilm can develop. Several studies have concluded that the volumetric nitrification capacity of a filter medium is directly proportional to its specific surface area (U.S. Environmental Protection Agency, 1975).

between passes since this significantly increases the treatment time and permits greater cooling to occur.

## 2.4.2 Support media for nitrification

The primary difference between media for BOD removal and for nitrification is in the size of the specific surface area which can be tolerated. The low biomass yields and slow growth rates of nitrifying organisms result in significantly thinner biofilms, and there is consequently a reduced risk of clogging in a nitrifying filter. Therefore the

Furthermore, nitrification will normally follow at least two other treatment processes (including settlement) and so there is minimal risk of blockage due to rags or fibres remaining in the waste water when it reaches the nitrification stage.

For trickling filter media, the maximum specific surface area used in practice is

approximately 230 m<sup>2</sup>.m<sup>-3</sup>, achieving up to 0.3 kg.m<sup>-3</sup>.d<sup>-1</sup> nitrification (Boller & Gujer, 1986). However, higher specific surface areas can be used in submerged aerobic biofilters (SABF) and fluidised beds since the air (added at the base of the medium) has a scouring action to prevent blockage. Using static media of specific surface area 500 m<sup>2</sup>.m<sup>-3</sup> in a SABF, nitrification rates above 3 kg.m<sup>-3</sup>.d<sup>-4</sup> have been achieved, albeit using

a concentrated synthetic waste water at high pH (Vogelpohl et al., 1990). In a fluidised  $\overline{\phantom{a}}$ bed reactor, fine media such as sand  $(2000 \text{ m}^2 \text{ m}^3)$  can be used to achieve nitrification rates of up to 6 kg.m<sup>-3</sup>.d<sup>-1</sup> (Tijhuis et al., 1992)

## 2.5 Modelling of trickling filters

- Fully empirical models which attempt to correlate filter performance to applied  $1)$ operating conditions (Gullicks & Cleasby, 1986; Roberts, 1973; U.S. Environmental Protection Agency, 1975).
- ii) Semi-empirical models where empirical correction factors are used to improve the accuracy of mass-balance based models (Bruce & Merkens, 1973; Germain, 1966; Gromiec et al., 1972; Schultze, 1960; WRc, 1987).
- iii) Mechanistic models which incorporate biofilm kinetics and nutrient mass transfer equations (Furumai & Rittmann, 1992; Grady & Lim, 1980). These models

Most of the models in the literature are based, at least in part, on empirical observation

due to the complexity of the trickling filter process. There are three classes of models:

necessarily are greatly simplified, and are therefore of limited application in real situations.

One of the major problems with modelling a trickling filter is the concept of partial wetting of the medium (Albertson & Eckenfelder, 1984; Crine *et al.*, 1990; Parker & Merrill, 1984). Partial wetting is caused by the flow of liquid over a filter medium, which tends to form channels and leave parts of the medium dry. The dry areas of medium do not contribute to the treatment process, but the size of these areas cannot be measured directly, and is dependent on many different factors, including the hydraulic and organic loading rate, temperature, medium type and the distribution method

## (Atkinson & Ali, 1978; Gullicks & Cleasby, 1986; Hosono, 1980; Hutchinson, 1975).

Several attempts have been made to model partial wetting, but most of the models make assumptions for the sake of simplicity which renders the model non-applicable to real situations. Some models assume that the particles of medium are perfectly spherical

(Atkinson & Ali, 1976; Howland, 1958; Muslu, 1986). Others assume constant biofilm thickness (Atkinson & Ali, 1976; Atkinson & Ali, 1978) or that the particles of the medium are very small (Mills & Dudukovic, 1981).

Only the research by Hinton & Stensel provides analysis of hydraulics in a real filter medium colonised with biofilm (Hinton & Stensel, 1991). They conclude that the hydraulics are laminar flow interrupted (and mixed) by periodic falling liquid drops, but

Additional difficulties in model formulation arise from the complex inter-relationships between the factors affecting trickling filter performance. For example, the air temperature affects the wastewater temperature (Bayley & Downing, 1963), the pH optimum (Quinlan, 1984), half-saturation constants for oxygen and substrate ( $K_{SO}$  and

the frequency of falling drops and the size of un-wetted areas are unpredictable.

 $K_{SN}$ ) (Knowles *et al.*, 1965; Quinlan, 1980; Sharma & Ahlert, 1977), microbial growth rate (Downing *et al.*, 1964), and the activity of biofilm-grazing organisms (Särner, 1980).

Any model of trickling filter performance will therefore require an empirical factor to allow for the un-wetted fraction of the medium surface area.

Further difficulties are caused by the complex and varied nature of waste water, the multitude of different media and filter configurations, and the fact that, unlike anaerobic treatment, the conditions inside a trickling filter cannot easily be controlled.

Few published models take any account of non-steady state conditions. The Monod equation, taken as a basis for nearly all models, assumes that the growth rate changes

instantaneously in response to changes in conditions. Some of the inaccuracies implicit in this assumption have been investigated by a number of workers (Hermanowicz  $\&$  $\sim$ Ganczarczyk, 1985; Mateles et al., 1965; Storer & Gaudy, 1969).

The U.S. Environmental Protection Agency produced two publications relating to

Gullicks and Cleasby proposed some empirical design curves, which take into account effluent recycling, medium characteristics and temperature, as well as the hydraulic and

nitrogen removal in the 1970's. The first was a design manual which enables the calculation of basic design criteria based on the performance required from the filter (U. S. Environmental Protection Agency, 1975). This was followed by a more general review of nutrient removal technology (U.S. Environmental Protection Agency, 1978). However, the manual is of limited applicability since the pilot scale trials on which it is based used predominantly tall filter structures containing corrugated vertical plastic media, as used in the research of Duddles (Duddles et al., 1974).

The conclusions were that the maximum potential rate of nitrification is dependent upon the specific surface area of the medium, and that the composition of the waste water, particularly in terms of BOD and suspended solids, has a highly significant effect. The optimal ammonia loading rate was found to lie within the range 600 1000 M2 of the medium surface area per kg of ammonia oxidised per day.

ammonia loads (Gullicks & Cleasby, 1986).

The Water Research Centre produced a computer model known as the Sewage Treatment Optimisation Model or STOM (WRc, 1987). This utilises two semi-empirical formulae to predict the nitrification performance of a trickling filter, based on the assumption that ammonia is assimilated by heterotrophs at a rate proportional to the BOD removal. However, this model was produced at a time when there were few data available on purpose-built nitrifying filters in the UK, so it is based on the performance of traditionally designed filters. The model uses a high value for the ammonia halfsaturation constant in order to fit predictions to observed data, but this assumption leads

to an under-estimation of filter performance at low ammonia loadings.

However, none of these models take account of the predation of biomass by higher organisms, the effects of diffusion on pH, or the problems caused by diurnal variations in the ammonia load to the filter (Gujer  $\&$  Erni, 1978). The incorporation of all of these

parameters into a design calculation would be impossibly complex, so many designers rely on a pilot-scale trial to verify a full-scale design.

## 2.6 Other applications of trickling filters

A modification of the basic trickling filter is the biofilm -controlled nitrifying trickling filter (BCNTF) (Parker *et al.*, 1989). The BCNTF consists of a tall filter  $(6 - 7 m)$ 

containing a cross-flow plastic medium, which is flooded at intervals of about one week. The flooding cycle inhibits the growth of biofilm-grazing organisms, particularly filter flies, which have been shown to consume sufficient biomass to have a significant deleterious effect on nitrification performance (Boller & Gujer, 1986).

It has recently been suggested that a nitrifying trickling filter preceded by anaerobic treatment might provide an efficient method for treating municipal waste water with minimal sludge production (Mergaert *et al.*, 1992). The principle of operation is that the

Another application is the use of an aerated solids-contact tank (ASC) downstream of the trickling filter (Norris *et al.*, 1982). This combined system, sometimes called the trickling filter/ solids contact process (TF/SC) has been shown to be cheaper in terms of capital, operating and maintenance costs than conventional activate sludge, rotating biological contactors, or combined activated sludge/ trickling filter processes (Lorence & Schroedel, 1990; Matasci *et al.*, 1986; Parker, 1987; Parker *et al.*, 1990). The ASC contains fine-bubble aeration, which is reported to promote flocculation of solids washed out of the trickling filter. The hydraulic residence time in the ASC is only 10 60 minutes, although the solids retention time is increased by subsequent settlement and sludge return.

anaerobic stage breaks down complex organic compounds and removes  $70$ ganic compounds and removes  $70 - 80\%$  of the BOD. The trickling filter would then remove most of the residual BOD, solids and ammonia. The process would rely on a high influent COD  $(>500 \text{ mg.dim}^3)$  and would be sensitive to shock loads or toxic inhibitors, but it is estimated that sludge production would be up to 80% less than for aerobic treatment and energy costs would be reduced.

## Chapter 3: Equipment, materials & methods

During the course of this research, work was carried out at laboratory, pilot and full scale. Laboratory scale experiments were devised to investigate the characteristics and responses of nitrifying bacteria in a controlled environment. Pilot scale research was used to attempt to validate the laboratory scale work, and to determine the behaviour of

Cranfield sewage treatment works (STW) serves a small catchment area covering Cranfield Institute of Technology and the associated housing. The wastewater is relatively weak and mainly of domestic origin, although there is a small trade

the organisms under more realistic conditions. The pilot scale results were corroborated by comparison with a full scale nitrifying filter on the same site.

## 3.1 Cranfield sewage treatment works

## 3.1.1 General description of the works

settlement, three single-pass biological filters containing a plastic random medium and humus settlement.

A diagram of the sewage works prior to 1992 (also showing the sampling points numbered within circles) is given in Figure 3.1. Sampling point 1 represents the settled

contribution from research laboratories on campus, a photographic laboratory and a metal plating workshop.

The nature of the catchment results in highly variable loads to the sewage works. The maximum population on site is estimated at 3,500, but this number drops to approximately 1,000 during weekends. Oily run-off from the airfield and occasional spillages can cause a considerable increase in the pollution loads reaching the sewage treatment works which may last for several days.

The works was extensively modified in the early 1980's to incorporate primary

influent to the works, point 2 is the recycled effluent from the first filter pass, and points 3 and 4 are respectively the final effluent before and after the aeration ditch.

Wastewater from the catchment area drains into an ejector station, from where it is pumped into the primary settlement tank via an inlet sump. The inlet sump serves the purpose of reducing surges when pumping starts.



#### Figure 3.1: Diagram of Cranfield sewage treatment works (1984-1991)

### 3.1.2 Primary settlement

The primary settlement tank is a single pyramidal unit of the Dortmund type. Dortmund tanks are designed to allow an extended sludge retention in order that the sludge volume is reduced due to anaerobic degradation. Sludge is removed manually through a bellmouth into the storage tanks, which were originally designed as settlement tanks.

The sludge storage tanks are emptied on a weekly basis by tanker.

The scum-boards on the tank are of insufficient depth, such that a high degree of solids loss occurs when scum is manually scraped off the tank, or when the sludge rises due to gas production. The resultant frequent blockage of the filter distributor arms led to the installation of screening sacks in the siphon chambers. These remove solids larger than approximately 3mm and have proved very effective, although the sacks require

> No. of ejectors Capacity (each) No. of primary tanks Type Dimensions Upflow velocity 4 10 dm<sup>3</sup>.s<sup>-1</sup> for 30 seconds every minute  $\mathbf{l}$ Pyramidal, Dortmund 8.6 x 8.6 x 7.9 m depth (I m sidewall)  $0.5$  m.h<sup>-1</sup> (at 10 dm<sup>3</sup>.s<sup>-1</sup>)

> > $Mean$  0.17  $m.h^{-1}$

frequent changing.

The basic details of the inlet works and primary settlement are as follows:

There are three trickling filters at Cranfield STW, each dosed through rotating distributor arms driven by a siphon in the distribution chamber. Prior to the major alterations completed in 1982, the filters contained mineral media and had a history of excessive biological growth leading to filter ponding. The medium was replaced in 1982 with Etapak 160, a random plastic medium manufactured by ETA Process Plant Ltd.

#### 3.1.3 Biological filtration

Until an ammoniacal nitrogen discharge consent of 10 mg.dm<sup>-3</sup> was imposed in 1984, all three filters were operated in parallel. In an attempt to achieve this standard, one of the biological filters was converted to a nitrifying filter by pumping a proportion of the unsettled secondary effluent back to the distribution chamber. The recycled flow is approximately 70% of the total flow.

Basic details of the biological filters are as follows:

No. of filters Mode of operation Dimensions Media (upper 1.60 m) (lower 0.23 m) Distributors Hydraulic load  $(1<sup>st</sup>$  stage) Organic load  $(1<sup>st</sup> stage)$ Hydraulic load (2<sup>nd</sup>)  $\text{magC}$ ) 3 Stage 1: two filters in parallel Stage 2: recycle through one filter  $12.2$  m diameter,  $1.83$  m deep Etapak 160 (SSA 150 m<sup>2</sup>.m<sup>-3</sup>) 100-150 mm grade stone Rotary, siphon driven Mean  $0.77 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ Mean  $0.087 \text{ kg.m}^{-3}$ .d<sup>-1</sup> Mean  $1.1 \text{ m}^3 \text{.m}^{-3} \text{.d}^{-1}$ 

#### 3.1.4 Humus settlement

with nitrified recycled effluent in the humus tank, there is a severe problem of denitrification. The humus tank is not fitted with scum boards and there is consequently a high degree of solids loss due to denitrifying sludge producing gas and floating to the surface of the tank.

Effluent from the trickling filters drains into a pyramidal humus tank, of the same

dimensions as the primary tank. Due to the blending of non-nitrified single pass effluent

Sludge removal was originally achieved manually, but an automatic system was installed in 1990 to enable more frequent de-sludging in an attempt to alleviate the solids loss. Sludge is pumped to the head of the works for co-settlement with the crude wastewater.

Clarified effluent gravitates through a flow recorder into an aeration ditch which was added to the works in about 1975. The aeration ditch consists of a 40m long shallow channel prior to the final discharge, containing 25mm blast furnace slag. This was intended to aerate the effluent and provide some further biological treatment. In practice the solids loading is too high so the slag rapidly clogged and the analyses of samples

before and after the ditch currently show negligible differences.

The effluent from the works is discharged into Chicheley Brook, where it constitutes between 20% and 50% of the total dry weather flow. Chicheley Brook runs across mainly arable land for approximately 9 miles before discharging into the River Great Ouse at Newport Pagnell. The current local policy requires a minimum of Class 2 water quality *(ie* the water should be capable of supporting coarse fishing).

Current and recent effluent consent standards for the works are given in Table 3.1,

Between 1984 and 1992 there were no significant modifications to the sewage treatment works. In June 1992 (after the pilot-scale research reported here was completed) a new tertiary nitrifying trickling filter was commissioned, which will be described in a later section.

together with anticipated consents for the next two years.

#### Table 3.1: Effluent consents for Cranfield sewage works.



#### anticipated discharge consents.

The consent covering the period 1988-1991 was a temporarily relaxed consent to allow for the construction of a nitrification stage in the treatment process.

Comprehensive data on the performance of Cranfield sewage works have been gathered by the Biotechnology Centre since September 1986.

## 3.2 Pilot scale trickling filters.

3.2.1 Pilot scale filter construction

Four pilot scale trickling filters were constructed at the sewage works for this research (Plate 1). A diagram of a filter is given in Figure 3.2. The larger of the filters was manufactured specifically for this research by ETA Process Plant Ltd. The walls were polypropylene of 4mm thickness. The filter was fitted with three sampling ports, eight shuttered ventilation holes around the base and a 38 mm diameter outlet pipe. The medium was supported on a coarse PVC grill positioned 300 mm above the base of the



#### Plate 1: Pilot scale trickling filters



filter. The base was filled with concrete to prevent the accumulation of water and debris.

The other three filters were adapted from pump sumps, provided by Entec Pollution Control Ltd. They were manufactured from fibreglass of thickness 2-4mm. Holes were drilled around the base to provide ventilation, and the plastic media were supported on a fine grid of plastic-coated wire. Several large blocks of concrete were positioned in the base of the blast furnace slag, filter to provide additional support. Two sampling ports were fitted to each filter, consisting of 500 mm lengths of 38 mm diameter uPVC pipe cut in half to form a collecting trough.

The ventilation holes in the bases of the filters provided an aeration area equivalent to 6.2% of the upper surface for the three small filters and 7.6% for the large filter. No lt: ý evidence of dissolved oxygen deficiency in the effluent was noted during the study.

The dimensions and contents of the four pilot scale filters are summarised in Table 3.2

Filter number				
Medium	Etapak 210	Blast furnace slag	Etapak 160	Flocor RS
Diameter $(m)$	$\bm{\mathsf{I}}$ . $\bm{4}$	).9		0.9
Medium depth $(m)$	1.8	1.2		
Aspect ratio	0.78	$0.75\,$	0.75	0.75
Medium volume $(m^3)$	2.77	0.76	0.76	0.76
Surface area $(m^2)$	1.54	0.64	0.64	0.64

Table 3.2: Summary of the pilot scale filter characteristics.

## 3.2.2 Filter media

The characteristics of the four media are given in Table 3.3. The media are also shown

in Plate 2.

#### Table 3.3: Characteristics of the media.



\* typical characteristics.



#### Plate 2: The media used in the pilot scale filters

comparison between the results obtained at pilot and full scale. The Etapak media were manufactured and supplied by ETA Process Plant Ltd. of Rugeley, Staffs.

Etapak 160 was selected for this research because it is used in the full scale filters at Cranfield STW. Its inclusion in the pilot scale study therefore provides a useful

Blast furnace slag was selected as the best example of a mineral medium for nitrification due to its higher specific surface area than other mineral media. It has been widely used for many years as a trickling filter medium, usually in the larger 50mm or 100mm grades for carbonaceous oxidation (BOD removal) (Wilkinson, 1958). More recently, it has also been used as a medium for nitrification.

It was evaluated for use in large filters at Wanlip STW, Leicester (Upton & Cartwright,

1984) and at Davyhulme STW, Manchester (Dolan *et al.*, 1990). Blast furnace slag was also selected for use in a large nitrifying filter at Burnley STW (Hill et al., 1992). Although cheap and readily obtainable, mineral media can incur high construction costs in situations where ground conditions cannot support the weight of a high density

#### medium.

Blast furnace slag is a byproduct of the steel industry from the high-temperature fusion of fluxing stone with coke ash and the siliceous and aluminous residues remaining after reduction and separation of iron from the ore (Emery, 1978). The precise composition and nature of slag varies between sources and is dependent upon the composition of iron ore, the iron extraction process and the method of cooling the slag. General reviews of

the characteristics of blast furnace slag may be found in Nurse and Midgley (1951) and Scott et al. (1986).

Etapak 210 was included in the study in order to evaluate the effects of the specific surface area and medium geometry, since these are the characteristics in which Etapak 160 and 210 differ.

Flocor RS provided the opportunity to evaluate PVC as a material for biomass attachment, and to examine the effectiveness of a corrugated cylinder compared to the complex shapes of the Etapak media. Flocor media are manufactured by BS Flocor Ltd of Telford. The medium used in this research was supplied by Entec Pollution Control Ltd. of Andover.

## 3.2.3 Pipework and wastewater distribution

Wastewater was pumped from the siphon chamber of the sewage treatment works using a Flygt model CS3041-250 submersible centrifugal pump. The water was carried in a 50mm diameter pipe to an in-line strainer to remove fibres and any solid particles larger than 2mm. The pipe then branched into a 25mm diameter pipe to the large Etapak 210

filter and a 38mm diameter pipe to the other three filters. The latter pipe was progressively narrowed down to 12mm diameter in order to maintain an equal pressure head and ensure an even distribution of wastewater to all three smaller filters.

The feed to the large filter passed through a 25mm diameter ball valve, a 4-40 dm<sup>3</sup>/min

flowmeter and a 12mm diameter solenoid valve before distribution by a single, downward-facing nozzle (type BNM136). The feed to the other three filters was split into three streams, each passing through  $38$ mm diaphragm valves, a 1-10 dm<sup>3</sup>/min flowmeter and a 12mm, solenoid valve before distribution by similar but smaller nozzles (type BNM46) (Plates 3& 4).

positioned, pointed downwards, just above the surface of the media at a height calculated to give optimal distribution over the entire surface.

Both types of nozzles are designed to create a solid-cone spray pattern and were

The nozzles were manufactured in brass by Delavan Ltd. The pipes, valves and the in-line strainer were made by Durapipe Plastics Ltd., Cannock. The flowmeters were manufactured by Platon Flowbits Ltd., Basingstoke (model PG). The solenoid valves were made by Vento Solenoids Ltd. and were controlled by cyclic relay timers made by RS Components.



#### Plate 3: Effluent distribution in the blast furnace slag filter

At the beginning of the study, the daily flow was measured at the effluent of each filter using a tipping trough (designed for the project and built by Knight Engineering, Thruxton). These were used as a check on the accuracy of the in-line flowmeters, and to verify that the flow through the filters was constant over a 24-hour period. The tipping troughs are shown operating in Plate 5. However, it was found that the tipping troughs were prone to jamming, and that the flow remained sufficiently constant for continual measurement of the flow to be unnecessary, so their use was abandoned after three months.

#### 3.3 Full scale nitrifying trickling filter

Cranfield STW is a typical example of a works where a tertiary nitrifying trickling filter provides the ideal upgrade to meet improved ammonia standards. The secondary effluent is well oxidised but is not reliably nitrified. A trickling filter provides the means to



Plate 4: Effluent distribution in the Etapak 160 filter



Plate 5: Tipping troughs during the filling phase of operation

nitrify the effluent without the expense and operational requirements of an activated sludge process.

The design of the new nitrifying trickling filter for Cranfield sewage treatment works was based upon results obtained during this research and from published experience from Wanlip and Burnley sewage treatment works (Hill et al., 1992; Upton & Cartwright, 1984). Construction of the filter began in December 1991 and it began receiving water on 25<sup>th</sup> June 1992. A revised process schematic of Cranfield sewage treatment works, incorporating the tertiary nitrifying trickling filter and associated ancillaries, is shown in Figure 3.3.

The filter was built above ground and constructed of reinforced concrete. It is divided by a central partition into two sections measuring 5m x 5m with a minimum medium depth of 3m. Effluent from the nitrifying filter passes through a new circular settling tank of diameter 8m fitted with a fixed bridge scraper mechanism.

Since commissioning the nitrifying filter, the sampling points for the routine monitoring of the treatment works have been changed. The revised sampling points are shown on Figure 3.3 and are denoted by the encircled numbers 1-4. Sampling point I is the settled influent, 2 is the humus tank effluent, 3 is the effluent from the nitrifying filter, blended with any underflow which has bypassed the nitrifying filter, and 4 is the final effluent.

The base of the filter is open on two sides to provide aeration through an area of 1.5% of the filter surface area.

The filter contains a layer of Naylor filter-floor tiles supporting the blast furnace slag medium, which is predominantly 25mm grade, but with 300mm deep layers of 50mm grade slag at the base and surface of the filter to prevent medium blockage and ensure adequate drainage. Settled secondary effluent from the humus tank is applied to the filter at a rate of 3  $m<sup>3</sup>$ . $m<sup>-3</sup>$ . day<sup>-1</sup>. A recycle system is built into the pumping chamber to ensure that this application rate is maintained during periods of low flow through the treatment works.

In order to study biological activity within the filter, the filter was equipped with six aluminium neutron probe tubes to monitor biomass accumulation and two intermediatedepth sampling systems. These each consist of a section of 300 mm diameter plastic pipe mounted vertically in the centre of the filter, with sections of half-pipe feeding into

Effluent is applied to the filter via a grid of eighteen upward-facing nozzles (Delavan RA160), which are designed to discharge large droplets at a wide spray angle (140'). This system, coupled with a low wind shield around the filter, minimizes the risk of spray drift which has been a problem at other similar installations. The nozzles are equipped with quick-release brackets to facilitate cleaning, and contain no internal vanes. In practice, only one incidence of partial blockage was reported in the first three months of operation.


To assess the comparative performance of the filter media at removing BOD, the pilot scale filters were initially loaded with primary settled wastewater. Nitrification requires a BOD concentration below a certain maximum concentration, so this stage of the

research was designed to establish the capacities of the media to nitrify when subjected to BOD concentrations above the maximum concentration. Application of a concentrated wastewater also enabled a rapid accumulation of biomass on the filters, reducing the start-up time.

it at intervals of 300 mm down the depth of the filter. Samples may be collected from the intermediate depths by lowering a small vessel down the central shaft.

3.4 Operation of the pilot-scale filters

#### 3.4.1 Start-up and phase I operation

The pilot plant began receiving primary settled wastewater on 31st August 1990. The application rate was set at  $1.4 \text{ m}^3.\text{m}^{-3}.\text{day}^{-1}$ , approximately double the low volumetric loading of the full scale filters.

There were initially some problems of fouling in the flowmeters, valves and nozzles, leading to unreliable dosing. Problems of this type are frequently encountered in pilot scale research due to the inherent difficulties involved in passing a non-homogeneous

liquid through scaled-down equipment (Wheatley, 1976). These problems were ameliorated by fitting in-line filters to the wastewater feed pipes and solenoid valves to induce periodic dosing when low hydraulic loading rates were employed.

The flow rate through each filter was checked on a daily basis, both by measuring the instantaneous flow and using the tipping troughs to record the flow over a 24-hour period. Following the fitting of the in-line strainers and solenoid valves, the flow rate remained constant for long periods and the use of the tipping troughs was abandoned.

Periodic dosing mimics the conditions found in a filter fed by a rotating arm, gravity siphon or tipping trough system. Periodic dosing also enables the flow-control valves (diaphragm and ball valves) to be opened further, thereby reducing the likelihood of fouling while still operating at a low hydraulic loading rate. The combination of periodic dosing, in-line filters and the choice of large diameter nozzles, meant that a consistent

flow rate could be maintained over at least a two-week period.

 $\overline{\phantom{a}}$ 

#### 3.4.2 Phase 2 operation

For this period of research, the feed to the filters was switched over to secondary

effluent *(ie* following a single pass through one of the full scale trickling filters for  $\ddot{\phantom{0}}$ carbonaceous oxidation). After an initial period when the hydraulic loading to the filters was varied over the range  $1 - 10 \text{ m}^3 \cdot \text{m}^{-3}$ . day<sup>-1</sup>, it was decided to use a loading of 6 m<sup>3</sup>. m<sup>-1</sup> <sup>3</sup>.day<sup>-1</sup> as the basis for long-term operation of the filters.

This loading was selected following comparisons with full scale nitrifying filters at Burnley and Wanlip, and after comparison of the results obtained with the pilot scale filters at variable loadings. Furthermore, a loading of  $6 \text{ m}^3 \text{.m}^{-3}$ . day<sup>-1</sup> gave good effluent distribution over the filter surfaces using the spray nozzles and enabled continuous dosing, thereby eliminating the need for the solenoid valves which had been liable to blockage.

The in-line strainer required cleaning approximately every three months, although the cleaning frequency was increased during Spring and early Summer when large numbers of filter snails were washed from the trickling filters in the full scale works. Due to the absence of any settling between the outlet of the full scale filters and the pump supplying the pilot scale filters, the snail shells accumulated in the strainer, eventually contributing to its blockage.

Since a reliable automatic sampler was not available, samples were taken manually as follows:

Fouling of the pipes, valves and flowmeters by biological growth was minimised by periodically stopping the flow, removing the nozzles and then re-starting the pump with all valves closed except one. The resulting high flow rate through the open pipe was usually sufficient to shear off most of the growth which could be collected in a bucket and disposed of. This process was repeated in sequence for all four pipelines, usually on a bi-monthly basis. Residual fouling was removed annually by dismantling and cleaning the flowmeters and solenoid valves and passing a test-tube brush through the intermediate pipework.



Samples from the full scale works were composited from three composites taken during

the day (8 a.m., mid-day and 4 p.m.). Samples were taken at intervals of 6 days in order to eliminate any regular weekly patterns in discharges to the sewer while still maintaining a routine sampling schedule.

For the pilot scale research, it was decided that snap samples would be appropriate since the flow through the filters was constant and any fluctuations in the wastewater strength would be reduced by the balancing effects of the primary settlement tank and sewer network (retention time at least 18 hours at average flow). Samples of approximately 550 cm' were taken from the outlet pipes of the filters and stored, with the minimum of air in the sample bottle, for an hour to simulate the effects of settlement. Settled samples were routinely analysed for ammonia, nitrate, suspended solids, turbidity and B.O.D. as described in Section 3.6. The interval between successive samples was randomised to eliminate any weekly trends in wastewater characteristics, but was usually between 1 and 6 days.

The are four standard methods available for the determination of ammonia nitrogen in wastewater. The Nesslerisation techniques were rejected due to their inaccuracy and susceptibility to interference. The distillation and titration method was used for the majority of this research, although an ion-selective electrode was also assessed.

#### 3.6.1a Distillation and titration

Ammonia is quantitatively recovered from the sample by distillation under alkaline conditions, and collected in a solution of boric acid. Ammonia is then determined by titration against sulphamic acid solution. This is a standard technique (D.o.E., 1972) and was selected as the most suitable method for most of the work because, although it is slow, it is accurate, quick to set up and can be left unattended. The standard procedure

uses hydrochloric acid for titration, but for this research sulphamic acid was used instead because it can be prepared as a primary standard from the solid form, thereby avoiding reliance on the concentration of a diluted liquid acid. Results were expressed as mg.dm<sup>-3</sup> of ammoniacal nitrogen (NH<sub>4</sub>-N) and are subject to errors of  $\pm 5\%$ .

#### 3.6.1b Ammonia electrode

This method measures both nitrate and nitrite using the residue from the determination of ammonia by titration (D.o.E., 1972). However the concentration of nitrite in most wastewater is negligible, so the technique is widely used for nitrate measurement. Oxidised nitrogen is reduced to ammonia by heating with Devarda's alloy (composition approximately 45% Al, 50% Cu and 5% Zn). The ammonia is distilled off and measured by titration as in Section 3.6.1a. Results were expressed in mg.dm<sup>-3</sup> of oxidised nitrogen and are subject to errors of  $\pm 10\%$ .

The sample is made alkaline by the addition of concentrated NaOH, which converts ammonium ions present into ammonia gas. The partial pressure of the dissolved gas is then measured using an ion-selective ammonia electrode (Kent Industrial Measurements Ltd. model 8002-8) according to the method of Evans & Partridge (Evans & Partridge, 1974). This method was tried alongside the titration method for a period of three weeks, but was found to give inconsistent results and to require frequent recalibration of the electrode. Its use was therefore discontinued.

#### 3.6.2 Total oxidised nitrogen

The principle of the Dilution Method for B.O.D. determination is that the dissolved oxygen concentration of the diluted sample is measured before and after incubation for five days in the dark at  $20^{\circ}$ C. Nitrification was inhibited in order that the oxygen depletion was attributable only to biological oxidation of organic matter in the sample.



The method used is a standard technique (D.o.E., 1972) and allylthiourea (ATU) was used as the nitrification inhibitor. The measurement of dissolved oxygen concentrations was performed by the iodometric titration technique (D.o.E., 1972). This is based on the oxidation of iodide to iodine by manganous hydroxides, and subsequent titration to determine the amount of iodine. This method is also known as the Winkler analysis. Results were expressed in mg.dm<sup>-3</sup> of B.O.D. and are subject to errors of  $\pm 10\%$ .

An appropriate volume of the sample was filtered using a Hartley funnel through a pre-weighed 70 mm glass fibre filter paper (Whatman GF/C) with the smooth side of the paper facing upwards. The filter paper was then dried in an oven at 105'C for two hours. The suspended solids was determined by the weight difference and results were expressed in mg.dm<sup>-3</sup> (D.o.E., 1972; Melbourne, 1964). The method is subject to errors of  $\pm$ 5%.



This was determined by the standard procedure (D.o.E., 1972) using a Hach model 43900 Ratio/XR Turbidimeter. This instrument measures nephelometric turbidity units (NTU) of a sample in a standard cell of approximately 30  $cm<sup>3</sup>$  (part no. 20849-00). Nephelometers measure the ratio of the intensity of the scattered light at 90' to the intensity of the transmitted light. They are more suitable for this application than absorptiometers due to their higher sensitivity and reduced interference from coloured compounds in the sample. The limit of precision for turbidity is  $\pm 5\%$ .

3.7 Other determinands

#### $3.7.1$  pH

## The pH was measured electrometrically using either a Gallenkamp "pH Stick" combined

electrode or a Kent pH electrode connected to a Beckman Monitor 11. The meter was calibrated daily with standard buffers at pH4, 7 and 10 to ensure an accuracy of  $\pm 0.1$ pH.

#### 3.7.2 Total solids and volatile solids

Total solids was determined using the standard procedure (D.o.E., 1972) involving the

drying of a measured volume of sample in a pre-weighed porcelain dish at 105'C for at least three hours, or until constant weight was achieved. Results were expressed as milligrams of solids per dm<sup>3</sup> of sample (mg.dm<sup>-3</sup>) and are subject to errors of  $\pm 10\%$ .

The proportion of volatile matter within the sample was then determined by heating the dish in a Gallenkamp muffle furnace at  $600^{\circ}$ C for two hours and measuring the difference in weights. The sample was allowed to cool to room temperature in a desiccator before each weighing. Results were expressed as volatile solids as a percentage of total solids and are subject to errors of  $\pm 10\%$ .

The temperature (in  ${}^{\circ}C$ ) of the pilot scale filters was monitored continuously using a Grant Instruments "Squirrel" Data Logger model 1202 with eight thermistors. The thermistors were positioned in the intermediate sampling ports and at the bases of the filters to measure the temperature at the centre and outlet of each filter. Data accumulated by the data logger were downloaded directly to a computer spreadsheet using Grant Instruments' transfer software. The accuracy of thermistors was periodically checked using a water bath to ensure a precision of  $\pm 0.2$ °C.

The only problems encountered with this equipment were due to damage to the thermistor cables, presumably by rats. Despite efforts to protect the cables, periodic damage continued to occur, hence some data have been discarded.

## 3.7.4 Retention time

The retention times were measured using the tracer technique. Lithium (as LiCI) was selected for use as the tracer because:

1) It was not present in the wastewater at above  $0.1$  mg.dm<sup>-3</sup>.

2) Lithium can be detected quickly, accurately and in low concentrations by flame photometry.

- 3) It does not react with, or adsorb to, the constituents of the biofilm to any significant extent.
- 4) It is non-toxic at these concentrations and does not affect the biofilm (Anderson *et al.*, 1991; Gray, 1981; Tomlinson & Chambers, 1979).
- 5) Lithium chloride is cheap, stable and readily soluble in water.

Factor 3 is particularly important since any retention time experiment is effectively measuring the chromatographic characteristics of the selected tracer. Therefore it was essential to select a tracer which would remain dissolved in water and not be attracted

For the retention time measurements on the full scale filters, the flow rate was monitored continuously using an ultrasonic flow meter connected to a data logger

to, or repelled by the media in any way.

Dyes and ammonium salts were rejected as tracers due to the complex nature of wastewater, and radio-isotopes were considered too difficult to handle.

In each experiment, an appropriate amount of LiCl was dissolved in a minimal volume of wastewater and added to the feed to the filter. The amount of LiCl was determined by estimating the retention time of the filter and calculating the mass of lithium to give a peak effluent concentration of approximately 30 mg.  $dm^{-3}$ . Samples of the filter effluent were then taken at suitable intervals from the outlet stream immediately below the filter.

This method minimizes the time lags due to pipework, while ensuring that the tracer is

administered in exactly the same way as normal wastewater.

(Warren Jones "Sentinel 420"). The flow to the pilot scale filters was sufficiently constant not to require continuous monitoring, so measurements were taken before and after the tracer experiments.

Samples were allowed to settle for an hour before measuring the lithium concentration using a flame photometer (Ciba-Corning model 410). Readings taken from the flame photometer were converted into actual lithium concentrations using a calibration curve

similar to the graph shown in Figure A2.1 (Appendix 2). Calibration curves were prepared by diluting a 1000 mg.dm<sup>-3</sup> lithium stock solution with wastewater effluent, collected before the start of the experiments. A new calibration curve was constructed for each set of experiments in case of interference from other substances present in the wastewater.

The retention time was obtained from the results by plotting the lithium concentration against time, then calculating the geometric mean of the curve (the centre of gravity of the curve) following the method used by Truesdale (1953) and Levenspiel (1972). The

tracer recovery achieved was typically 80-90%.

#### 3.7.5 Biofilrn accumulation

The technique selected to monitor the growth of biomass in the filters was the neutron probe method. The neutron probe measures the moisture volume fraction in a sphere of approximately 300 mm radius. To monitor the accumulation of biomass it is necessary to assume that the moisture volume fraction of the filter medium is proportional to the amount of biomass present.

Although this technique has been shown by Wheatley (Wheatley, 1976) to be unreliable in some cases, it has been used effectively by a number of other workers (Bruce  $\&$ Merkens, 1970; Upton & Cartwright, 1984). The instrument used was an Institute of Hydrology Neutron Probe Mark 11 L, manufactured by Didcot Instrument Company (Bell, 1969). Aluminium alloy access tubes (41.9 mm inside diameter, 44.5 mm outside

diameter) were positioned in each filter at a distance of at least 200 mm from the side wall to prevent interference. The tubes were kept plugged at the top with rubber bungs when not in use and the bases were left open for drainage.

times between the different media and measured only the water which is trapped either within organisms or in pockets of the media.

When the neutron probe readings were taken, the wastewater feed to the filter was turned off and the filter was allowed to drain for at least an hour until the effluent flow was insignificant. This method therefore eliminated the variations in liquid retention

The measurements taken from the neutron probe were converted into percentage moisture volume fractions (%MVF) by dividing by a blank reading, obtained by immersing the probe in a large tank of water.

#### 3.7.6 Air convection through the pilot scale filters

Each set of readings was performed when the wind speed was less than  $0.7 \text{ m.s}^{-1}$ , and careful note was taken of air and filter temperatures, as well as wind direction. To eliminate the effects of wind gusts on the results, a large number of readings were taken,

The rate of air convection through the pilot scale filters was measured using a laboratory anemometer (Airflow Developments Ltd model LCA6000). The anemometer was positioned over the pipe at the base of the filters to measure the rate of air entering the filter. The anemometer vanes were the same diameter as the outlet pipe, and all other ventilation holes around the base of the filters were sealed, thereby ensuring effective measurement.

obviously erroneous readings were discounted, and the average of the remainder was

taken.

# 3.8 Laboratory nitrification experiments

In order to examine the kinetics of nitrification under controlled conditions, it was decided to develop a culture with a stable enriched proportion of nitrifying organisms. This culture would then be used as an inoculum for laboratory experiments to determine the rate of nitrification under various conditions of pH, temperature and substrate concentration.

Initial attempts were made to obtain enriched cultures of nitrifying organisms from sewage works effluent and from activated sludge, but following various problems this approach was abandoned and pure cultures were obtained from the National Collections of Industrial and Marine Bacteria Ltd (N. C. I. M. B. ) culture collection.

#### 3.8.1 Selection in nutrient medium

A 1 dm<sup>3</sup> inoculum of Cranfield sewage works final effluent was suspended in 9 dm<sup>3</sup> of

a medium containing  $Fe^{3+}$ ,  $Mg^{2+}$ ,  $Ca^{2+}$ , ,  $\mathcal{L}_{\mathcal{A}}$ Cl<sup>-</sup>,  $PO_4$ <sup>3-</sup> and  $SO_4$ <sup>2-</sup> supplements dissolved in distilled water. This is a simple mineral-supplemented medium based on the medium used in B.O.D. measurement. The suspension was fed daily with ammonia, as  $10 \text{ cm}^3$ of 20 g.dm<sup>-3</sup> ammonium chloride solution, to give a daily addition of 5 mg.dm<sup>-3</sup> of ammoniacal nitrogen. In addition to the carbon sources present in the inoculum, methanoate (as 10 cm<sup>3</sup> of 20 g.dm<sup>3</sup> sodium methanoate solution) was added weekly. Methanoate is a single-carbon source which can be utilised by chemolithotrophs (including nitrifiers) but not by heterotrophic bacteria. The precise composition of the medium is given in Appendix 3.

Approximately 10 dm<sup>3</sup> of the medium were sparged with air at 2 dm<sup>3</sup>.minute<sup>-1</sup> (0.2) volumes. volume<sup>-1</sup>. minute<sup>-1</sup>) in a perspex cylinder of diameter 140 mm and height 800 mm. The cylinder was covered with aluminium foil to maintain total darkness, thereby inhibiting the growth of algae.

# 3.8.2 Selection in sewage works effluent

About 10 dm' of Cranfield STW final effluent were aerated in a perspex cylinder as above. Initially, ammonium chloride (20 g.dm<sup>-3</sup>) was used as the ammonia source but this was later changed to urea (20 g.dm<sup>-3</sup>) following problems with stabilisation of the pH. The pH was kept close to 8 by frequent additions of 5 M sodium hydroxide. Sodium formate was added periodically to a concentration of 5 mg.dm<sup>-3</sup> to provide a

single-carbon carbon source.

## 3.8.3 Selection in activated sludge using settled wastewater as a feed

The medium used for the culture of nitrifying organisms was based on a culture medium used by Srinath and Prakesam (Prakasam *et al.*, 1972) and on the medium suggested in

the N.C.I.M.B. Catalogue of Strains, 1990. The composition of the medium is detailed in Appendix 3.

Pure cultures of *Nitrosomonas europaea* were grown in serial culture in this medium, starting in 250 cm<sup>3</sup> conical flasks incubated at  $30^{\circ}$ C on a rotary incubator (200 rpm).

10 dm' of activated sludge from Cotton Valley Sewage Works, Milton Keynes, was vigorously aerated in a  $25 \text{ dm}^3$  plastic drum. The Milton Keynes plant is a nitrifying activated sludge plant. The mixed liquor suspended solids (MLSS) was monitored daily. The sludge was fed daily by allowing the sludge to settle, pouring off half the supernatant, and replacing with the same volume of Cranfield settled wastewater to give a reed to micro-organism ratio  $(F:M)$  of 0.05  $0.1$  mg B.O.D. (mg MLSS). The only sludge wastage was due to poorly settled sludge being poured off with the supernatant. This technique produces a long sludge age which reduces the competition against slow-growing nitrifying organisms. The plastic drum was kept covered throughout to prevent the growth of algae.

## 3.8.4 Growth of nitrifying organisms in pure culture

Cultures were transferred under aseptic conditions to progressively larger flasks up to 2.5 dm<sup>3</sup>, from which samples of pure culture were taken for the kinetics experiments.

## 3.8.5 Experiments on nitrification kinetics

For each experiment, approximately 1 dm<sup>3</sup> of medium was centrifuged in 250 cm<sup>3</sup> aliquots at a relative centrifugal field (RCF) of 4420 g (at  $r_{\text{max}}$ ) for 15 minutes. The pelleted bacteria were washed with 0.15 M NaCl, re-centrifuged and then suspended in 2.5 dm<sup>3</sup> of 0.05 M potassium phosphate buffer at pH 7.6 and 32 $^{\circ}$ C (except for the experiments on variable pH, when different buffer concentrations were used).

The inoculum suspension was then placed in an incubator at  $32^{\circ}$ C to maintain the temperature until the inoculum was required (maximum I hour).

No attempt was made to ensure absolute asepticity of the solutions since the media used (with the exception of the experiment involving glucose/glutamic acid) were too simple

For the experiments on the effects of temperature, twenty identical 250 cm<sup>3</sup> flasks were prepared, each containing  $150 \text{ cm}^3$  of  $267 \text{ mg}$ .  $\text{dm}^3$  ammoniacal nitrogen (as ammonium sulphate) dissolved in phosphate buffer. Two flasks were placed in water baths within

to support significant bacterial growth over the short duration of the experiments.

Progress of the kinetics experiments was monitored by measuring the accumulation of nitrite in the medium. There are two major advantages in measuring nitrite production rather than ammonia removal: nitrite can be measured accurately and rapidly at low concentrations using a simple procedure (Appendix 3) and the determination of ammonia would be susceptible to interference by the uptake of ammonia by contaminants. However, nitrite cannot be used to follow nitrification in enriched or mixed cultures since nitratifying bacteria would be present.

#### 3.8.5a Effect of temperature

incubators at each of the various temperatures used for the experiment ( $0^{\circ}$ C,  $4^{\circ}$ C,  $10^{\circ}$ C, 15°C, 22°C, 25°C, 32°C, 37°C and 50°C). The remaining two flasks were incubated at  $22^{\circ}$ C for use as controls (no inoculum added). A further two flasks, containing only phosphate buffer (150 cm<sup>3</sup>), were incubated at  $22^{\circ}$ C for use as blanks (no substrate).

When the flasks had reached their respective temperatures, 50 cm<sup>3</sup> of Nitrosomonas inoculum was added to each flask, diluting the ammoniacal nitrogen concentration to

 $200 \text{ mg.dim}^3$ . Samples (10 cm<sup>3</sup>) were taken immediately and at intervals of 90 minutes thereafter, for the determination of nitrite (the product of ammonia oxidation by Nitrosomonas) as described in Appendix 3.

#### 3.8.5b Effect of PH

mg.dm<sup>-3</sup> ammoniacal nitrogen (as ammonium sulphate) and placed in an incubator at 32'C to equilibrate the temperature. Blank and control flasks were also prepared

Phosphate buffer solutions were made up according to Table 3.4 below, in order to give solutions of pH between 5.8 and 8 on addition of the inoculum in 50 cm<sup>3</sup> of 0.03 M phosphate buffer pH 6.95.

Table 3.4: Compositions of the Phosphate Buffer Solutions.



Duplicate flasks were filled with 150 cm' of phosphate buffer solution containing 267

(without substrate and inoculum respectively).

 $50 \text{ cm}^3$  of Nitrosomonas inoculum (in phosphate buffer) was added to each flask (except the control) and the flasks were replaced in the incubator at  $32^{\circ}$ C. A 10 cm<sup>3</sup> sample was taken immediately and at intervals of 90 minutes thereafter for the determination of nitrite as described in Appendix 3. The pH of the samples was periodically checked to

#### ensure that the experimental pH was being maintained.

#### 3.8.5c Effect of ammonia, glucose/glutamic acid and allylthiourea

For each set of experiments, 18 flasks were filled with  $150 \text{ cm}^3$  of phosphate buffer containing duplicates of 9 different concentrations of, respectively, ammoniacal nitrogen (as ammonium sulphate), D-glucose and L-glutamic acid, and allylthiourea. Flasks for  $\mathcal{L} = \mathcal{L} \times \mathcal{L} = \mathcal{L} \times \mathcal{L}$ the experiments involving glucose/glutamic acid and allylthiourea also contained 267  $mg.dim^{-3}$  ammoniacal nitrogen (as ammonium sulphate).

Flasks were incubated at 32°C. The progress of the reaction was monitored by

measuring the nitrite concentration using Griess-Ilosvay's reagents I and  $\Pi$  (Appendix

3).

#### 3.9 Data manioulation

Raw data were typed into a computer spreadsheet (Microsoft Works v.2.1) where the loads, rates and averages were calculated. Temperature data from the data logger were downloaded to Lotus 1-2-3 before exporting to Works. Graphs were plotted using Harvard Graphics v.3 and lines, where drawn, have been calculated by the software's

#### analysis of variance.

Where quoted, statistical analysis uses the t-distribution method for testing significance.

# Chapter 4: Results

This chapter will describe briefly the experiments performed at laboratory, pilot and full scale, and will indicate the main findings which led to further experiments. Detailed descriptions of the experimental equipment and methods can be found in Chapter 3. Full analyses of the experimental data are given in Chapters 5-7 and the raw data are

presented in Appendices 4 onwards.

### 4.1 Laboratory scale research

Laboratory scale research was undertaken to establish the response of nitrifying bacteria to changes in their habitat. While conditions in a laboratory experiment can never be truly representative of the natural habitat, the laboratory does permit single-variable controlled experimentation.

Initial work was focused on obtaining an enriched culture of nitrifying organisms for use

in kinetics experiments. However, the cultures did not achieve an adequate density of

nitrifiers so, after several attempts using different media, the approach was abandoned

and pure cultures of nitrifying organisms were obtained commercially.

## 4.1.1 Nitrifying culture enrichment experiments

The initial experiment was an attempt at enriching the nitrifier content of an inoculum of Cranfield STW effluent using a defined mineral medium. It was hoped that the use of a defined mineral media would eliminate the possibility of contamination of the kinetics experiments.

The experiment was performed as described in Section 3.8.1, and dosed with ammonia (as ammonium chloride). Samples were analysed for pH, ammonia and nitrate. Data are presented in Appendix 4. The conclusions drawn from this experiment are discussed in Section 5.1.1.

Since an enriched culture of nitrifying organisms was not obtained from the defined medium, nutrient deficiency was suspected. However, it was thought unnecessary to attempt to identify the deficiency, so a second experiment was performed using sewage works effluent as a non-defined growth medium. The methodology is described in Section 3.8.2, and the conclusions from the experiment are discussed in Section 5.1.2. Raw data are presented in Appendix 4.

While some enrichment of the nitrifying population was achieved by this means, the rate of nitrification was insufficient for use as an inoculum for kinetics experiments. To increase the activity of the culture, nitrifier enrichment was attempted in a culture of activated sludge as described in Section 3.8.3. The raw data are presented in Appendix 4 and the results are discussed in Section 5.1.3.

> temperature;  $\bullet$

- pH;  $\bullet$
- ammonia concentration;  $\bullet$
- B. O. D. concentration (as glucose/glutamic acid);  $\bullet$
- allylthiourea concentration.  $\bullet$

#### 4.1.2 Growth of pure cultures of nitrifying organisms

Pure cultures of nitrifying organisms, obtained commercially, were successfully grown

to a high cell concentration as described in Section 3.8.4. Observations made during the

culturing process are summarised in Section 5.2.

#### 4.1.3 Use of pure cultures for nitrification kinetics experiments

Nitrifying bacteria, grown in pure culture, were harvested as described in Section 3.8.5 for use as inocula in nitrification experiments. Several experiments were devised to establish the effect on nitrification in pure culture of the following variables:

Each set of experiments was performed in a similar way. Multiple flasks of Nitrosomonas europaea were incubated with only a single variable for each experiment. Each experiment contained both blanks and controls to prevent any interference.

The methodology is described in Sections 3.8.5(a), (b) and (c). Raw data are supplied in Appendix 5, and the results are discussed in Section 5.3.

4.2.1 Phase 1: Carbonaceous oxidation

During phase 1 of operation, covering the period 31<sup>st</sup> August 1990 to February 1991, the pilot scale filters were loaded with settled raw wastewater as described in Section 3.4.1. The purpose of this was to evaluate the performance of the media for BOD removal, and to reduce the start-up time for phase 2 by coating the media with a layer of attached biomass to provide a polysaccharide matrix to which other bacteria may adhere.

From February 1991 until the end of the pilot scale research period in September 1993, the pilot scale filters were dosed with recycled effluent from the sewage works *(i.e.* wastewater which had passed through the first stage of the full scale trickling filters). The BOD of this wastewater was typically below 20 mg.dm<sup>-3</sup>, which is regarded as a

The purpose of this phase of research was to evaluate the four media with regard to their suitability as media for nitrification, and to study the effects of loading and climatic conditions on the nitrification process.

The results obtained during this phase of research are discussed in Section 6.1 and the

raw data are presented in Appendix 6.

#### 4.2.2 Phase 2: Nitrification

good quality feed onto a nitrifying stage.

The raw data obtained during this phase of research are presented in Appendix 7 and are analysed in Section 6.2.

4.3 Full scale research

### 4.3.1 Existing filters at Cranfield sewage treatment works

The performance of the full scale sewage treatment works has been monitored by the Biotechnology Centre since 1986. The three trickling filters all contain Etapak 160, one of the media used for the pilot scale research, enabling some comparison of the results obtained at pilot and full scale.

Cranfield STW and its operation are described in Section 3.1. Raw data are tabulated in Appendix 8 and an analysis of the data is given in Section 7.1.

4.3.2 New nitrifying filter at Cranfield sewage treatment works

Towards the end of the period of the research, a new nitrifying filter was commissioned at Cranfield STW. The filter was designed with the assistance of the author, and using results obtained from the pilot scale research described here. The construction and operation of the filter are detailed in Section 3.3. Initial results are presented in Appendix 9 and discussed in Section 7.2.

# Chapter 5: Discussion of laboratory scale research

This chapter will interpret and discuss the results of the experiments which have been described in previous chapters. For brevity, this chapter will illustrate, in graphical or tabular form, only the results which are relevant to the discussion. Other results and data are presented in Appendices 4 and 5.

## 5.1.1 Nitrifier selection in a defined medium

The preliminary experiments involved a defined mineral medium to reduce the risk of interference from unknown substances in the subsequent kinetics experiments. However, this medium did not achieve a significant increase in the rate of nitrification during the 47-day course of the experiment. The maximum nitrification rate achieved was only 0.09 mg  $NH<sub>4</sub>-N.dim<sup>-3</sup> h<sup>-1</sup>$  (Appendix 4) and the slow rate of nitrifier population enhancement suggested that this method would not yield sufficient nitrifying organisms for kinetics experiments. Furthermore, the buffering capacity of the medium was insufficient to prevent rapid fluctuations in the pH (Figure 5.1). Later successful growth of nitrifiers on a slightly different defined medium (Section 5.1.2) suggests that the absence of molybdenum may have limited the growth of the organisms, although this was not proven.

#### 5.1.2 Nitrifier selection in a non-defined medium

A greater enrichment of the nitrifier population was achieved using undiluted Cranfield

sewage works final effluent as a non-defined medium. From an initial rate of 0.015 mg  $NH<sub>4</sub>-Ndm<sup>-3</sup> h<sup>-1</sup>$ , a ten-fold rate enhancement was obtained after 22 days (Appendix 4). However, this enhancement was not sufficient to enable accurate monitoring of kinetics experiments.

Difficulties were experienced in maintaining the pH between 7 and 8 due to the low buffer capacity of the final effluent. During the later stages of the experiment, the pH frequently dropped to below 6 overnight (Figure 5.1). The minimum pH recorded was 4.3, which is considerably below the minimum pH for nitrification of 4.9 recorded by Prakasam. & Loehr (1972).

#### 5.1.3 Nitrifier selection in activated sludge

4 ·<br>. . 18-Oct 24-Oct 01-Nov 14-Nov 17-Nov 22-Nov 30-Nov Date  $\cdot$  - Defined Medium  $+$  Non-Defined Medium Figure 5.1: pH changes during the enrichment of nitrifying organisms in defined and non-derined media

To achieve a larger population of nitrifiers, attempts were made to achieve sufficient enrichment of a sample of activated sludge taken from Milton Keynes STW, a nitrifying activated sludge plant. However, after an enrichment period of two months, a crude assay of the nitrifying activity revealed that the enrichment was insufficient to enable accurate comparisons of nitrification rates under varying conditions. The enrichment approach was consequently abandoned in favour of pure cultures of nitrifying organisms.

10 T



For one period during the enrichment process, neutralisation of the culture was suspended to determine the minimum pH at which nitrification could occur. Due to the higher buffering capacity of activated sludge, changes in pH were more gradual than those in the defined and non-defined media. The minimum pH measured was 3.6 pH units (Figure 5.2). This suggests that nitrifying organisms are capable of adapting to wide variations in pH.

Following the failure of the enrichment techniques, pure cultures of *Nitrosomonas* europaea and Nitrobacter agilis were obtained commercially and were grown in a defined medium (Appendix 3.1.2).

The medium included a pH indicator, which enabled the pH to be checked visually on



a regular basis. Sterilised sodium bicarbonate solution was added when the indicator began to turn yellow. The frequency of bicarbonate additions increased with time, demonstrating that growth was occurring. Sub-dilutions were made when the frequency reached approximately 2 bicarbonate additions per week.

Flasks were occasionally sacrificed to check the dissolved oxygen concentration by iodometric titration. In no instance did the measured DO fall below 2 mg.dm<sup>-3</sup>. Once

sampled, these flasks were discarded.

Due to the lack of organic carbon in the medium (apart from traces produced by the lysis of dead cells) contamination of the medium during culturing was not found to be a problem, although aseptic practices were used for serial culturing and pH neutralisation.

heat retention by submerged growth nitrification systems gives these processes a significant advantage over nitrifying trickling filters in areas susceptible to cold winters.

Temperatures between  $0^{\circ}$ C and  $50^{\circ}$ C were used in the experiments, although the higher temperature is outside the normal range experienced in sewage treatment works. The

#### 5.3 Use of pure cultures for kinetics experiments

Since nitrite, the intermediate product of nitrification, is generally not detected in significant quantities in sewage works effluent, it follows that the oxidation of ammonia to nitrite by Nitrosomonas europaea is usually the limiting step in nitrification. For this reason, efforts were concentrated on studying the effects of temperature, pH, BOD, allylthiourea and ammonia concentration on the rate of ammonia oxidation by N. europaea.

#### 5.3.1 The effects of temperature on ammonia oxidation

Temperature has been widely reported as a critical factor in nitrification. The greater

results indicate an approximately logarithmic increase in the rate of nitrite production up to a maximum at 37'C (Figure 5.3). Thereafter, the rate decreases rapidly until all activity is lost at  $50^{\circ}$ C.

Interestingly, when the flasks kept at  $50^{\circ}$ C were allowed to cool, full activity was regained, hence the bacterial cells had remained viable but inactive. Barritt reported that

the thermal death point of pure cultures of *Nitrosomonas* was in the range 54<sup>o</sup>C 58'C

The measured nitrite production rates can be used to determine a value for the constant,  $k<sub>r</sub>$  in the van't Hoff-Arrhenius equation (Equation 2.15) by plotting the natural logarithm of the reaction rate against temperature for the logarithmic section of the temperature relationship (Figure 5.4). The value for  $k_T$  thus derived is 0.093°C<sup>-1</sup>, which is in accordance with reported values for the growth of Nitrosomonas of  $0.095$ -0.12 $^{\circ}$ C<sup>-1</sup>

#### (Barritt, 1933).



#### (Downing & Knowles, 1966; Ekama & Marais, 1984; Knowles et al., 1965).



Figure 5.4: Ln[reaction rate] plotted against temperature to determine  $k_T$ , the constant in the van't Hoff-Arrhenius equation

## 5.3.2 The effects of pH on ammonia oxidation

There is some dispute in the literature regarding the optimum pH for the oxidation of ammonia by Nitrosomonas, and the response of the bacteria to pH values outside the optimum range. Early research involved the use of pure cultures and should, theoretically, have provided an indication of the response of the nitrifying bacteria in a controlled environment without interference from other factors (Barritt, 1933; Engel & Alexander, 1958; Meyerhof, 1916). However, the wide variations in the results obtained cast serious doubts on the validity of the early data. The reasons for the discrepancies are not clear, although differing experimental temperatures may be a cause. However, gil differing experimental tempe insufficient information was published to enable a critical analysis of the experimental techniques used.

Later research involved the use of mixed bacterial cultures and non-defined media (Antoniou et al., 1990; Prakasam & Loehr, 1972; Wild et al., 1971). It is therefore conceivable that the effects observed are due to some more complex phenomena rather than a simple bacterial response.

Pure cultures were used for this research in order to minimise interference from external factors. Experiments were performed with pH between 5.7 and 8.0. The lower limit is

The results indicate that the optimal pH for nitritification at  $32^{\circ}$ C lies in the range 7.8-7.9 pH units (Figure 5.5). The effect of pH on the rate of ammonia oxidation can be closely approximated in the range pH 5.8-7.8 by the following linear equation:

below the pH values commonly experienced in the bulk liquid phase, but is representative of nitrification in a low-alkalinity wastewater or in a thick biofilm, where mass flux limitations may reduce the pH below that of the bulk liquid.



$$
\mu = \mu_{Max} (0.50pH - 2.90) \tag{5.1}
$$

The results obtained are re-plotted in Figure 5.6 for comparison with earlier literature data. The results are consistent with those obtained by other workers using nitrifying activated sludge (Antoniou et al., 1990; Wild et al., 1971).

The data to support the derivation of Equation 5.2 are not published. The differences between Downing's equation and the data and equation derived from this work are



The equation commonly used to calculate the effects of pH on nitrification was derived empirically by Downing and Knowles (1966) and subsequently adopted by the U.S. Environmental Protection Agency (1975):

$$
\mu = \mu_{Max} (1 - 0.83(7.2 - pH))
$$
 (5.2)

illustrated in Figure 5.7. It is apparent from the graph that Downing's equation exaggerates the effects of pH, particularly since none of these experiments allowed any acclimatisation period to the pH. If a culture was allowed to acclimatise to the pH, as would normally be the case at full scale, the effects of the pH may appear less than predicted by either of the equations.



The effects of the ammonia concentration on *Nitrosomonas* have been previously studied by other workers (Anthonisen et al., 1976; Boon & Laudelout, 1962; Painter, 1970;

0%  $5.6$   $5.8$   $6$   $6.2$   $6.4$   $6.6$   $6.8$   $7$   $7.2$   $7.4$   $7.6$   $7.8$  8 pH

• Measured Data  $\cdot$  -Equation 5.1  $\div$  Data from Wild et al. - Downing's Equation

Prakasam & Loehr, 1972; Wiesman, 1994; Wong-Chong & Loehr, 1975; Yang & Alleman, 1992). There is consensus that ammonia oxidation by Nitrosomonas follows Michaelis-Menten kinetics. Some workers have reported zero order (Daigger & Nicholson, 1990; Liu & Capdeville, 1994; Wong-Chong & Loehr, 1975) or first order

Figure 5.7: A comparison of measured data and derived equations for the effect of pH on nitritification

#### 5.3.3 The effects of substrate concentration on ammonia oxidation

kinetics [Srna, 1975] but a closer examination of their data reveals that an insufficient range of substrate concentrations and a poor choice of experimental conditions has restricted their data points to the upper or lower portions of the Monod curve, where the kinetics tend towards zero and first order respectively.

Ammonia concentrations up to 1000 mg  $NH_4-N$ . dm<sup>-3</sup> were used. Each assay was carried out at 32'C and pH 7.2. The results indicate an enhanced rate of nitritification at higher ammonia concentrations (Figure 5.8).

Several workers have reported inhibition of nitritification at high concentrations of free  $\overline{\phantom{a}}$ ammonia (NH<sub>3</sub>) (Anthonisen *et al.*, 1976; Prakasam & Loehr, 1972; Wiesman, 1994). However, at pH 7.2 and 32<sup>o</sup>C, the free ammoniacal nitrogen concentration at an

This series of experiments was intended as a check on the validity of the experimental protocol as well as a verification of the literature results.

 $0\%$  $\frac{1}{100}$ ,  $\frac{1}{200}$ ,  $\frac{1}{200}$ ,  $\frac{1}{400}$ ,  $\frac{1}{500}$ ,  $\frac{1}{600}$ ,  $\frac{1}{700}$ ,  $\frac{1}{900}$ ,  $\frac{1}{100}$ 0 100 200 300 400 500 600 700 800 900 1,000 Ammonia Concentration (mgNH,-N.dm<sup>-3</sup>) Figure 5.8: The effect of substrate concentration on ammonia oxidation by Nitrosomonas europaea



ammonium-N concentration of 1000 mg.dm<sup>-3</sup> is only 11 mg.dm<sup>-3</sup> (Appendix 1). Anthonisen *et al* indicated that inhibition increases over the range 10-150 mg.dm<sup>-3</sup> (as free ammoniacal nitrogen) (Figure 2.4). Wiesman obtained a value for  $K_{IH}$ , the halfinhibition constant, of 540 mg  $NH<sub>3</sub>-N.dim<sup>-3</sup>$  (Wiesman, 1994) hence no significant inhibition was expected in these experiments.

The rate of ammonia oxidation is shown to follow Michaelis-Menten kinetics by replotting the results on a double-reciprocal plot (Figure 5.9) (Lineweaver & Burk, 1934). The value thus obtained for the half-saturation constant,  $K_{SN}$ , is 38.6 mg.dm<sup>-3</sup>. This value is considerably higher than other reported values obtained using pure cultures at lower temperatures (Table 5.1).



Figure 5.9: Double-reciprocal plot showing the effect of substrate concentration on ammonia oxidation

# Table 5.1: A comparison of values for the half-saturation constant,  $K_{SN}$ , obtained from the literature.



It is clear from the table that there is considerable variation in the values of  $K_{SN}$ reported, with no clear correlation with either the type of culture or the temperature. A

possible reason for the high measured value, and perhaps for the wide range of reported values, is that the pure culture used in this research was maintained with a non-limiting supply of ammonia (212 mg  $NH_4-N. dm^{-3}$  initially) hence the efficiency of the mechanism for the uptake of ammonia may have been reduced. Since the cultures were

not permitted time to acclimatise to the experimental ammonia concentrations, the halfsaturation constant was measured as a high value. This hypothesis was not tested experimentally and insufficient detail is published to verify it in the literature.

### 5.3.4 The effects of B.O.D. concentration on ammonia oxidation

This experiment was designed to determine whether nitritification could be inhibited by the presence of two readily-degradable short-chain organic compounds: D-glucose and

#### L-glutamic acid. The results are illustrated in Figure 5.10.

In a mixed culture, it is known that autotrophic nitrifying organisms cannot compete effectively with faster-growing heterotrophic m icro- organisms. Consequently, nitrification usually occurs subsequent to carbonaceous oxidation, since the concentration of organic carbon sources is low enough to limit heterotroph growth. This phenomenon would normally mask any inhibitory effect of readily-degradable carbon sources on nitrification which may occur due to, for example, a switch to a more energy-efficient metabolic pathway or the competitive inhibition of ammonia uptake.

The apparent effect is not due to deamination of the glutamic acid during medium sterilisation, since amine nitrogen comprises less than 10% by weight of glutamic acid and the highest glutamic acid concentration used was 136 mg.dm<sup>-3</sup>. Hence even total deamination of the medium would increase the initial ammoniacal nitrogen concentration from  $200 \text{ mg.cm}^{-3}$  to only  $213 \text{ mg.cm}^{-3}$ . g.dm: Figure 5.8 has demonstrated that this would

Figure 5.10 shows a gradual increase in the rate of nitritification over the range of glucose/glutamic acid concentrations used in the experiment. It is unlikely that this effect could be due to contamination of the culture, since the experiment was carried out over a period of only 5.5 hours and any competition from heterotrophs growing on the glucose/glutamic acid mixture would tend to inhibit the rate of nitritification. Since the experiment involves the determination of nitrite production, rather than ammonia removal, the effect cannot be due to uptake of ammonia by contaminant organisms.

have a small effect on the rate of nitritification.

This result implies that Nitrosomonas europaea may not be an obligate chemolithotroph as suggested by Winogradsky, but a facultative chemolithotroph, which can utilise carbon dioxide as a carbon source but can also metabolise organic carbon compounds (Winogradsky  $\alpha$  Winogra  $\overline{\phantom{a}}$  $\ddot{\phantom{a}}$ radsky, 1933). In this case, either glucose or glutamic acid was

metabolised more efficiently than carbon dioxide, hence the rate of nitritification increased.

Radioactive tracer experiments using radio-labelled carbon could be used to determine whether Nitrosomonas can indeed utilise organic carbon sources, or whether the apparent effect was due to some form of interference or contamination. However, this aspect of research was not pursued further.



# 5.3.5 The effects of allylthiourea concentration on ammonia oxidation

Allylthiourea (ATU or thiosinamine) is a highly toxic compound which has been widely used as a specific inhibitor of nitrification in the BOD test. The standard concentration of ATU used in the BOD test is 500  $\mu$ g. dm<sup>-3</sup> (Montgomery & Borne, 1966; Wood et al., 1981) although some workers have recommended the use of up to 5 mg.dm $^{-3}$  (Abendt & Fischermeier, 1983; Raff, 1985). ATU has been shown to have an insignificant effect

on the rate of carbonaceous oxidation by heterotrophs at concentrations up to 5 mg.dm<sup>-3</sup> (Abendt & Fischermeier, 1983; Raff, 1985; Tyers & Shaw, 1989).

A range of allylthiourea concentrations between  $0.01$  and  $100 \mu M$  (1.16  $\mu$ g.dm<sup>-3</sup>) 11.6

#### mg.  $dm^{-3}$ ) was used in the experiments. The results are plotted in Figure 5.11.

The inhibitory effects of ATU are believed to be due to the complexing of copper ions, which are essential enzyme co-factors in the metabolism of Nitrosomonas. Homologues of ATU such as thiourea and methylthiourea (MTU) exert a more powerful inhibitory effect but are less practical to use.

The half-saturation constant,  $K_{I}$ , for inhibition of nitritification by ATU can be calculated from a double-reciprocal plot (Figure 5.12) (Lineweaver  $\&$  Burk, 1934). Only the first six data points have been included since, at higher ATU concentrations, the reaction was inhibited by greater than 90%, hence nitrite concentrations were low and

Figure 5.11 shows a characteristic inhibition curve with 95% inhibition of nitritification occurring at an ATU concentration of only  $100 \mu g \cdot dm^{-3}$ . At the concentration used in the BOD test, the graph indicates that inhibition is 99% complete.

#### measurement was less accurate.

The value of  $K_{I}$  obtained from Figure 5.12 is 7.0  $\mu$ g. dm<sup>-3</sup> (the negative reciprocal of the intercept on the horizontal axis). This value confirms that, by extrapolating these results, inhibition of nitritification in the BOD test using  $500 \mu g \cdot dm^{-3}$  ATU is 99% complete.





# Chapter 6: Discussion of pilot scale research

## 6.1 Pilot scale research, phase 1

# 6.1.1 Operation of the pilot scale filters for carbonaceous oxidation

During the first phase of research, the pilot scale trickling filters were dosed with effluent from primary treatment (screened and settled) in order to determine the effectiveness of the four media for secondary treatment and to accelerate the maturation of the filters (Section 4.2.1). This phase of research covered the period September 1990 to February 1991.

During this period of study, 23 samples of the feed to the pilot scale filters were taken. Samples of wastewater at this stage of the treatment process have been analysed since 1986, so the characteristics of the wastewater during the study may be compared with historical data to ensure validity. The data are summarised in Table 6.1 and Figures 6.1

Table 6.1: Statistical analysis of the settled crude wastewater at Cranfield STW.








As Table 6.1 indicates, the average wastewater strength during this phase of research was generally lower than the historical average, with the exception of the ammonia strength. This may be attributed to increased dilution of the incoming flow during the late autumn and winter.

The period of study was also characterised by a more consistent strength of wastewater, as indicated by the lower standard deviations. The greater consistency can be attributed to the infrequency of accidental spillages within the sewer catchment during the period of study, which have occasionally resulted in a considerable short-term increase in wastewater strength.

The hydraulic load to the pilot scale filters was set at  $1.4 \text{ m}^3.\text{m}^{-3}.\text{d}^{-1}$ , approximately double the low volumetric loading of the full scale filters. This loading rate was achieved using solenoid valves to provide periodic dosing.

## 6.1.2 Start-up and assessment of BOD removal in the pilot scale filters

Table 6.2 summarises the removal rates achieved by the four media during this phase of research. The results are not illustrated graphically because the number of samples was not sufficient to obtain a statistically valid assessment of the media. The figures quoted in the table are intended for comparison between media, and not as accurate nutrient removal rates.

The greater BOD removal in the blast furnace slag medium enabled the establishment of a nitrifying population towards the base of the filter, where the BOD concentration was typically less than 20 mg.dm<sup>-3</sup>. Consequently, nitrification rates achieved by the

As predicted, the blast furnace slag medium in pilot scale Filter 2 achieved higher rates of BOD and suspended solids removal than the plastic media. This is attributed to a more rapid start-up due to the coarser surface of the media and a high degree of physical filtration as effluent passed through the fine pores in the slag medium.

a neutron probe. The results, expressed as the percentage by volume occupied by water (moisture volume fraction or  $\%$ MVF) are illustrated in Figure 6.4.

blast furnace slag filter were also considerably greater than the rates achieved by the plastic media filters.

The physical filtration through the blast furnace slag medium, coupled with a higher rate of biomass colonisation of the media, led to a rapid accumulation of solids within the medium. It was observed that, on the upper layers of media, the fine pores had become blocked. If this experimental phase had continued, the interstices between individual pieces of media would have blocked also and the filter would have begun to pond *(i.e.* the applied effluent would have been unable to pass through the filter).

The accumulation of biomass and biological solids was measured during Phase I using

## Table 6.2: Summary of the performance of the pilot scale filters during phase 1.



Figure 6.4 clearly illustrates the rapid accumulation of biomass to almost 12% by volume within the blast furnace slag medium and provides a vivid comparison with the plastic media, where the accumulation of biomass is less than 1.2% in all cases. As will be discussed later, this technique of dosing the pilot scale filters with stronger primary effluent was a successful means of obtaining a rapid start-up and stabilisation of the biological population.

6.2 Phase 2: Operation of the pilot scale filters for nitrification

From February 1991 until the end of the study, the pilot scale filters were operated as tertiary nitrifying filters (Section 4.2.2). The filters were routinely sampled to assess their performance as nitrifying filters (Sections 6.2.1 to 6.2.4) and the temperatures of



the filters were monitored continuously (Section 6.2.5). In addition, intermittent analyses were undertaken to determine the retention time characteristics of the media (Section  $(6.2.6)$  and the extent of biomass growth within the filters  $(6.2.7)$ .

Apart from a brief period at the beginning of Phase 2 of the research, the pilot scale filters were dosed from a common feed at a volumetric loading rate of approximately  $6 \text{ m}^3$ .m<sup>-3</sup>.d<sup>-1</sup>. The characteristics of the feed are summarised in Table 6.3.

The average BOD and ammoniacal nitrogen concentrations in Table 6.3 are characteristic of the effluent from an under-performing non-nitrifying sewage treatment

works. The average suspended solids concentration is high, reflecting the fact that the pilot scale filters were dosed with unsettled filter effluent. Another important characteristic to be derived from the table is the extreme variation in the strength of the wastewater, illustrated by the high standard deviations. The variation is distinctive of a sewage treatment works where the filters are over-loaded, hence pollutants are

occasionally breaking- through and imposing a high instantaneous loading on downstream processes.

Table 6.3: Characteristics of the influent to the pilot scale filters during phase 2 of the research.





\* These data were obtained during short-term pollution incidents within the catchment.

In all of these respects, a tertiary nitrifying filter provides an appropriate means to achieve consent compliance by reducing the average BOD and ammonia concentrations, improving the settlement characteristics of the solids and diminishing the short-term peaks in the loading.

### 6.2.1 BOD removal in the nitrifying pilot scale filters

Due to the day-to-day variation in the influent strength to the pilot scale filters, it is not appropriate to plot graphs showing the variation in BOD strengths over time. Furthermore, the design of sewage treatment works is generally based on the requirements to meet 95%ile limits for BOD, ammonia and suspended solids. Consequently, the performances of the pilot scale filters are illustrated by means of

cumulative frequency graphs. These graphs enable the average performance to be gauged from the shift of the curve to the left, as well as portraying the reliability of the filters by the shape of the curve (the higher the maximum gradient of the curve, the more reliable the process).

For ease of comparison, the cumulative frequency distribution curves for the influent to

the filters and the effluent from each of the four filters are shown on Figures 6.5 and

6.6. The key statistical data are surnmaxised in Table 6.4.

#### Table 6.4: Summary of BOD removal rates.



Figures 6.5 and 6.6 clearly demonstrate a higher rate of BOD removal in the blast furnace slag filter than in the plastic media filters. The average BOD removal rate of 82 g BOD. $m^{-3}$ . $d^{-1}$  compares favourably with rates of 54-62 g BOD. $m^{-3}$ . $d^{-1}$  for the other filters (Table 6.4). Of the plastic media filters, Flocor RS appears to exhibit marginally better performance than either of the Etapak media, but the difference does not seem to be statistically significant. The better performance of the blast furnace slag medium is due to the greater active biomass population. It is postulated that the corrugations in the



Figure 6.5: Cumulative frequency graph of BOD concentrations in the influent and effluent from the pilot scale filters (full range).



and effluent from the pilot scale filters (low range data only).

Flocor RS medium reduce the shear stresses arising from liquid flow and hence permit a higher biomass accumulation than the other plastic media filters.

To determine the effect of media loading on the rate of BOD removal, these data are illustrated in Figures 6.7 to 6.10. Values derived from the figures are presented in Table

Despite the high degree of scatter, which is inevitable in a pilot scale study subjected to loading and climatic changes, Figures 6.7 to 6.10 clearly demonstrate a linear relationship between the rate of BOD removal and the filter loading. The removal of BOD in nitrifying trickling filters is thus confirmed as a pseudo first-order process.









#### Table 6.5: Summary of BOD removal data derived from Figures 6.7 to 6.10.



For secondary trickling filters (for carbonaceous oxidation), the effluent BOD load has

been related to the applied load, the retention time and a rate constant (Schultze, 1960):

$$
S_e = S_0 e^{-kt}
$$
 (6.1)

where  $S_e$  and  $S_0$  represent the effluent and influent BOD concentrations respectively, k is the reaction rate constant, and t is the retention time. This equation may be rearranged to express the percentage BOD removal as a function of the rate constant:

$$
\frac{\mathcal{S}_0 - \mathcal{S}_e}{\mathcal{S}_0} = \mathcal{K} \tag{6.2}
$$

where K is equal to 1- $e^{-kt}$ , the removal constant for the medium.

By plotting the applied load against the BOD removal achieved, the removal constant for each of the media may be determined from the gradients of the best-fit lines. The removal constant is a dimensionless number which represents a true measure of the BOD removal performance of the media when operating as nitrifying media at a

volumetric loading of 6  $m^3.m^{-3}.d^{-1}$  (a typical loading for nitrifying trickling filters).

It is interesting to note that, in each case, the best-fit line intercepts the X-axis at a point away from the origin. This contrasts with the simplified Schultze equation where a plot of BOD load against removal gives an intercept at the origin *(i.e.* steady-state conditions are assumed). The intercepts on the X-axes in Figures 6.7 to 6.10 indicate the extrapolated minimum effluent BOD loads under non-steady state conditions. The minimum effluent BOD comprises solids washed out of the filter.

Samples were allowed to settle for 30 minutes prior to analysis, hence the minimum

effluent BOD determined by these experiments may be greater than that achieved in full scale systems, where a longer settlement period is permitted. It is postulated that the lower minimum effluent BOD achieved by the blast furnace slag medium is due to a combination of better BOD removal within the filter and improved humus settlement characteristics.

The blast furnace slag medium provides an enhanced rate of BOD removal and enhances the settlement of the humus solids hence achieving a lower minimum effluent BOD.

The plastic media all accomplished similar rates of BOD removal, significantly lower than the removal rate of the blast furnace slag medium. It is interesting to note that the minimum effluent BOD concentration of the high specific surface area media (Etapak

210 and Flocor RS) are comparable and markedly lower than that of the Etapak 160

medium. This may be due to the Etapak 160 medium supporting a smaller but more metabolically active population of micro-organisms than the other plastic media, hence the BOD of humus solids in the effluent is correspondingly higher.

While the above data correlate the actual in situ BOD removal rates achieved by the four media, further conclusions may be drawn by normalising the data with respect to the temperature. The temperature was monitored continuously at the centre of each filter, enabling the removal rates to be corrected to 20°C by dividing measured rates by a factor  $\Theta$ , where  $\Theta = 1.035^{\text{(T-20)}}$  (Schultze, 1960). Figures 6.11 to 6.14 illustrate the

Table 6.6 may be used to predict the BOD removal performance of the four media when operating as nitrification media at a hydraulic loading of 6  $m^3.m^{-3}.d^{-1}$  under non-steady state BOD loading conditions and at temperature  $T$  (°C):

correlation between the applied BOD loading and the normalised BOD removal rate. The key features of Figures 6.11 to 6.14 are summarised in Table 6.6.

$$
S_e = 1.035^{(T-20)} S_0 K_{20} - S_{min20}(K_{20} - 1)
$$
 (6.3)



Figure 6.11: Correlation between applied BOD load and the temperaturecorrected BOD removal rate for Etapak 210.



corrected BOD removal rate for blast furnace slag.



Figure 6.13: Correlation between applied BOD load and the temperaturecorrected BOD removal rate for Etapak 160.



Figure 6.14: Correlation between applied BOD load and the temperaturecorrected BOD removal rate for Flocor RS.

# Table 6.6: Summary of normalised BOD removal data derived from Figures 6.11 to 6.14.



Figure 6.15 indicates that nitrification in the plastic media filters was significantly inferior to the performance of the blast furnace slag medium. Key statistics from the illustrated data are tabulated in Table 6.7.

As for BOD removal, comparative nitrification rates of the pilot scale filters cannot be interpreted from plots of performance aggainst time due to the daily variation inherent in experiments in uncontrollable conditions. Performance is therefore illustrated by means of a cumulative frequency graph (Figure 6.15).

Table 6.7 illustrates the superior nitrification achieved by the blast fumace slag filter. Nitrification is not complete even in this case due to the shallow depth of the filters and the variable high loading rates. In contrast, the plastic media filters achieved a consistent  $\overline{1}$ yet low level of nitrification.



### Table 6.7: Summary of nitrification rates in the pilot scale filters.



To determine the effects of loading on the rate of nitrification, the data are re-plotted in Figures 6.16 to 6.19. Best-fit curves have been illustrated on the graphs but the low rate of nitrification achieved by the plastic media filters, coupled with the scatter due to varying conditions, cause such high errors in the best-fit lines that they cannot be considered as statistically valid in these cases.

where N represents the rate of nitrification  $(g.m^{-3}.d^{-1})$ ,  $N_{max}$  is the maximum rate of nitrification under the experimental conditions of hydraulic load and media depth, L is the loading rate  $(g.m^{-3}.d^{-1})$  and  $K_{SN}$  is a form of the half-saturation constant which is indicative of the interface between first and zero order kinetics.

Data are plotted as the applied load against the rate of removal, since this plot illustrates that the rate of removal tends towards a maximum value as the load increases. The data of other workers, who have stated that nitrification is a zero-order reaction, may be replotted in this way to demonstrate that the kinetics actually follow a Monod-type equation (Equation 6.4) (Liu & Capdeville, 1994). At low nitrification rates (high residual ammonia concentration) the kinetics approach zero order whilst improved nitrification (low residual ammonia concentration) gives kinetics approaching first order.

Due to the low correlation of the best-fit lines for Figures 6.16, 6.18 and 6.19, information derived from Figure 6.17 only is summarised in Table 6.8. The values tabulated are valid at the hydraulic loading rate used for this research,  $6.36 \text{ m}^3.\text{m}^{-3}.\text{d}^{-1}$ , as other loading rates will affect the retention time.

$$
N = \frac{N_{\text{max}} L}{K_{\text{SN}} + L}
$$
 (6.4)

Using the figures derived below, a set of curves may be generated to predict the performance of blast furnace slag, nitrifying trickling filters (Figure 6.20). By normalising the volumetric ammonia loading rates into filter surface loading rates, the curves may be extrapolated to cover a range of filter depths. 4-







nitrification in the Etapak 160 filter.



60 80 100 120 140 160 180 200 220 240 20 40  $\overline{0}$ Ammonia Loading Rate (g NH<sub>4</sub>-N.m<sup>-3</sup>.d<sup>-1</sup>) Figure 6.19: The effect of ammoniacal nitrogen loading on the rate of nitrification in the Flocor RS filter.

### Table 6.8: Summary of nitrification data derived from Figure 6.17.





While the curves on Figure 6.20 are only strictly applicable to filters with a surface hydraulic loading rate of 7.6  $m^3.m^{-2}.d^{-1}$  (as for the pilot scale filters), they may also be used to estimate the performance of nitrifying trickling filters at other hydraulic loading rates by using the relationship determined by retention time studies in Section 6.2.6 (Equation 6.6).

No correlation can be detected between the rate of nitrification and the temperature in

III

any of the pilot scale filters. Even when the nitrification rates are corrected for the load (using the curve on Figure 6.17) to compensate for lower ammonia concentrations in the influent during warm weather, no correlation is evident. Nevertheless, temperature has been shown to be an important factor in nitrification at laboratory scale and in other pilot and full scale studies (Gujer & Boller, 1986; Jansen et al., 1992; Sen et al., 1992; Shammas, 1986; Tendaj et al., 1992).

To determine the extent of denitrification in the pilot scale filters, samples of the influent and effluent were routinely analysed for oxidised nitrogen concentration as well as ammoniacal nitrogen. Oxidised nitrogen includes both nitrite and nitrate, but since nitrite was never detected at concentrations greater than 0.1 mg. dm  $\degree$  the analysis can be

The reasons for a lack of correlation between nitrification and the temperature in this research are not clear. However, the thermistors were located at the centres of each filter and the filters, standing above ground and having relatively small radii, were subject to greater cooling and heating effects than would be found at full scale. It is postulated that the temperature of the peripheral volume of each filter may have varied sufficiently from the temperature recorded at the centres to mask the effects on the nitrification rates.

## 6.2.3 Denitrification in the pilot scale filters

Trickling filters are designed as aerobic biological processes. However, if the ventilation through the filter is inadequate or if the biofilm depth is such that oxygen does not diffuse to the inner layers of biomass, anoxic conditions may exist. Under anoxic conditions and in the presence of nitrate and an organic carbon source, denitrification may occur.

considered as the determination of nitrate. If it is assumed that all urea present in the

crude wastewater has been converted to ammonia, that the rate of ammonia generation

by anaerobic deamination of protein is negligible, and that nitrogen loss by heterotrophic

uptake or ammonia stripping is negligible then the measured removal of nitrogen (nitrate

plus ammonia) is due to denitrification.

There were no distinct trends in the summated ammoniacal and oxidised nitrogen concentrations with either time or filter loading, hence the results are presented in Table 6.9 as the mean loadings during the period of study.

Table 6.9: A comparison of mean nitrogen removal rates in the pilot scale filters





The pilot scale filters were designed to have adequate ventilation. Common design practice for nitrifying trickling filters is to allow a ventilation area *(i.e.* holes in the base of the filter) equivalent to 2-5% of the plan area of the filter. The large pilot filter (containing Etapak 210) had a ventilation area of 7.6% while the other filters each had

The results tabulated above indicate a low level of denitrification in each of the pilot scale filters. Despite the high standard deviation, a t-distribution test indicates a certainty of greater than 95% that nitrogen loss is occurring in each filter. The certainty is more than 99.9% for the blast furnace slag and Flocor RS filters. The blast furnace slag filter recorded a higher rate of nitrogen removal than the plastic media filters.

an area of 6.2%. The dissolved oxygen concentration in the influent and effluent from the filters was checked periodically during the period of research, but at no time did it drop below 4.2 mg.dm $^{-3}$ . It is therefore believed that oxygen transfer into the liquid phase of the filter was not limiting.

nitrogen removal and the quantity of biomass within each filter. The conclusion is that oxygen limitation in the pilot filters was due either to slow mass transfer between the aqueous phase and the biofilm or to diffusion limitations within the biofilm itself; probably the latter. This confirms the earlier findings of other workers (Chen *et al.*, 1989; Harremoës, 1982; Revsbech *et al.*, 1989; Siegrist & Gujer, 1987).

#### 6.2.4 Solids removal in the pilot scale nitrifying filters

As will be discussed in later chapters, the accumulation of biomass varied markedly between the different media. There appears to be a correlation between the extent of

In addition to reducing the BOD and ammonia concentrations in the final effluent,

tertiary trickling filters provide a useful role in improving the settleability of suspended solids and buffering short-term peaks in the concentration of solids imposed by upstream processes. The increased settleability results from the conversion of finely-divided solids and colloidal matter into biomass, which is eventually sloughed as larger, denser particles. Since the fine solids and colloids also contribute to the BOD, it is anticipated that efficient solids removal is related to BOD removal.

As described in Chapter 3, the wastewater dosed to the pilot scale filters was unsettled effluent from the plastic media secondary filters at Cranfield STW. A tertiary nitrifying filter would normally be dosed with a settled wastewater, hence the suspended solids

loading applied to the pilot scale filters was unusually high.

The solids comprise not only finely divided solids and colloids, but also some humus solids shed by the secondary filters. Since, at equilibrium, the humic content of a filter is approximately constant, the net influx of humus solids is equal to the loss in the filter

effluent (although the passage of humus solids through a filter tends to improve its flocculation). The anticipated differences in the solids removals achieved by the different media will therefore be mainly due to improved capture of fine solids and colloids.

In addition to suspended solids, the pilot filter effluents were analysed for total solids (TS) to determine the total quantity of solids shed and the settleability of the solids. Samples for total solids analysis were agitated immediately prior to filtering, whereas suspended solids samples were settled for 30 minutes. The difference therefore represents the concentration of solids which are settleable in 30 minutes.

It should be noted that, in a full scale treatment works, tertiary filter effluent would normally be settled for at least two hours prior to discharge. Furthermore, the increased depth of a full scale filter would improve the flocculation of the solids. The suspended solids concentrations recorded at pilot scale are therefore considerably higher than would be achieved at full scale.

The sharp peaks in the solids load to the filters in late April to early June 1991 were due to two spillages within the catchment area of Cranfield STW. The first, a spillage of heating oil, formed a thick layer on the surface of the primary settlement tank and flowed under the scum boards, coating the media in the secondary filters with a film of

There were no discernable seasonal variations in the ratios of suspended solids to total solids in any of the pilot filters. Consequently, results are expressed as suspended solids only.

The suspended solids loads to the pilot filters, and the effluent loads, were generally more consistent over time than other measured determinands. It is interesting to note the response of the pilot filters to shock loads and to examine the phenomenon of Spring sloughing, hence Figure 6.21 is illustrated as an example. Graphs of the seasonal performance of the other filters are not included since this type of plot does not adequately demonstrate the differences in performance between the media.



oil. This caused a massive detachment of biomass from the secondary filters, but did not

have such a severe effect on the pilot scale filters. The oil persisted for several weeks as sticky deposits were flushed from the sewerage system. The second spillage, of aviation fuel, was less damaging, causing only a short-term loss of biomass.

The results obtained during the periods of abnormally high solids loads illustrate the effectiveness of tertiary nitrifying filters at reducing the magnitude of shock loads. The effluent solids load appears to be reasonably constant, particularly for the blast furnace slag filter, and is independent of the applied load.

Unfortunately, the spillages obscure the Spring sloughing of biomass for 1991. However,

the sloughing may be distinguished during March and April 1992. There was an earlier period of sloughing during January 1992 which was probably caused by the period of unusually high temperatures during mid-January.

The performance of the individual filters is best illustrated by plots of the applied volumetric solids load against the effluent solids load (Figures 6.22 to 6.25).

In each of Figures 6.22 to 6.25, two distinct phases can be seen (indicated as proportional and non-proportional data points). At low loadings, there is a clear proportionality between the effluent solids load and the applied load. In this region of

the graph, the filter is in equilibrium; the growth of biomass plus the solids removed by

filtration are similar to the sum of the solids lost in the effluent and the solids degraded into soluble or gaseous matter, hence the net solids content of the filter is approximately constant.

At higher loadings (due to oil spillages) the effluent solids is independent of the applied load. This can be explained by the fact that the solids shed by the secondary filters during oil spillages were large flocs, several millimetres in diameter, which were removed at high efficiency during passage through the pilot filters. These values have









Figure 6.25: The efficiency of solids removal in the Flocor RS pilot filter.

#### been discarded for the calculation of the best-fit lines.

The best-fit line should be regarded as an approximation in each case since the data do not correlate well. However, there are two clear trends common to each graph: the bestfit lines intercept the Y-axis above the origin and the gradient of each line is approximately 0.6 (gradients range from 0.58 to 0.68). The positive intercepts signify that, when extrapolated to zero solids load, the filters would shed a small amount of solids to compensate for the growth in biomass.

The fact that the best-fit line is linear is notable because, if the small positive intercept

on the Y-axis is neglected, the percentage solids removal achieved by the filter medium

may be assumed to remain constant at any solids loading.

Key statistics from the data illustrated in Figures 6.22 to 6.25 are tabulated in Table 6.10.

## Table 6.10: Summary of suspended solids removal data.



The tabulated data indicate the relatively poor suspended solids removal performance of the Etapak 160 medium. This is believed to be due to the lack of liquid storage capacity within the medium. The other plastic media (Etapak 210 and Flocor RS) are shaped such that water is retained, to an extent, within the media. Similarly, blast furnace slag is porous, hence water is held within the medium itself. The liquid retention

allows time for the flocculation process to occur, thereby improving the settleability of

the solids and achieving a degree of solids degradation.

It should be noted, however, that the different design of the Etapak 210 pilot filter may

have influenced the results. The filter had a concrete base, screeded towards the outlet pipe at a shallow angle. The angle was insufficient to prevent deposition of solids, which gradually accumulated into a thin sludge layer unless the filter was cleaned out manually. This layer enabled better flocculation of solids as the effluent percolated through the sludge. In contrast, the bases of the other pilot filters were narrower and more steeply sloped, so solids deposition was negligible.

The total solids results tabulated above correspond well to the BOD and ammonia removal performances of the four media, since removal of these nutrients will result in the generation of solids as biomass. The blast furnace slag filter, which achieved the highest BOD and ammonia removal rates, produced the most solids. The plastic media filters, which each achieved similar BOD removal and low rates of ammonia removal, produced comparable quantities of solids. The exception to this is the Etapak filter, where the filter design may have caused the apparently low solids generation rate.

It is interesting to note that the solids yield (the solids generated divided by the BOD and ammonia removed) is apparently very high. Solids yields for trickling filters are generally within the range 0.6-0.9 g SS per g BOD and 0.25 to 0.35 g SS per g of ammoniacal nitrogen. Comparison of the blast furnace slag, Etapak 160 and Flocor RS filters gives a yield on BOD of 2.5-3.0  $g.g^{-1}$  and a low yield on ammoniacal nitrogen.

The likely cause for this apparently high yield is the unusually high solids load to the pilot filters. A proportion of these solids will represent a source of nutrients, yet these nutrient were not detected by the BOD test as samples were settled prior to analysis.

Other possible influences in the apparently high solids yields were the oil spillages during the early period of the research. It is believed that the pilot filters may have accumulated a larger quantity of solids than were detected by irregular sampling. Furthermore, the method used to accelerate start-up of the filters, by dosing with settled crude wastewater, may have allowed the filters to accumulate more biomass than could be maintained when operated as tertiary filters.

The longer hydraulic retention time of the blast furnace slag filter enables improved removal of colloidal matter and a greater degree of flocculation of fine particles. This is demonstrated as a higher fraction of the total solids in the filter effluent being settleable in a 30 minute period. This characteristic is of obvious benefit in tertiary nitrifying filters, since a well- flocculated filter effluent will settle effectively and produce a low suspended solids concentration in the final effluent.

Temperature is well known to be a major influence on the rates of BOD removal and nitrification. It is therefore important that a filter medium is able to minimise the heat loss by convective cooling, while still maintaining adequate air flow for aeration. All four media used in the research provided sufficient aeration to maintain the dissolved oxygen concentration in the filter effluent at above  $4 \text{ mg.dim}^3$ .

Attempts were made to measure the rate of air convection through the pilot scale filters as described in Section 3.7.6. However it was found that, even on an apparently still day, convection currents were masked by eddies of wind around the filters. Although it was obvious that there was a net movement of air into the bases of each filter, the fluctuations and occasional reversal of the rate of flow precluded accurate measurement.

 $\overline{a}$ Figures 6.26 to 6.28. Data are plotted as filter temperatures relative to the blast furnace

Three of the pilot filters were identical and received similar thermal inputs from solar radiation and warm influent, and were subject to similar wind conditions. Most of the

temperature differences between these filters were therefore due to convective cooling by air rising through the filters.

Temperatures at the centre and below the base of each filter were logged continuously.

The daily averaged temperatures at the centres of the filters for a complete year (with

the exception of data lost due to damage to the thermistor cables) are illustrated in

slag medium as this type of plot best illustrates the differences between the filters. It

should be noted that the Etapak 210 filter is larger, of different design, and is not sheltered from the wind to the same extent as the smaller filters. Any comparisons with the Etapak 210 medium should be made with due regard for this fact.

Each of the graphs has a similar pattern, indicating that there are no distinguishable variations between the three plastic media. In conclusion, there are no significant differences in the mean daily temperatures of any of the media on test when operated as tertiary nitrifying trickling filters. zna<br>Započela za postala za nastala za zarodnika za zarodnika za zarodnika za zarodnika za zarodnika za zarodnika z<br>Započela za nastala za nastala za nastala za zarodnika za zarodnika za zarodnika za zarodnika za zarodnika

The figures demonstrate that the blast furnace slag filter initially had better heatretention characteristics than the plastic media. This was due to the high biomass content of the slag filter following the high loadings during phase I of the research. As the

excess biomass was lost, the air spaces between pieces of media were reopened and the temperature of the blast furnace slag filter approached that of the plastic filters.







 $-6$ Apr May Jun Jul Aug Sep Oct Nov Dec Jan Feb Mar 1991 1992 Date Figure 6.28: The difference in temperature between the blast furnace slag and the Flocor RS pilot filters.

The data were also analysed to determine the extent of the diurnal variations in temperature. It is anticipated that the greater specific heat capacity of the slag medium will manifest in a greater stability of temperature.

Table 6.11: Temperature data for the pilot scale filters (April 1991 - April 1992).



In contrast to the activated sludge process, the retention time of a trickling filter is related to the filter depth and the specific surface area of the medium, as well as the hydraulic loading rate. The following relationship has been used to express the retention time (in days) (Howland, 1958; Sinkoff *et al.*, 1959):

The tabulated values demonstrate that the blast furnace slag medium maintained a marginally more stable temperature than the plastic media. The stability enables the biomass to maintain a more constant rate of activity, thereby minimising the variation in the penetration of nutrients through the layers of biofilm and retaining a greater total biomass population.

$$
t = \frac{CA^m d}{Q^n} \tag{6.5}
$$

where C, m and n are dimensionless constants, d is the filter depth (metres), A is the specific surface area of the medium  $(m^2.m^3)$ , and Q represents the hydraulic loading rate  $(m^3, m^{-3}, d^{-1}).$ 

# Since the values of C, A and m are constant for a given medium, Equation 6.5 may be simplified as follows:

$$
= \frac{C'd}{Q''}
$$
 (6.6)

where C' incorporates the terms C, A and m (units  $m^{-1}$ ).

Values for the constants C' and n were determined for the four media in the pilot filters by measuring the retention time at different hydraulic loading rates. The results are illustrated in Figure 6.29. Data are plotted as logarithms to obtain linear relationships where the gradient is equal to  $-n$  and C' may be obtained from the intercept and the filter depth. Values derived from the figure are summarised in Table 6.12. Only three data points were obtained for each filter, but the good linearity indicates that the derived constants may be considered accurate in each case.

Values of n, which are dependent on the geometry and packing of the medium, have been reported by other workers to lie within the range 0.3  $1.0$  (ALKINSON & All, 1976; Jiménez et al., 1988). However, these studies used different experimental conditions (very high hydraulic loads and submerged packed beds respectively).

Table 6.12: Constants relating the retention time to the hydraulic load for the pilot

#### scale filter media (derived from Figure 6.29).

	Etapak 210	<b>Blast</b> furnace slag	Etapak 160	Flocor RS
Value of n (no units)	0.93	.44	1.25	1.35
Value of $C^{*}$ $(m^{-1})$	0.016	0.184	0.044	0.062


The higher values of n determined in these experiments, for trickling filters operated at conventional loadings, indicate that the hydraulic load has a greater effect on the retention time than was previously thought. This is believed to be due to the thin-film flow characteristics of conventionally loaded filters; as the hydraulic loading increases, the liquid film depth increases thereby overcoming surface tension effects and increasing the velocity of flow down the filter.

time. The retention time is proportional to the rate of BOD removal (Equation 6.1) and the filter depth is related to the nitrification rate (Figure  $6.20$ ), hence, if temperature

The exception is the Etapak 210 filter, where the different filter design allowed a degree of ponding at the base of the filter. This had the effect of reducing the apparent value of n. Furthermore, the distribution nozzle for this filter produced a considerably wider

spray pattern at high hydraulic loadings, which may have introduced some wall effects.

Equation 6.6 shows that C' and the filter depth, d, are both proportional to the retention

effects are ignored, the performance of a filter may be gauged by the product of the depth and the value of C'.

The value of C' determined experimentally for blast furnace slag was three times that of Flocor RS, and over four times that of Etapak 160. Comparable retention time would therefore be expected of a blast furnace slag filter of 1.2 m depth, a Flocor RS filter of 3.6 m depth and an Etapak 160 filter of 5m depth.

#### 6.2.7 Biomass accumulation on the filter media

At equilibrium, the rate of biomass accumulation is equal to the rate of loss through sloughing. Although filters may take several years to reach full equilibrium from startup, the latter stages of maturation mainly involve stabilisation of the macro-organism population. Furthermore, the pilot filters were dosed with settled crude wastewater during phase I to accelerate start-up by rapidly coating the media with biomass to facilitate attachment. It is therefore anticipated that, following 18 months of maturation

Measurements were taken at approximately two-month intervals during the period of research. Readings were taken at various depths in each filter to determine the distribution of biomass. Readings are adjusted by reference to a blank measured in

in a nitrification role, the biofilm within each pilot filter will be close to the equilibrium

thickness allowing for seasonal variation.

The biomass population of the filters was measured by two methods: non-intrusively using a neutron probe, and destructively by dismantling and emptying the filters at the end of the period of research. While other researchers have included removable media baskets in pilot filters to enable limited biomass sampling (Gray & Learner, 1984), it is believed that this is the first time a complete pilot scale filter has been dismantled for biomass measurement.

#### 6.2.7a Neutron probe measurement

August 1990 on start-up (before any biomass attachment had occurred). The moisture measured in each blank comprises "bound" water (water that does not drain from the filter) as well as neutron absorbance by the medium itself. The importance of the blank measurement is discussed further by Harvey et al. (1963). Higher readings taken subsequently are due to neutron absorbance by biomass (active or inactive), macroorganisms, water bound to biomass and inert humic solids within the filters.

Figure 6.30 illustrates the extremely rapid start-up achieved during phase 1 of the research (up to February 1991). Indeed, it is probable that the blast furnace slag filter

Month/Year  $\text{I}-$ Etapak 210 + Blast Furnace Slag  $\text{I}-$ Etapak 160  $\text{I}-$  Flocor RS ASONDJFMAMJJASONDJFMAMJ<br>1990 | 1991 | 1992  $1990$  1  $1991$  1  $1992$ Figure 6.30: Mean moisture volume fractions for each pilot filter medium versus time.

The neutron probe technique provides a measure of the percentage moisture volume fraction (% MVF), which represents the fraction of the volume sampled which contains hydrogen atoms bound to oxygen (assumed to be water). The % MVF is also referred to as the mean saturation of voids (Gray & Learner, 1984). The mean % MVF for each medium are plotted in Figure 6.30.

![](_page_146_Figure_3.jpeg)

would have ponded had the feed strength not been reduced. It is also notable that the blast furnace slag filter continued to lose biomass throughout phase 2 of the research, hence the period of start-up may have been too long.

Another notable feature of the figure is the distinct period of Spring sloughing between December 1991 and February 1992. The phenomenon is not well-defined in the previous year due to the concurrent change from phase 1 of research to phase 2.

Biomass accumulation in the plastic media filters occurred much more slowly and to a lesser extent than the slag filter. This is due to a lower rate of biomass growth on the plastic media as well as a reduced accumulation of humic solids by physical filtration

of the effluent due to the higher voidage of the plastic media.

ASONDJFMAMJJASONDJFMAMJJA<br>1990 | 1991 | 1992 1990 1 1991 1 1992 Month/Year  $-0.3m + 0.6m \times 0.9m$   $\pm 1.2m - 1.5m$ Figure 6.31: Moisture volume fractions at various depths in the Etapak 210 filter.

To determine the extent of growth at different levels in each filter, the data are replotted in Figures 6.31 to 6.34. With the exception of the Etapak 160 medium, it can be

![](_page_147_Figure_6.jpeg)

![](_page_148_Figure_0.jpeg)

![](_page_148_Figure_1.jpeg)

ASONDJFMAMJJASONDJFMAMJJA<br>1990 | 1991 | 1992 1990 1 1991 1 1992 Month/Year  $-0.3m + 0.6m \times 0.9m$ 

Figure 6.33: Moisture volume fractions at various depths in the Etapak 160 filter.

![](_page_149_Figure_0.jpeg)

seen that colonisation of the filters began in the upper layers and gradually spread downwards. This is to be expected, since the concentration of nutrients is highest at the filter surface, hence growth rates are faster. It is thought that the different maturation pattern of the Etapak 160 medium is due to the very open nature of the medium, hence the upper layers are more subject to cooling and to predation of biomass by insects.

The figures accentuate the extreme variation in the biomass content of the plastic media filters with time. In contrast, the blast furnace slag medium exhibits even biomass colonisation throughout the filter depth and at all times of the year. This may be due to the improved heat retention, lower voidage and the presence of many small pores in the

medium which inhibit grazing by macro-organisms. However, the small scale of the filters may have exaggerated the variation in the  $\%$  MVF of the plastic media filters, since the variation in the larger Etapak 210 medium filter is notably less than for the smaller filters.

### <u>6.2.7b</u> Biomass determination by dismantling the filters

In August 1992, the pilot scale filters were sequentially decommissioned and emptied. The medium was removed from each filter within six hours, and samples of media with biomass attached were taken at regular intervals down the depth of each filter. Biomass was quantified by measuring the volatile matter attached to each piece of media. To reduce sampling errors, a large number of media pieces were analysed.

Blast furnace slag is stable at high temperature hence the volatile matter was measured by weighing the media after drying at  $105^{\circ}$ C and at  $600^{\circ}$ C. However, since the biomass could not be removed from the slag medium, the dry matter and the percentage of volatile matter in the biomass could not be determined.

PVC and polypropylene melt at just over  $100^{\circ}$ C, so the media was dried at 35 $^{\circ}$ C to loosen the biomass which was then scraped off and collected for analysis. Other methods for removing biomass using organic solvents or acids were rejected because they were found to be ineffective and may have affected the results. A summary of the results is tabulated in Table 6.13 and the full data are listed in Appendix 7. The volumetric volatile matter content of each filter at various depths is also plotted in Figure 6.35.

The results confirm those obtained by the neutron probe: the blast furnace slag filter contains considerably more biomass per unit volume than the plastic media filters. The distribution of biomass within the slag filter is also more consistent than that of the plastic media filters, with the possible exception of the Etapak 210 filter where the increased scale may have affected the results.

An interesting feature of all four curves in Figure 6.35 is that the upper few centimetres of each filter carried significantly less biomass than underlying layers. This is due to a combination of predation by macro-organisms (mainly insects, worms and snails) and to the effects of exposure (increased sunlight and greater fluctuations in temperature).

![](_page_151_Figure_0.jpeg)

Many full scale filters, particularly those built below ground level, cannot be readily monitored by direct examination of media from the lower depths of the filter. While not necessarily an accurate method for measuring biomass growth, the neutron probe he represents a rapid non-intrusive method for estimating growth within such filters to

This observation is significant for the correct operation of filters, since operators generally rely on an examination of the surface to determine whether a filter is in danger of ponding. These results concur with the conclusions of Gray & Learner who stated that surface observation is not a reliable method; a filter may already be ponding below the surface before any effects are visible (Gray & Learner, 1984).

![](_page_151_Picture_4.jpeg)

# Table 6.13: Summary of data obtained during dismantling of the pilot scale filters.

![](_page_152_Picture_39.jpeg)

## Chapter 7: Discussion of full scale research

### 7.1 Comparison between full scale and pilot scale results

The full scale filters at Cranfield STW contain Etapak 160, one of the media evaluated in the pilot scale filters. In order to examine the effects of scale and to validate the pilot

scale results, this section will discuss the correlation between pilot and full scale data.

Unfortunately it was not possible to design the pilot scale filters to match the full scale units, in particular the dosing system and the elevation of the filters. Furthermore, because of the low average loading applied to the full scale filters, it was decided to operate the pilot scale filters at a rather higher loading to accelerate start-up. Consequently, some deviations in performance between the pilot and full scale filters are attributable to differences in the filter temperature, hydraulics, or other factors affected by these variations in design.

Furthermore, the configuration of Cranfield STW precludes routine sampling of the separate effluents from the first and second stage filters because the two effluents are mixed at the inlet to the humus tank. Mid-treatment samples from the works therefore comprise a mixture of secondary and recycled effluent, hence the performance should be adjusted accordingly in comparisons with pilot scale results.

During Phase I of the pilot scale research, the pilot filters were dosed with the same settled raw wastewater as the full scale filters. The volumetric loading rate of the pilot filters was set at  $1.4 \text{ m}^3 \text{.m}^{-3}$ . d<sup>-1</sup>, approximately double that of the full scale filters (it was not practicable to mimic the diurnal load fluctuations of the full scale filters).

The mean removal rates of BOD, suspended solids and ammoniacal nitrogen during phase I of research are summarised in Table 7.1, together with the data for the pilot scale filters taken from Table 6.2.

# Table 7.1: Comparative performance of the full scale secondary filters and the pilot scale filters during phase 1.

![](_page_154_Picture_87.jpeg)

three types on trial) failed to match the rate of BOD removal in the full scale works. This may be accounted for by the fact that the growth of biofilm on the plastic media was too slow to approach a steady-state within the six month period, as discussed in Section 6.2.7.

The above table demonstrates that, even allowing for the recycle in the full scale works, the performance of the pilot scale Etapak 160 filter during the six months of start-up did not achieve the performance of the full scale plant. Even the Etapak 210 and Flocor RS media (with higher specific surface areas, the better-performing plastic media of the

Similarly, the sparse biofilm growth in the pilot filters reduced the capture of solids and precluded significant nitrification.

In contrast, the blast furnace slag medium achieved superior BOD, solids and ammonia removal, but the accumulation of solids within the medium was so rapid that ponding was imminent (Section 6.1.2).

It was therefore not feasible to continue phase I of the pilot scale research and allow the plastic media filters to mature more fully. Research by Wheatley (1976) has shown that plastic filters may take several years to fully mature, and this time was not available within the period of research. It was consequently not possible to verify that the performance of the pilot and full scale filters at Cranfield STW were comparable.

To verify the kinetics of BOD removal, the applied and effluent loads from the full scale first stage filters at Cranfield STW are plotted in Figure 7.1. The straight line on the graph verifies that the kinetics are pseudo-first order and indicates that the filters achieve

a consistent rate of removal despite occasional fluctuations in the load.

Figure 7.2 illustrates the relationship between the applied ammonia load and the rate of nitrification in the first stage of the full scale filters. The kinetics appear to be pseudo zero order, but this is due to the high residual ammonia concentration ( $>>K_{\rm sv}$ ); at lower residual concentrations it is expected that the curve would deviate from the straight line. In contrast to BOD removal, nitrification can be seen to be erratic, varying between approximately 20% and 80% ammonia removal. The rate of nitrification could not be correlated to the season, the BOD loading or to the solids loading, but a slight correlation was noted with the hydraulic loading, indicating that the retention time may

### be a limiting factor.

![](_page_156_Figure_0.jpeg)

![](_page_156_Figure_1.jpeg)

## 7.2 The tertiary nitrifying trickling filter at Cranfield STW

### <u>7.2.1</u> Design & operating experience

Following the early results from this research and published experience from other purpose-built tertiary nitrifying trickling filters (Dolan et al., 1990; Hill et al., 1990 & 1992; O'Neill & Horan, 1992; Pullin, 1976; Upton & Cartwright, 1984), it was decided to construct a similar installation at Cranfield STW to improve effluent quality, particularly with respect to ammonia and suspended solids. The improvements were necessary to ensure compliance with the ammonia consent introduced in 1986 and to allow construction of additional housing within the catchment. The author assisted Dr A.D. Wheatley and Mr K.A. Johnson (then of the Biotechnology Centre at Cranfield) to design the filter.

Due to the elevation of the site, the filter had to be constructed above ground with a pumped feed. Effluent from the filter gravitates to a new circular humus tank, then back

to the pumping station where effluent may be recycled through the filter if the flow through the works is low (Section 3.3).

The filter was sized based on a hydraulic load of only  $3 \text{ m}^3 \text{.m}^{-3} \text{.d}^{-1}$  at a flow rate of  $30\%$ greater than the average flow through the works. The humus tank was designed on a mean upflow velocity of only  $0.36$  m.h<sup>-1</sup> to allow for anticipated increases in flow to the works. The tank is de-sludged manually, usually twice per day because the original humus tank upstream of the nitrifying filter is inefficient and allows a high solids load onto the nitrifying filter (Section 3.1.1c). Despite the low hydraulic load, rising sludge is not a problem in the new humus tank due to the low volatile content of the sludge

and the low metabolic rate of the sloughed biomass.

Following published experience at Wanlip and Burnley STWs (Challenger *et al.*, 1987; Hill *et al.*, 1992; Upton & Cartwright, 1984) and other reports (Vaughan & Ravetto, 1981a  $\&$  1981b), wastewater is distributed onto the filter using upward facing open-pot

spray nozzles. This method has proven to be reliable against blockages of the nozzles, but some spray drift can occur despite placing windbreaks around the periphery of the filter. The drifting problems at Cranfield are accentuated by the exposed nature of the site and by the relatively small scale of the filter when compared to the other installations previously mentioned.

7.2.2 Performance of the tertiary nitrifying filter

The filter was commissioned on 24<sup>th</sup> June 1992, although the flow rate was restricted to 3.8  $dm<sup>3</sup>. s<sup>-1</sup>$  (a hydraulic load of only 2.2 m<sup>3</sup>.m<sup>-3</sup>.d<sup>-1</sup>) due to incorrect selection of distribution nozzles. Following the installation of larger nozzles on 18<sup>th</sup> August 1992, the full design flow of 5.2 dm<sup>3</sup>.s<sup>-1</sup> (3.0 m<sup>3</sup>.m<sup>-3</sup>.d<sup>-1</sup>) was passed through the filter.

It is thought that the maturation may have been accelerated by considerable inoculation of the new filter by the original plastic-medium nitrifying filter. If attachment to the plastic medium is not effective, as suggested by the pilot scale results, the filter effluent will contain relatively high concentrations of metabolically active nitrifying bacteria.

The performance of the filter was monitored intensively until the end of the period of study in September 1992. In addition to routine sampling at the sewage works, the influent to and effluent from the filter were analysed for ammoniacal nitrogen and the

accumulation of biomass within the filter was monitored using the neutron probe.

Nitrification in the new filter is illustrated in Figure 7.3 and the data are re-plotted to show the correlation between the applied load and the rate of nitrification in Figure 7.4.

Figure 7.3 demonstrates the extremely rapid start-up achieved by the new filter; compliance with the  $7 \text{ mg.dim}^3$  consent being achieved within one week and nearcomplete nitrification within three weeks. Although commissioning took place in midsummer, hence temperatures were favourable, this must be regarded as unusually fast initiation of nitrification.

![](_page_159_Figure_0.jpeg)

![](_page_159_Figure_1.jpeg)

This explanation is given further credence by the observation that the humus tank (downstream of the original nitrifying filter) frequently suffers from rising sludge as metabolically active solids in the effluent produce gases.

Figure 7.4 shows that nitrification within the new filter appears to be zero-order with respect to the ammonia loading at the loads applied so far. However, it is anticipated that the curve will flatten at higher loadings as the effluent ammonia concentration increases and the kinetics change from apparent zero-order to Monod-type. This transition may be seen clearly in the published data from Burnley STW, where a much larger but similarly designed nitrifying filter has been operating since 1986. Figure 7.5 represents a summary of the data obtained at Burnley STW during the experimental phase of operation when the hydraulic load was varied between 2 and 24  $m^3.m^{-3}.d^{-1}$ 

Comparison of the influent and effluent ammoniacal nitrogen concentrations in Figure 7.3 demonstrate that the commissioning of the new filter has enabled compliance with

(Challenger et al., 1987). For comparison, the data from Cranfield STW (Figure 7.4) are superimposed on the Burnley data.

The data obtained from Cranfield STW indicate a significantly better performance than the Burnley filter, despite the similar design, media and effluent strength. However, the Burnley data were obtained during the months September to February, when average temperatures are lower, hence a reduced rate of nitrification is expected.

![](_page_161_Figure_0.jpeg)

 $-$ Ammonia Removed  $-$  Cranfield Data  $-$  % Ammonia Removal

Figure 7.5: Ammoniacal nitrogen removal versus the applied loading at Burnley STW (Challenger et al, 1987) with data from Cranfield STW superimposed.

## Chapter 8: Conclusions

- 8.1 Laboratory scale research
- 1) Nitrifying organisms do not grow well in the first defined medium used (Section 5.1.1 and Appendix 3.1.1). However, successful growth was achieved

- 2) The minimum pH reached by the nitrifying culture was 4.3 for growth in sewage works effluent and 3.8 for growth in activated sludge. This demonstrates that, if permitted to acclimatise, nitrifiers can tolerate a pH lower than the pH 4.9 previously reported (Prakasam & Loehr, 1972).
- Temperature has a logarithmic effect on ammonia oxidation up to a maximum  $3)$ rate at  $37^{\circ}$ C, above which the rate tails-off rapidly. For the logarithmic phase,
	-

using a slightly different medium (Appendix 3.1.2), suggesting that molybdenum may be an essential requirement for growth of nitrifiers.

where the experimentally measured value for  $k_T$  of 0.093°C<sup>-1</sup> concurs with previously published data (Downing & Knowles, 1966; Ekama & Marais, 1984; Knowles et al., 1965).

- 4) Although nitrification is negligible at  $50^{\circ}$ C, cells remain viable and will resume activity on a reduction in temperature.
- 5) The optimal pH for nitrification in pure culture lies in the range 7.8-7.9 pH units. The effect of pH on the rate of ammonia oxidation can be closely approximated in the range pH 5.8-7.8 by the following equation:

the following equation applies:

$$
\mu = \mu_{15} e^{k_T (T-15)}
$$
 (8.1)

$$
\mu = \mu_{Max} (0.50pH - 2.90) \qquad (8.2)
$$

These results are consistent with published data by other workers using nitrifying activated sludge (Antoniou *et al.*, 1990; Wild *et al.*, 1971), but differ from Downing's equation which has been adopted by the U.S. E.P.A.:

- 6) In pure culture and without time for acclimatisation, nitrification follows Monod kinetics with a measured value for the half-saturation coefficient,  $K_{SN}$ , of 39 mg  $NH_4$ -N.dm<sup>-3</sup>. It is believed that this high value is due to non-limiting ammonia concentrations in the culture medium resulting in inefficiency of the bacterial system for the uptake of ammonia. This may also account for the wide variation in reported values for  $K_{SN}$ .
- 7) An increase in the BOD (as glucose & glutamic acid) resulted in an increase in the rate of nitrification in pure culture. This suggests that *Nitrosomonas* may be a facultative chemolithotroph, rather than obligate. This result may not be seen in mixed culture due to the dominant effects of competition by heterotrophs.
- 8) Inhibition of nitrification by allylthiourea was found to be 99% complete at the recommended concentration used for the BOD test. The half-saturation constant for inhibition was measured as  $7.0 \mu$ g.dm<sup>-3</sup>.

$$
\mu = \mu_{Max} (1 - 0.83(7.2 - pH))
$$
 (8.3)

a study into the nutritional requirements of nitrifying bacteria to confirm the requirement for molybdenum and whether Nitrosomonas is obligately or

This research has revealed several areas for further work, including:

 $\overline{\phantom{m}}$ 

facultatively chemolithotrophic.

an investigation into the acclimatisation of Nitrosomonas to substrate concentration, temperature, pH and inhibitor concentration.

#### 8.2 Pilot scale research

No correlation was found between the rate of nitrification and the temperature, 6) although it is thought that this was an artifact due to the small diameters and

- 1) Under high loadings (phase 1), the blast furnace slag medium was more rapid to acquire an active biological population than the plastic media, although excess accumulation of biomass and ponding were imminent after only five months.
- 2) The blast furnace slag medium achieved an average of 85% BOD removal and 60% nitrification during phase 1 whereas the similarly-loaded plastic media achieved only 33-60% BOD removal and 1-22% nitrification.
- 3) The wastewater used for the pilot scale nitrification study is typical of the final effluent from an under-performing trickling filter works where tertiary nitrification may be usefully employed. The exception is that the wastewater used in this study is unsettled, hence it contains an unusually high concentration of flocculated solids.
- 4) BOD removal is confirmed as a pseudo first order reaction and normalised BOD removal constants have been determined for the four media.
- 5) Consistent nitrification was achieved by all media in the study, but higher rates

were achieved by the blast furnace slag. A set of design curves have been derived for use in designing slag medium filters.

exposed location of the pilot scale filters.

7) Some denitrification was detected. Since the dissolved oxygen concentration in the filter effluent remained above 4 mg.dm<sup>-3</sup>, anoxia was due to excessive depth of the biofilm rather than a lack of ventilation.

- to obtain an average of about 60% removal of suspended solids.
- 9) Due to the higher rates of BOD removal and nitrification, solids generation in the blast furnace slag filter was greatest but the solids were more easily settled than the plastic media filters.
- 10) Apart from when the blast furnace slag medium was nearly ponded, there were no significant differences in the mean daily temperatures in the pilot filters. However, diurnal fluctuations in the temperature were significantly greater in

8) All media except Etapak 160 achieved good solids flocculation in the effluent

rapid qualitative determination which may be of use in diagnosing problems with trickling filters.

the plastic media.

- 11) Sinkoff s equation (relating the hydraulic retention time to the hydraulic load and the media characteristics) has been used to determine experimental values for the constants C' and n for each media (Sinkoff *et al.*, 1959). The values of the constant n are believed to be greater than unity at conventional hydraulic loadings due to surface tension effects.
- 12) Correlation between neutron probe and direct analytical methods for measuring biomass in the filters was poor, but the neutron probe was found to give a

Unfortunately, there were insufficient time and resources to enable further studies of the attachment of nitrifying organisms to the support medium. However, these studies

suggest that the course surface texture and porous nature of the blast furnace slag significantly enhance the development of an active bacterial population. This could be confirmed using electron microscopy to study the attachment of bacteria to the media.

Although the use of a real wastewater and an external location ensured the validity of the data, the consequent variation in performance of the filters made interpretation difficult and masked some of the responses which have been well-proven  $(e.g.$ temperature dependence of nitrification). To improve the significance of the conclusions of further pilot scale studies, a compromise should be reached to allow some control over climatic variables without jeopardising the validity of the data.

#### 8.3 Full scale research

1) Data collected over a six-year period from the mature full scale filters at Cranfield STW verify the pseudo first order kinetics of BOD removal.

- 2) Nitrification in the first stage filters appears to be zero-order, but this is due to a high residual ammonia concentration (hence the ammonia concentration remains much greater than  $K_{SN}$ , the half-saturation constant for nitrification). Nitrification is believed to be unreliable due to variations in the hydraulic load to the filters.
- $3)$ Results obtained from pilot scale research have been used to design a full scale nitrifying trickling filter at Cranfield STW. Initial results indicate that the filter started-up extremely rapidly, achieving consent compliance within one week. 4

The new nitrifying filter at Cranfield STW was equipped with aluminium access tubes for neutron probe analysis and with sampling shafts to enable samples to be taken at various depths. The filter is divided into two sections which may be dosed at different hydraulic loads. This installation provides a good opportunity for further research into the effects of hydraulic load on biofilm accumulation and filter performance.

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At equilibrium, the concentrations of the ionised and unionised forms of any dilute acid or base can be related to a dissociation constant,  $K_a$  (acid) or  $K_b$  (base):

#### Appendix 1: Dissociation of ammonia and nitrous acid

#### 1.1 Dissociation constants

The dissociation constants for acids and bases are reported as the negative logarithms,  $pK_a$  and  $pK_b$  respectively.

For any dilute aqueous solution,  $pH + pOH = 14$ 

$$
K_a = \frac{[H_3O^+][A^-]}{[HA]}
$$

$$
(A1.1)
$$

$$
\boldsymbol{K}_{b} = \frac{[\boldsymbol{B}\boldsymbol{H}^{\dagger}][\boldsymbol{OH}^{\dagger}]}{[\boldsymbol{B}]}
$$
 (A1.2)

where the dissociation of an acid is represented as:

$$
HA + H2O = A- + H3O+
$$
 (A1.3)

$$
B + H2O \rightleftharpoons BH+ + OH-
$$
 (A1.4)

#### 1.2 Dissociation of ammonia

The dissociation contant,  $pK_b$ , for ammonia is dependent on the temperature, but at 10-35'C the value is approximately 4.77. The pH of an ammonia solution can be related to the ratio of ammonium ions to ammonia by substituting for  $[OH]$  in Equation A1.2:

#### Therefore:

$$
\frac{[NH_4^+]}{[NH_3]} = 10^{(9.23-pH)}
$$

$$
(\mathsf{A}1.6)
$$

$$
K_b = \frac{[NH_4^+] . 10^{(\rho H - 14)}}{[NH_3]} = 10^{-4.77}
$$
 (A1.5)

The relationship between the pH of a dilute solution and the ratio of ammonium ions to ammonia is represented in Figure A1.1.



Figure A1.1: The concentration ratio of ammonium ions to ammonia in dilute solution at pH 0-14.

#### 1.3 Dissociation of nitrous acid to nitrite

The dissociation contant,  $pK_a$ , for nitrous acid at between 10 and 35 $\degree$ C is approximately 3.35. The pH of a solution of nitrous acid can thus be related to the ratio of nitrous acid to nitrite by substituting for  $[H_3O^+]$  in Equation A1.1:

$$
K = \frac{[NO_2] . 10^{-pH}}{=} = 10^{-3.35}
$$

 $(A1.7)$ 





The relationship between the pH of a dilute solution and the ratio of ammonium ions to ammonia is represented in Figure A1.2.



#### Appendix 2: Lithium standard curve for retention time analysis

Standard curves for the determination of lithium by flame photometry were prepared when required using a standard 1 g.dm<sup>-3</sup> solution of lithium (Hach). An example of a standard curve is presented below.



Lithium concentration  $(mg.dm<sup>-3</sup>)$ 

Figure A2.1: Typical standard curve for lithium concentrations determined by flame photometry.

#### Appendix 3: Composition of media and reagents

#### 3.1 Media used for the growth of nitrifying organisms

#### 3.1.1 Mineral-supplemented medium

The mineral-supplemented medium used for the cultivation of an enriched culture of nitrifying organisms (Section 5.1.1a) was composed as follows:



The medium for the selection of nitrifying organisms in pure culture (Section 3.8.4) was composed as follows:

Magnesium sulphate (heptahydrate)



Ferric chloride (hexahydrate)

Ammonium molybdate

Distilled water

 $MgSO<sub>4</sub>$ .7 $H<sub>2</sub>O$  $100 \text{ mg.dim}^{-3}$  $FeCl<sub>3</sub>.6H<sub>2</sub>O$  7 mg.dm<sup>-3</sup>  $(NH_4)Mo_7O_{24}.4H_2O$  6 mg.dm<sup>-3</sup> to  $1 \text{ dm}^3$ 

To each dm<sup>3</sup> of this medium was added 1  $cm<sup>3</sup>$  of indicator solution:

Ferrous sulphate (heptahydrate) Di-sodium EDTA Phenol Red Distilled water  $FeSO<sub>4</sub>$ .7H<sub>2</sub>O  $Na<sub>2</sub>H<sub>2</sub>C<sub>2</sub>H<sub>4</sub>N<sub>2</sub>[CH<sub>2</sub>COO<sup>-</sup>]$ 450 mg.dm $^{-3}$  $250 \text{ mg} \cdot \text{dm}^{-3}$ 250 mg.dm<sup>-3</sup> to  $1 \text{ dm}^3$ 

The medium was sterilised at  $121^{\circ}$ C and 1 bar<sub>g</sub> for 15 minutes in an autoclave and allowed to cool. Sterile  $\text{Na}_2\text{CO}_3$  (50 g.dm<sup>-3</sup>) was then added to neutralise the solution (indicated by a pink colouration) before use.

#### 3.2 Determination of nitrite concentration

#### Dissolve the sulphanilic acid in a warm mixture of acetic acid and distilled water. Filter, make up to 1 dm<sup>3</sup> with distilled water and store in the dark.

Reagent II:

The concentration of nitrite was determined using Griess-Ilosvay's Reagents I and 11.

#### Reagent I:

Sulphanilic acid

3.33  $g$ .dm<sup>-3</sup>

Glacial acetic acid

 $200 \text{ cm}^3 \text{ dm}^{-3}$ 

1-naphthylamine-7-sulphonic acid Glacial acetic acid

 $1.33$  g.dm<sup>-3</sup> 80 cm<sup>3</sup>.dm<sup>-3</sup>

Add the glacial acetic acid to the 1-naphthylamine-7-sulphonic acid slowly and with continuous stirring. Dilute with distilled water, filter, make up to  $1 \text{ dm}^3$  with distilled water and store in the dark.

An aliquot of sample, less than  $40 \text{ cm}^3$  and containing less than 10 µg of nitrite as N,

was added to a 50 cm<sup>3</sup> volumetric flask and diluted to approximately 40 cm<sup>3</sup>. To each flask was added 2 cm<sup>3</sup> of reagent I and the flask was mixed thoroughly. The reaction was left to stand at room temperature for 20 minutes, then 5 cm<sup>3</sup> of reagent II was added. The mixture was diluted to 50 cm<sup>3</sup>, mixed, left to stand for a further 20 minutes, then the optical density of the sample and a blank were read at 525 nm using a Cecil CE594 double beam spectrophotometer. The optical density was then converted into

nitrite concentration using a calibration curve similar to Figure A3.1.





### Appendix 4: Enrichment of a nitrifying culture - experimental data

#### Table A4.1: Selection of Nitrifiers in a Defined Medium



Table A4.2: Selection of Nitrifiers in a Non-Defined Medium

				Rate of	
Time	pH	Ammonia-N	Nitrate-N	nitrification	
(days)		$(mg.dm-3)$	(mg.dm <sup>3</sup> )	$\sqrt{\frac{mg.dim^{-3}.d^{-1}}{m}}$	
		17.5	$\overline{16.0}$		
		$\mathsf{I7.4}$	16.8		
		16.6			
		.7.3	16.1		
		$\vert$ $7.0$	16.6		
		16.3	73		
		16.0			
$\mathbf{A}$					
		235			
		$\mathbf{\Omega}$			
		20.2	39.5		
I Y	6.5	$\blacksquare$			
		◠	56.0		
	v.J				
سک د			22.6		
		32.8			
ا سماسته	0.0 <sub>1</sub>	18.9	41.0	⌒ $1 \cup . \cup$	
	Г.	17.2	37.5		
	$\overline{ }$ O,				
26	5.6	$\blacksquare$			
28		<u>11.2</u>	28.5		
$\Omega$ $\Omega$	4.3	10.3	2Q 30.1	V.4	



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#### Table A4.3: Selection of Nitrifiers in Activated Sludge

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# experiments  $\cdot$  ,  $\overline{ }$  $\mathbf Q$  $\overline{\phantom{0}}$  $\overline{\phantom{a}}$ **CD UQ CIIO**  $\mathfrak{m}$ <u>ra</u>  $\Box'$  $\dot{S}$ Appendix

# 二 effect of temperature on nitrificatio<br>initial ammonia-N concentration, pH 7.6)



Table AS.1: The (using 20 mg. dm<sup>-3</sup>

		$\frac{0.0}{0.0}$	30.2	262			$\frac{4279}{9794}$		4268	5295	5530	403
	24.0		0.06	233	390	879	1906		3820	4943	5148	40 <sub>1</sub>
		$\frac{8}{21}$	25.2	202		$\frac{319}{798}$		$\frac{1661}{3416}$			4636	411
		$rac{1}{\sqrt{10}}$					$\left \frac{5!}{7!}\frac{1!}{2!}\frac{2!}{2!}\right $	$\frac{ 826}{2118}$			922	383
								$\frac{1}{2} \left  \frac{1}{2} \right  \frac{1}{2} \left  \frac{1}{2} \right  \frac{1}{2} \left  \frac{1}{2} \right  \left  \$				
								$\frac{1}{2} \frac{1}{2} \frac{$				
Time	<b>lours</b>	ontrol	$\bullet$	$\overline{\mathcal{A}}$	$\sum$			52525			$\left  \frac{4}{3} \right $	50
	id E	under C O										

 $\overline{Q}$  $\mathbf{\hat{5}}$  $\overline{10}$ 

effect of temperature on nitrification<br>initial ammonia-N concentration, pH 7.6)

when cooled Table A5.3: The effect of heating at 50°C on nitrification<br>(using 200 mg.dm<sup>-3</sup> initial ammonia-N concentration, pH 7.6).



#### The  $mg.dm<sup>-1</sup>$ 200 AS Table dsing

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Table A5.4: The effect of pH on nitrification.<br>(using 200 mg.dm<sup>-3</sup> initial ammonia-N concentration







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concentration The effect of ammoniacal nitrogen<br>oncentrations, pH 7.6, 32°C). The  $\mathbf{\mathsf{o}}$ ammonia  $\dot{\bm{\circ}}$  $\mathcal{L}$  $\blacktriangle$ Table (low

#### effect of concentration The (high ammonia  $\dot{5}$  $\bullet$  $\boldsymbol{\mathcal{S}}$  $\blacktriangleleft$ Table.

# acid. glucose/glutamic



7.6).



**as** BOD BOD 1 contained no inoculum and 0 mg.dm<sup>-3</sup> I<br>2 contained no inoculum and 75 mg.dm<sup>-3</sup> Control itrol

effect of The  $\dim$  $mg$  $\bullet$  .  $\bullet$  $\overline{ }$  $\bullet$ 200  $\mathbf{v}$  $\mathcal{A}$ Table<br>(using



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 $\overline{a}$ 



Table A5.8: The effect of<br>(using 200 mg.dm<sup>-3</sup> initial am



 $\frac{1000 - 000}{1000}$  experiment 2. effect of allylthiourea on nitrification<br>initial ammonia-N concentration, 32°C, pH Table A5.9: The e

# Appendix 6: Pilot scale filter performance data - phase <sup>I</sup>





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# Appendix 7: Pilot scale filter performance data - phase 2

Table A7.1: Filter performance data



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## Table A7.2: Pilot scale filter temperature data



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 $\sim 40\%$ 



 $\sim 10^{-11}$ 



 $\mathcal{L}(\mathcal{A})$  .



\* The mean was calculated by excluding data from periods when a thermistor cable was damaged to ensure valid comparison.

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\* The mean was calculated by excluding data from periods when a thermistor cable was damaged to ensure valid comparison.





Volume Fractions (without adjustment for media)





adjustment for media)

Volume Fractions (after











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80 kg.m<sup>-3</sup> ssumed bulk density:  $\Delta$  $\ast$ 

 $m^2.m^{-3}$ . 200 area: surface specific Assumed  $*$ 

# slag furnace Blast  $\ddot{2}$

Filter

 $\sim 100$  km s  $^{-1}$ 



1350 kg m<sup>-3</sup> area: ပ္လ surfa density specific values ssunned bulk Assumed bemmes  $\mathcal{A}$ ⊲.  $*$ 

 $\bullet$ 

 $\mathbf{m}^2$ 

 $\mathbf{\overline{u}}$ 

 $70$ 

 $\bullet$   $\bullet$ 

 $\bigstar$ 





 $3082$ vera epth filte  $\overline{\text{cm}}$  $\overline{\bigcirc}$  $\Box$ 











face area: 150 m<sup>2</sup>.m<sup>2</sup>.  $70 \text{ kg} \cdot \text{m}^{-3}$ surf Assumed bulk density: specific Assumed  $* *$  $\ast$ 

RS Flocor  $\ddot{ }$ 

 $m^2.m^{-5}$ . face area: 230 50 kg.m<sup>-3</sup> surf Assumed bulk density: specific! Assumed  $*$  $\ast$ 



Filter



217

 $\langle \bullet \rangle$  ,  $\alpha$  ,  $\gamma$ 

## Appendix 8: Performance data for Cranfield STW (1986-1992)

Table A8.1: Performance prior to commissioning the new nitrifying trickling filter









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 $\sim 200$  km s  $^{-1}$ 







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#### Appendix 9: Performance data from the new nitrifying , trickling filter at Cranfield STW





### Appendix 10: Previous publications

## Nitrification in Trickling Filters; some Further Research.

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## published in Nutrient Removal from Wastewaters, Editor N.J. Horan, Technomic, U.S.A.

In the past, many sewage treatment works have been designed without any specific requirement for nitrification. More recently however, the initiative towards cleaner rivers and the general acceptance of a minimum class 11 enforced by the National Rivers Authority (NRA) has resulted in more stringent ammonia discharge consents. Whereas only a few years ago, permissible ammonia concentrations were as high as 20 or 30 mg. 1<sup>-1</sup>, many sewage works are now having consents of  $5-10$  mg.<sup>1-1</sup> imposed upon them.

### Introduction

The incorporation of a nitrification stage into the design of a new sewage works is easily accomplished by elongation of activated sludge lanes or of rotating biological contactor (RBC) units. However, the difficulty and expense of up-rating an existing plant has led to an increase in the popularity of separate tertiary treatment stages. Such systems can often be

Nitrifying trickling filters offer a relatively cheap method of tertiary biological nitrification. They are easy to design and construct, have been well researched and characterized (I.W.E.M., 1988; Upton & Cartwright, 1984; Hill *et al*, 1992) and offer simple and reliable operation. The space requirements, while not as small as submerged biological filters, are nevertheless within reason for most sites. Often only the filter itself is needed, although subsequent settlement may be required depending on the suspended solids permissible in the final effluent.

retro-fitted while leaving the upstream works operational.

The threat of tighter restrictions on total nitrogen discharges from large sewage treatment works suggests that nitrifying trickling filters, which can not readily denitrify, will be of limited use for these works in the future. However, there are a great number of smaller sewage treatment works which are facing ammonia discharge limits but where denitrification is not required. Little information is available on the performance of nitrifying tricking filters following high rate filtration in a small sewage works, or on the use of random plastic media in nitrifying filters.

This paper describes the results from pilot scale research where a comparison is made between four different types of support media in terms of their suitability for use in nitrifying trickling filters following high-rate filtration. In doing so, the basic design criteria for nitrifying trickling filters are discussed.

### The Characteristics Required for a Nitrifying Filter Medium

The support medium in a nitrifying trickling filter has two purposes: to provide an attachment surface for the nitrifying bacteria and to facilitate the passage of water and air through the filter.

The requirement for attachment is easily achieved to an extent because the bacteria exploited in sewage treatment can attach readily to most surfaces using an extracellular polysaccharide adhesive (Fletcher et al., 1976). However, the thickness of the bacterial film is limited by three factors: the liquid shear stresses on the surface of the film, the diffusion of nutrients (such as oxygen and ammonia) to the cells at the base (Harremöes, 1978) and the activity of grazing organisms. Liquid shear stresses should be minimized for an effective nitrification

medium since the nitrifying organisms may be restricted by competition from heterotrophic organisms at higher loading rates.

Nutrient diffusion limitations and grazing are thought to be responsible for the spring sloughing observed in trickling filters. During the winter, the activity of grazing macrofauna in the filter slows down, allowing a gradual thickening of the biofilm. As temperatures increase again during spring, the metabolism of the bacteria in the outer layers of the biofilm accelerates and they extract more nutrients from the effluent as it diffuses through the biofilm. The bacteria in the lower layers of the biofilm are therefore starved of nutrients and will die and detach from the support medium, taking the rest of the overlying biofilm with them. This process is therefore an inherent feature of all trickling filters and is essential to ensure the continuation of an active bacterial population in the biofilm.

The other characteristics required of a nitrifying trickling filter medium are that it must allow water and air to permeate freely through the filter. These characteristics are determined by the size, shape and packing geometry of the medium. Most media currently in use are randomly packed (i.e. the medium consists of many small pieces which are tipped into the filter at random) although some ordered media have been designed which employ larger selfsupporting blocks of various shapes to control the rate of drainage.

In addition to these criteria, the medium must have sufficient mechanical strength to withstand the weight of the layers of media above it in the filter. it is important, when calculating the required strength, to consider that the upper layers of media may become clogged with biomass or even completely flooded with effluent, thereby greatly increasing their weight. The medium should also be sufficiently durable to withstand many years of use without being adversely affected by freezing or by any substances likely to be found in sewage. The generally accepted test for durability is the standard Sodium Sulphite Soundness method (BS 1438,1971).

The four media used in this research are blast furnace slag, and three random-pack plastic media: Etapak 160 and 210, and Flocor RS. The blast furnace slag is 28 mm nominal grading, supplied by Summerfield and Lang from Santon. Etapak 160 and 210 are both random media made of polypropylene with specific surface areas of 150 and 200  $m^2.m^{-3}$  respectively and with a voidage of 96%. They were supplied by the manufacturers, ETA Process Plant Ltd. of Rugeley, Staffs. Flocor RS is another random medium consisting of short lengths of corrugated unplasticised PVC tubing. The medium has a specific surface area of  $240$  m.m.

### Experimental Methods

and a voidage of 97%. it was manufactured by BS Flocor Ltd and supplied by Entec Pollution Control Ltd. of Andover, Hants.

### Table 1: Characteristics of the Media.



Typical characteristics.

The Etapak 210 is contained in a pilot scale filter of depth 1.8 m and diameter 1.4 m. The other three media are in smaller pilot scale filters of depth 1.2 m and diameter 0.9 m. Effluent is applied to all four filters via single fixed downward-facing nozzles (Delavan BNM 46 for the three smaller filters and BNM 136 for the large filter).

The biomass accumulation within the filters was measured by using a neutron probe. The neutron probe measures the moisture volume fraction in a sphere of approximately 30 cm radius. To apply this to the accumulation of biomass it is necessary to assume that the moisture volume fraction of the filter media is proportional to the amount of biomass present. Although this technique was found by Wheatley (1976) to be unreliable in some cases, the technique has been used effectively by Bruce and Merkens (1970) and Gray and Learner (1984). The aluminium access tubes in each filter were positioned at a distance of at least 200 mm from the side wall to prevent interference. The tubes are kept plugged at the top with rubber bungs when not in use and the bottoms are left open for drainage. The measurements taken from the neutron probe are converted into moisture volume fractions by dividing by a blank reading obtained by immersing the probe in a large tank of water.

Each filter is fitted with thennistors to record temperatures using a data logger (Squirrel model 1202, Grant Instruments, Cambridge) and an aluminium access tube for use with a neutron probe (Institute of Hydrology Mark 11 L). Samples can be taken either at the bases of the filters or from two sampling ports placed at regular intervals down the filters. Samples were routinely analyzed for BOD, ammonia nitrogen, nitrate nitrogen, suspended solids and turbidity.

The pilot scale filters were designed to represent a cross-section of a full scale filter. The use

of pilot scale trickling filters utilising real waste water ensures that the results obtained are a true reflection of the full scale operation of trickling filters. It would be impossible to recreate in the laboratory the multitude of factors which affect real sewage treatment processes, such as the fluctuations in temperature and sewage composition and the effects of the weather and of grazing macrofauna. However, by including these factors in the experiments, there is inevitably an intrinsic degree of scatter in the results which must be compensated by taking a large number of samples, and examining general trends.

### Results

The filters have been operated in two modes so far: in the period from September 1990 to February 1991 the feed was primary settled sewage to determine whether the filters were capable of simultaneous BOD removal and nitrification. Since February 1991 the filters have been loaded at a higher rate with secondary effluent. Typical characteristics of these two feeds are given in Tables 2 & 3. It should be noted that the strength of the influent was highly variable, as is often found at small sewage works. In this case, the variation is not only diurnal and seasonal, but is also due to variations in the population at weekends and during university holidays.

Table 2: Analysis of the Influent to the Pilot Scale Filters during Phase 1. (1st September

### 1990 to 19th February 1991)



The better performance of the blast furnace slag can be attributed to two factors: longer retention time (typically 60 - 70 minutes compared to 10 - 25 minutes for the random plastic colonication of the filter by might media) and more extensive colonisation of the filter by micro-organisms as illustrated in the neutron probe moisture readings (Figures 1-4). However, it is unlikely that the performance of the blast furnace slag media could be maintained over long periods, as the biomass was accumulating rapidly in the media, particularly near the top of the filter, and it was anticipated that the filter would soon experience ponding problems (Figure 2). As expected, the plastic media filters showed no evidence of sufficient biomass build-up to cause concerns about ponding due to their higher voidage.

It was observed that large numbers of snails were washed out of the pilot filters, particularly during summer months. Snails are known to be voracious grazers of biofilm, and the siting of a nitrifying trickling filter after secondary filtration rather than activated sludge could result

Table 3: Analysis of the Influent to the Pilot Scale Filters during Phase 2. (February '91 to present)



### Phase 1: Primary Settled Sewage Feed.

During this phase of operation, primary settled sewage with a BOD usually greater than 100 mg.  $l^{-1}$  was applied to the filters at a loading rate of 1.5 m<sup>3</sup>.m<sup>-3</sup>.d<sup>-1</sup> in order to evaluate the BOD removal characteristics of the media. However, within five months it was apparent that the three random plastic media were not achieving effective BOD removal, whereas the blast furnace slag was removing up to 90% of the applied B.O.D. and 50% of the ammonia, despite having a media depth of only 1.2 m.
in snail eggs or larvae being transferred to the nitrifying filter. Therefore a nitrifying filter following high-rate filtration could be expected to exhibit more biomass-limitation than a filter following activated sludge.



# Figure 1: Moisture Volume Fractions for Etapak 210 Media

4%

Figure 2: Moisture Volume Fractions for Blast Furnace Slag



Figure 3: Moisture Volume Fractions for Etapak 160

Figure 4: Moisture Volume Fractions for Flocor RS

# Phase 2: Operation as a Tertiary Nitrifying Filter.

The feed was changed in February 1991 to secondary effluent with a considerably lower BOD but with sufficient ammonia nitrogen to support good nitrification (Table 3). This effluent is therefore typical of the unsettled effluent from a sewage works which is failing to meet Royal Commission Standards and is in need of a tertiary treatment stage.

Initially, the hydraulic load to the filters was varied over the range  $1 - 10 \text{ m}^3 \text{ m}^3 \text{ d}^{-1}$  in order

to determine the optimum loading for long-term research. It was found that the optimum hydraulic load was  $6 \text{ m}^3 \text{.m}^{-3}$ .d<sup>-1</sup>, since the nozzles provide good continuous distribution at this flow rate without producing a fine mist or excessive shear forces on the media surface. Published data from Burnley sewage works has shown that this loading rate gave optimal performance with their full scale nitrifying filter (Hill et al., 1992).

It is widely accepted that the BOD of waste water must be reduced to below about 20 mg. $1<sup>-1</sup>$ before effective nitrification will occur (Bovendeur et al., 1990; Hanaki et al., 1990). Therefore, a nitrifying trickling filter medium must also be capable of effectively removing residual BOD. Figures 5-8 show the BOD removal characteristics of the four media when operating as nitrifying filters (ie in Phase 2 of the project). All four media demonstrate pseudo first order kinetics, characterised by a straight line with an increased rate of BOD removal at higher loads. However the blast furnace slag medium demonstrates significantly better and more consistent BOD removal, resulting in average effluent BOD concentrations of only 12  $mg.l<sup>-1</sup>$  compared with 15 - 17 mg.  $l<sup>-1</sup>$  for the plastic media filters. The better performance ensures that the BOD will fall more rapidly below the critical limit for nitrification in the blast furnace slag filter, allowing nitrification to proceed earlier than in the random plastic media filters.

It should be noted that the straight lines drawn on these and later graphs are intended to show more clearly the general trends apparent in the data. The data, as presented here, are too scattered to obtain a meaningful "best-fit line".



Figure 5: BOD Load and Removal Correlation for Etapak 210.

Figure 6: BOD Load and Removal Correlation for Blast Furnace Slag.





# Figure 7: BOD Load and Removal Figure 8: BOD Load and Removal<br>Correlation for Etapak 160. Correlation for Flocor RS. Correlation for Etapak 160.

Figures 9-12 illustrate the performance of the four media in terms of ammonia removal. Despite the inevitable scatter inherent in pilot scale research, it can clearly be seen that the blast furnace slag media gives a higher and more consistent degree of nitrification than the random plastic media. At a hydraulic loading rate of 6  $m<sup>3</sup>$ .m<sup>-3</sup>.d<sup>-1</sup>, the yearly average was almost 50% ammonia removal, achieved within a filter depth of only 1.2 m. This compares with only 10-20% ammonia removal achieved by the three plastic media filters. On this basis, the volume required for a nitrifying filter employing random-pack plastic media would therefore be between 3 and 9 times the volume for an equivalent blast furnace slag filter.



-40' 0 20 40 60 80 100 120 140 160 180 200 220 240

Ammonia Load (g/m3. day)

Figure 9: Ammonia Load and Removal Correlation for Etapak 210.

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-40' 0 20 40 60 80 100 120 140 160 180 200

Ammonia Load (g/m3.day)

Figure 10: Ammonia Load and Removal Correlation for Blast Furnace Slag.







Figure 11: Ammonia Load and Removal Correlation for Etapak 160.

Figure 12: Ammonia Load and Removal Correlation for Flocor RS.

These results are comparable to those obtained in a pilot scale study before the construction of the full scale nitrifying trickling filter at Burnley sewage treatment works (Hill *et al.*, 1992). This work was carried out in a 2.3 m deep filter using coke as a medium and with hydraulic loadings between 6 and  $18 \text{ m}^3.\text{m}^{-3}.\text{d}^{-1}$ .

The consensus of opinion is that the kinetics of nitrification follow Monod kinetics (Sutton et al., 1981; Beg et al., 1983; Randall et al., 1984; Shammas, 1986).

$$
v = -\frac{\partial S}{\partial t} = V_{\text{max}} \frac{S}{K_g + S}
$$

Where:

 $v =$  nitrification rate (g.m<sup>-3</sup>.d<sup>-1</sup>)

- $S =$  substrate concentration (mg.l<sup>-1</sup> or g.m<sup>-3</sup>)
- $t = time (days)$
- $V_{max}$  = maximum nitrification rate (g.m<sup>-3</sup>.d<sup>-1</sup>)
	- $K_c$  = half-velocity coefficient (mg.l<sup>-1</sup> or g.m<sup>-3</sup>)

The value of the half-velocity constant,  $K_s$ , for nitrifying trickling filters is generally reported to be in the region of 2 mg.1<sup>-1</sup>. This implies that the nitrifying bacteria in the upper layers of a typical nitrifying trickling filter will be operating at near zero-order kinetics, where nitrification is independent of the substrate concentration. As the ammonia concentration drops down the depth of the filter, the half-saturation coefficient is approached and the rate of nitrification becomes progressively more dependent on the substrate concentration. The implication of this is that nitrification becomes gradually more prolonged as lower effluent ammonia concentrations are required, and the minimum practical effluent ammonia concentration is about  $0.5 \text{ mg.} l^1$ . The achievement of lower effluent ammonia concentrations

would require a disproportionately large filter volume.

One explanation for the poor performance of the random plastic media in relation to the blast furnace slag comes from an examination of the data on the temperature at the centres of the filters. The high voidages of plastic media permit higher rates of convective air flow through the filters, thereby increasing the effects of the ambient air temperature on the temperature within the filter. In contrast, the blast furnace slag has sufficient voidage when used as a nitrifying filter medium to ensure adequate aeration, but does not allow excessive air flow and thereby minimizes the heat loss through convection. This results in a more stable and generally higher temperature within the blast furnace slag. Figures 13-16 illustrate this by showing the temperatures at the centres of the pilot scale filters and the ambient air temperatures for a 48 hour period during November 1991.

sludge (Downing, 1968; Wild et al., 1971), river water (Knowles et al., 1965; Stratton et al., 1967) and pure culture (Buswell, 1954). In each of these cases, an approximate doubling of )<br>7) *:* the rate of nitrification was observed over the temperature range 10-20'C. It is proposed that this rate enhancement was not observed to the same extent in nitrifying trickling filters

### Figure 13: Heat Retention within the Etapak 210 filter. Figure 14: Heat Retention within the Blast Furnace Slag Filter.

If a filter is exposed to diurnal fluctuations in temperature, then the rates of BOD removal and nitrification will also vary, thereby changing the concentration profile of the filter and exposing the bacteria to continually changing conditions. Temperature fluctuations must therefore be minimized to ensure constant conditions for the development of a stable bacterial population.

Attempts were made to correlate the temperatures measured at the centres of the pilot scale filters to the rate of nitrification. However, the data were too scattered to determine any trends, probably due to the extreme diurnal fluctuations in temperature and waste water composition.

The effect of temperature on nitrification has been studied by other workers using activated









# Figure 15: Heat Retention within the Figure 16: Heat Retention within the Flocor<br>Etapak 160 Filter. RS Filter. Etapak 160 Filter.

because the open nature of trickling filters means that the temperature will not remain stable<br>for long periods, as is the case for activated sludge, river water and laboratory pure culture experiments. Therefore the temperatures measured in this research (measured hourly then averaged over the day) can not reflect the true variation in the temperatures experienced by the biofilm.

Perhaps the most fundamental difference with nitrifying trickling filters is that their depth (3-4 m) is significantly greater than the 1.8 m standard depth for BOD removal. This is primarily to increase the hydraulic retention time, but it will also improve the temperature stability of the filter. If random plastic media are used in the filter instead of blast furnace slag or similar mineral media, then the depth must be increased accordingly to maintain the required ammonia removal. In practice, this would mean constructing nitrifying filters of 10-15 m depth, which would incur high pumping costs. Such tall plastic media trickling filters are found in the United States, where power costs are cheap compared to the U.K..

The data from this pilot study have been used to design a full scale nitrifying filter at Cranfield sewage treatment works. The filter was commissioned in June 1992 and initial data is presented here. It is hoped to report further data on the performance of the filter in a future publication.

# Design Criteria for Cranfield's Nitrifying Trickling Filter.

A continuous and even wetting pattern is widely accepted as being preferable to the intermittent wetting commonly found on carbonaceous filters. This is easily achieved by the use of a fixed distribution system employing upwards-spraying nozzles. This system offers

Although several nitrifying trickling filters have been built simply by converting existing conventional trickling filters, there are several fundamental differences in the requirements which have been confirmed by this research and should be addressed in order to optimise the nitrification efficiency.



effective distribution of the waste water, enables a wide range of wetting rates to be used, and prevents the excessive colonisation of the filter by flies (chironomids and psychoda). On sites where appreciable diurnal variation in the waste water flow is experienced, it may be necessary to incorporate an automatic recycling system into the design to ensure that the filter is kept wetted during periods of low flow.

The hydraulic loading can be varied by the use of recycling, but the optimum nitrification performance has been found to be around 6  $m^3 \cdot m^{-3} \cdot d^{-1}$  (Hill *et al.*, 1992).

The ammonia loading depends upon the effluent quality required of the filter, but loads up to 0.1 kg  $NH_{4}$ -N.m<sup>-3</sup>.d<sup>-1</sup> appear to be the optimum for efficient nitrification. At a hydraulic loading of 6 m<sup>3</sup>.m<sup>-3</sup>.d<sup>-1</sup>, this corresponds to a concentration of 16.7 mg NH<sub>4</sub>-N.1<sup>-1</sup>.

The ventilation requirements for a nitrifying trickling filter are less than those for a carbonaceous filter because the specific activity of the filter is lower. It is essential that adequate ventilation is provided to ensure that parts of the filter do not become anoxic. However, excessive ventilation would lead to more rapid convection through the filter and consequently a greater heat loss during periods of cold ambient temperatures. The compromise usually reached is for a ventilation area of between I and 2% of the surface area of the filter instead of the  $5\%$  used for conventional filters (Hill *et al.*, 1992).

The use of upward-facing spray nozzles may lead to an unacceptable degree of spray drift at very high hydraulic loadings (greater than  $18 \text{ m}^3 \text{.m}^{-3} \text{.d}^{-1}$ ), but at lower loadings the spray has been found to be manageable by the use of windbreaks around the periphery of the filter.

The nitrifying trickling filter at Cranfield sewage treatment works was designed with a depth of 3 m, consisting mostly of 25 mm blast furnace slag but with 30 cm layers of 100 mm slag at the top and bottom. The volume of the filter is 150 m' and the current hydraulic loading rate is 3  $m^3.m^3.d^{-1}$ . At the time of writing, the ammonia load has not exceeded 35 g.m<sup>-3</sup>.d<sup>-1</sup>, although it is expected to reach 70 g.m<sup>-3</sup>.d<sup>-1</sup> during winter months and may reach 100 g.m<sup>-3</sup>.d<sup>-1</sup> if a planned development within the catchment area goes ahead.

Within three weeks of commissioning the filter, the ammonia removal had reached 90% with effluent ammonia concentrations below 1 mg.  $l^{-1}$  (Figure 17). The nitrification performance has

The media employed for nitrification are generally smaller than those used for BOD removal. Typically, nitrifying filters contain media down to 25 mm grading to maximise the specific surface area and the retention time. Some filters have larger grade media on the top 50 cm to prevent clogging due to excessive biomass growth at the top of the filter (where some BOD removal may occur). Larger grade media may also be used at the base of filters to ensure that air can circulate freely and to ensure good drainage.

Random plastic media, such as those used in this study, do not achieve the same specific nitrification rates as mineral media. This can, as previously mentioned, be compensated for to an extent by an increased depth of filter but this will lead to increased pumping costs and the filter may suffer from excessive heat loss leading to freezing problems during prolonged cold spells.

# Initial Data from Cranfield's Nitrifying Trickling Filter

 $\cdots \cdots \cdots \cdots$ 

remained linear with the applied load at influent concentrations between 2.5 and 15 mg.  $1<sup>-1</sup>$ (Figure 18), and it is anticipated that this relationship will be maintained up to the maximum design load of the filter.

Further data on the performance of the filter, together with details of the construction and operating costs for the filter will be reported in a future publication.



## Conclusions

Although random plastic media are capable of nitrification, the lower specific nitrification capacity infers that a proportionally larger filter is required to achieve the same degree of <u>i</u> treatment as a mineral medium filter. There are three main features of mineral media which<br>client them the experience it is faction as of expression give them the superior nitrification performance:

- -longer retention time
- -improved biomass development
- -better heat retention

The use of mineral media, coupled with the improvements over the design of conventional trickling filters, illustrates that nitrifying trickling filters can provide a cheap, efficient and, most importantly, a reliable method of adding nitrification capacity to sewage treatment works. They are particularly suited to small treatment works, where nitrification but not denitrification is required, and where the low maintenance and reliable operation are especially advantageous.

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