Mechanisms of penetration in cartridge filtration of de-ionised water

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MECHANISMS OF PENETRATION IN CARTRIDGE FILTRATION OF DE-IONISED WATER

by

JAMES MICHAEL BENTLEY

A Doctoral Thesis:

Submitted in partial fulfillment of the requirements for the award of the Doctor of Philosophy Degree of the Loughborough University of Technology

December 1991

c James Michael Bentley, 1991
ABSTRACT

A programme of research has been carried out into the performance of cartridge filters rated from 0.1 to 0.45 micron in response to steady and varying particle concentrations and water flows.

It was discovered that pulsing the water flow to these filters often resulted in release of particles which had been previously captured by the filter. For membrane filters the release of particles was instantaneous. However, for one filter with more depth, a considerable time was required for most of the particles released to be detected. This filter was described as a prefilter rather than a membrane filter.

Under conditions of steady state water flows it was discovered that even when sieving is the dominant mechanism of filtration the behaviour of a filter varies with time, particle size and concentration and filter loading.

For the prefilter three penetration processes were operating in parallel. One of these was direct penetration, with no time delay between upstream and downstream concentrations. The other two processes both resulted in a time delay between upstream and downstream concentrations, the penetrating particles experiencing a distribution of residence times within the filter. Each of these processes could be represented by a first order dynamic system, the time constant for one being minutes, the other being hours.

The degree of penetration through each of the processes mentioned above is not constant and under certain conditions a shift from delayed to direct penetration occurs, resulting in an increase in the total penetration.

The membrane filters tested also showed evidence of a time delay between upstream and downstream concentrations, similar to the short term delay experienced by the prefilter. However, there was no long term delay for the membrane filters.

A simple dynamic model for the performance of the prefilter is presented. The changes in the performance of the prefilter with time, particle loading and particle concentration are explained in terms of the structure of the filter medium and the particle size. In addition, the differences in performance between the prefilter and the membrane filters are explained by the differences in the media.
ACKNOWLEDGEMENTS

I would like to thank my supervisor, John Lloyd for his help and guidance over the last four years.

I would also like to thank Dr JIT Stenhouse, Mrs JM Whitehead and Mr I Sinclair, who have helped in different ways to make this thesis possible, and the sponsors of CERCCON for financing much of this work.

Finally, I would like to thank the following for their help, support and encouragement:

Mr M Davy, Mr N Wenden, Mr T Lilley, Mr P Sands, Mr M Allen, Mr A Hartland, Miss S Dyson, Mr S Cartright, Dr AS Ward.
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<th>Description</th>
<th>SI unit</th>
<th>Practical unit</th>
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<tr>
<td>A</td>
<td>Magnitude of step change in system input</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>(A_n)</td>
<td>Amount of step increase in concentration attempting to penetrate the filter by the (n)th penetration process</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>C</td>
<td>Slip correction</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>(d)</td>
<td>Diameter of sample tubing</td>
<td>m</td>
<td>mm</td>
</tr>
<tr>
<td>(d_f)</td>
<td>Filter thickness</td>
<td>m</td>
<td>(\mu m)</td>
</tr>
<tr>
<td>(d_p)</td>
<td>Particle size</td>
<td>m</td>
<td>(\mu m)</td>
</tr>
<tr>
<td>D</td>
<td>Downstream particle concentration</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>(D_n)</td>
<td>Downstream concentration due to the (n)th penetration process</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>(D')</td>
<td>Downstream concentration modified to account for upstream concentration</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>(F_D)</td>
<td>Stoke’s drag force</td>
<td>N</td>
<td></td>
</tr>
<tr>
<td>I</td>
<td>Increase in upstream concentration in the event of an impulse</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>(I_n)</td>
<td>The amount of an impulse which attempts to penetrate the filter by the (n)th penetration process</td>
<td>Number per m(^3)</td>
<td>Number per 100 ml</td>
</tr>
<tr>
<td>(k_D)</td>
<td>Rate constant for intrapore diffusive deposition</td>
<td>s(^{-1})</td>
<td></td>
</tr>
<tr>
<td>(k_s)</td>
<td>Rate constant for surface blocking</td>
<td>s(^{-1})</td>
<td></td>
</tr>
<tr>
<td>(l)</td>
<td>Distance from downstream side of test filter to start of sample tubing</td>
<td>m</td>
<td>mm</td>
</tr>
<tr>
<td>L</td>
<td>Length of sample tubing</td>
<td>m</td>
<td>mm</td>
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<tr>
<td>LRV</td>
<td>Log retention value</td>
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<td>-</td>
</tr>
<tr>
<td>(p(t))</td>
<td>Penetration at time (t)</td>
<td>%</td>
<td></td>
</tr>
<tr>
<td>M</td>
<td>Number of unblocked pores per unit area</td>
<td>Number per m(^2)</td>
<td></td>
</tr>
<tr>
<td>(n)</td>
<td>Particle concentration</td>
<td>Number per m(^3)</td>
<td></td>
</tr>
<tr>
<td>(p_s(t))</td>
<td>Steady state penetration</td>
<td>%</td>
<td></td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
<td>Unit</td>
<td></td>
</tr>
<tr>
<td>--------</td>
<td>-----------------------------------------------------------------------------</td>
<td>----------</td>
<td></td>
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<tr>
<td>$p_{st}(t)$</td>
<td>Steady state penetration due to the nth penetration process</td>
<td>%</td>
<td></td>
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<tr>
<td>P(t)</td>
<td>Deviation from steady state penetration at time t</td>
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<td></td>
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<tr>
<td>q</td>
<td>Sample flowrate</td>
<td>m$^3$/s</td>
<td></td>
</tr>
<tr>
<td>Q</td>
<td>Main test rig flowrate</td>
<td>m$^3$/s</td>
<td></td>
</tr>
<tr>
<td>R</td>
<td>Flow redistribution parameter decrement parameter</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>t</td>
<td>Time</td>
<td>s</td>
<td></td>
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<td>$t_0$</td>
<td>Time before which no particles are counted following a pulse</td>
<td>s</td>
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<td>$t_p$</td>
<td>Time for peak in particle counts to be reached following a pulse</td>
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<td></td>
</tr>
<tr>
<td>$t_R$</td>
<td>Residence time</td>
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<td></td>
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<td>T</td>
<td>Ratio of time, $t$ to time constant, $\tau$</td>
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<tr>
<td>$\nu$</td>
<td>Fluid velocity</td>
<td>m/s</td>
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<tr>
<td>$\bar{\nu}$</td>
<td>Mean fluid velocity</td>
<td>m/s</td>
<td></td>
</tr>
<tr>
<td>$v_{\text{max}}$</td>
<td>Maximum fluid velocity</td>
<td>m/s</td>
<td></td>
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<tr>
<td>Y(t)</td>
<td>Deviation from steady state of system output at time, $t$</td>
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<td></td>
</tr>
<tr>
<td>$\alpha_0$</td>
<td>Initial bed porosity</td>
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<td></td>
</tr>
<tr>
<td>$\beta$</td>
<td>Flow redistribution parameter</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>$\beta_1$</td>
<td>Initial flow redistribution parameter</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>$\gamma$</td>
<td>Number of pore volumes of fluid passed through filter media</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>$\delta(t)$</td>
<td>Unit impulse function</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>$\eta$</td>
<td>Particle removal efficiency</td>
<td>%</td>
<td></td>
</tr>
<tr>
<td>$\lambda_{sr}$</td>
<td>Initial reduced dimensionless filter coefficient</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>$\mu$</td>
<td>Fluid viscosity</td>
<td>Ns/m$^2$</td>
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<tr>
<td>$\tau$</td>
<td>Time constant</td>
<td>s</td>
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<tr>
<td>$\tau_D$</td>
<td>Time constant for decrease in concentration</td>
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<tr>
<td>$\tau_I$</td>
<td>Time constant for increase in concentration</td>
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<td>$\tau_n$</td>
<td>Time constant for nth penetration process</td>
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CHAPTER 1

INTRODUCTION
In 1985 the Centre for Engineering Research into Contamination Control (CERCCON) was founded at Loughborough University of Technology. The reason for its existence was that the U.K. semiconductor manufacturers were encountering particulate contamination problems which were limiting the advance of their technology. A programme of research was started at Loughborough, financed by semiconductor manufacturers and their suppliers.

One of the areas of work considered by CERCCON was the development of test procedures for the evaluation of cartridge filters for the production and maintenance of low particulate, ultra pure water. The major difficulty of this work was that by the mid 1980’s 0.1 micron rated filters were already in use but the most sensitive on-line optical particle counters available had detection limits of 0.3 micron. Attempts to use alternative detection methods with lower particle sizes were considered, but these were not successful as they involved sampling, when what was really needed was continuous monitoring of particle concentrations.

It was not surprising that the 0.1 and 0.2 micron rated membrane filters tested proved to be highly efficient at particle sizes above 0.3 micron, with very few particles penetrating the filters. More significant particle counts downstream of the test filters were observed when the water flow was pulsed, leading to the suggestion that in some cases particles which had been captured by a filter were released from the filter when the flow was pulsed.

Having tested the available 0.1 and 0.2 micron rated membrane filters it was decided that filters of larger particle rating be tested so that the 0.3 micron particle counter could be used to determine the effect of particle size under steady and pulsed water flow conditions. All the filters released significant numbers of particles when the flow was pulsed, even at particle sizes considerably larger than their rating.

The studies performed under steady water flow conditions enabled the transient nature of filtration at these sizes to be considered. It was found that particles have a residence time within the filter, the length of this residence time depending on the particle size, the filter pore size distribution and the depth of the filter medium.
The background to this research is outlined in Chapter 2, which is a literature review of cartridge filtration. It is shown in this chapter that there is a lack of knowledge concerning mechanisms in this type of filtration and in particular, in the transient nature of cartridge filtration. The experimental procedures and the programme of research are presented in Chapter 3 with the results in Chapters 4 to 6. Chapter 4 is concerned with the results of testing cartridge filters under pulsed flow conditions. The results of the steady state water flow tests are presented in Chapters 5 and 6. Chapter 5 contains the results for the filter which showed the most extreme transient behaviour, the 0.45 micron rated prefilter. The 0.4 and 0.45 micron membrane filter results are presented in Chapter 6. In Chapter 7 the results are analysed, leading to the mechanisms discussed in Chapter 8. Finally, in Chapter 9 the conclusions of the work are summarised and proposals for future work presented.
CHAPTER 2

CARTRIDGE FILTRATION - A LITERATURE REVIEW

2.1 Requirement for high purity water

2.2 Introduction to cartridge filtration

2.3 Methods of manufacture and rating of cartridge filters

2.4 Mechanisms of cartridge filtration

2.5 Conclusions
2.1 THE REQUIREMENT FOR HIGH PURITY WATER

There are many industries which require ultra pure process water. One industry for which the requirement is particularly high is the semiconductor industry. It has been stated that one of the principal sources of contamination in integrated circuit fabrication is high purity process water.\textsuperscript{12} Contamination in this context is not limited to that which is particulate, although particulate contamination is known to be a major problem for the manufacturer.\textsuperscript{3,4,5} The "\textsuperscript{1}{10}th rule" is commonly used, that is that defects of one tenth of the device minimum feature size are critical.\textsuperscript{3} By the mid 1980's, minimum feature sizes of 1 micron were achievable so that particles as small as 0.1 micron were regarded as critical to the manufacturing process. By the mid-1990's minimum feature sizes are predicted to be down to 0.25 micron.\textsuperscript{3}

Attempts have been made to determine the effect on semiconductor manufacturing yield of contamination, particulate or otherwise.\textsuperscript{6,7,8} It is not the purpose of this study to consider these models, but their existence does indicate the view of the semiconductor industry on particulate contamination.

Requirements for cleanliness levels of pure water have been issued\textsuperscript{9} and these are constantly being updated, with smaller particles at lower concentrations being regarded as critical. It is from this background that the work on filtration by the Centre for Engineering Research into Contamination Control commenced.
2.2 INTRODUCTION TO CARTRIDGE FILTRATION

Cartridge filters are often thought of as being membrane filters. However, although there are many membrane cartridge filters on the market, there are also other types of filter available in cartridge form.

The role of cartridge filtration is usually that of polishing, that is, removal of traces of particulates or bacteria, rather than the bulk filtration of visibly contaminated liquids. However, it must be remembered that what is regarded as pure water in some applications may be regarded as highly contaminated in others.

There is some confusion regarding the types of cartridge filter available because filter manufacturers do not all use the same terminology. It is therefore necessary to consider filter types in terms of the structure of their media and the mechanisms of filtration for which they are designed to operate, rather than the manufacturer's description.

The simplest filter type to consider is the screen filter. Ideally a screen filter would consist of a geometrically regular porous matrix. Particles are retained on the surface of the filter by sieving, mechanical-straining or screening. Particles larger than the pore size are retained, particles smaller than the pore size emerge in the filtrate. Because the particles are retained on the upstream surface of the filter this regime of filtration is referred to as surface filtration. This description does not indicate the mechanism of filtration but the desired location of the particles after filtration.

Similarly, the term depth filtration does not indicate the mechanism of filtration in operation; it simply implies that particles are captured within the depth, or thickness, of the filter medium.

Membrane filters are often regarded as screen filters because they are primarily used for removing particles larger than their pore size so that particles are captured on the upstream surface by sieving. However, membrane filters can also act as depth filters, relying on adsorptive and electrostatic properties to remove particles smaller than the pore size. A depth filter should therefore be thought of not as one which has considerable thickness but as one which captures particles within the depth of the filter.
medium. It follows that for certain particle sizes a membrane filter will act as a surface filter, the dominant mechanism being sieving, but that at different particle sizes the same filter may act as a depth filter.

The above discussion has not considered the effect of the distribution of pore sizes in the filter. It is often assumed that the pores in a membrane filter are all of equal diameter; however, in many cases this is not so. Filters are rated according to their pore size, two types of rating being used, namely, absolute and nominal.18,19 Absolute ratings imply that the filter removes all particles larger than the rated pore size. Nominal ratings are less well defined, but they indicate the size of particle which can be considered to be removed with high efficiency, typically 95%.

Because membrane filters are primarily used for surface filtration of particles larger than the pore size and because the pore sizes for membranes are regarded as being uniform, they are assigned absolute ratings.20 Filters which are designed to operate by depth mechanisms are normally assigned nominal ratings because the mechanisms of filtration are not absolute and the pore size distribution within the depth of the filter is less easily measured. However, there are claims that types of depth filter can be absolute and therefore should be rated accordingly.21
2.3 METHODS OF MANUFACTURE AND PARTICLE RATING OF CARTRIDGE FILTERS

There are many different types of cartridge filter available ranging from membranes of less than 10 micron thickness to filters designed to operate as depth filters with thicknesses of up to 25 mm. In ultrapure water systems it is at the point of use where filtration is most critical as this is the final filtration stage. Such filters are usually membranes as these are designed to be absolute filters for particles larger than the filter rating.

A thorough review of the development, rating and manufacture of membrane filters has been compiled by Porter. Although there are many types of membrane in use they can be conveniently thought of as belonging to one of two types - the "track-etch" Nuclepore type, which is a thin membrane with uniformly sized cylindrical pores and low porosity, produced by exposing a thin film to a beam of fission fragments producing tracks through the thickness of the film. Pores are subsequently produced by etching. The other type of membrane includes those made by other processes such as stretching or phase-inversion, which are thicker with much higher porosity and have the appearance of a "felt" rather than a sheet containing cylindrical pores.

An alternative method of classification of membrane filters is into charged and uncharged filters. In some cases the membrane surface is modified to give it a positive charge. This is designed to improve the depth filtration characteristics of the filter by electrostatic attraction of negatively charged particles in the water.

Because the efficiency of filtration at the point of use is so critical in maintenance of ultra pure water there has been much discussion in recent years regarding the method of rating membrane filters. Absolute rating of membrane filters was successfully carried out for pharmaceutical applications. Membrane filters were rated according to their ability to retain bacteria. Filters which produced sterile effluent when challenged with \(10^7\) Pseudomonas diminuta per square centimetre of filter surface area under standard conditions were considered
absolute rated sterilizing filters. These filters were accepted as 0.2 micron absolute rated filters, although it is known that the *P. diminuta* organism is larger than 0.2 micron. Although this method of rating filters is ideal for determining whether a filter is absolute with respect to removal of the *P. diminuta* organism, it is clearly unacceptable as a method of rating a filter as absolute for 0.2 micron particles.

In order to determine by a non-destructive test the retention level of every filter manufactured, the bubble point integrity test is used. The pressure required to overcome the capillary forces in the pores of a fully wetted membrane is inversely proportional to the pore diameter. When pressure is applied to the pores of a wetted membrane the liquid from the largest pores will evacuate first. The pressure at which the capillary forces are first overcome is the bubble point value and this indicates the size of the largest pore.

If the bubble point value is known for a membrane which is known to be absolute for *P. diminuta*, other membranes with the same bubble point value may be assigned the same rating. However, when such a membrane is assigned a rating of 0.2 micron and, even worse, when a membrane with a bubble point value twice that of the membrane absolute for *P. diminuta* is assigned an absolute rating of 0.1 micron, it is not possible to say that the absolute rating is equal to the largest diameter pore.

The above questioning of the rating system is justified by filtration studies which have shown that particles considerably larger than the filter rating are able to penetrate membrane filters under certain conditions. It is therefore not possible to predict the performance of a membrane filter, or even the size of the largest particle which will be able to penetrate the filter, on the basis of the rating assigned to it. This is clearly not a satisfactory situation when membrane filters are used at the point of use in ultra pure water systems.
2.4 MECHANISMS OF CARTRIDGE FILTRATION

With the high demand for low particulate water a large amount of work has been carried out in recent years, much of which has been concerned with evaluation of filters rather than mechanisms of filtration. One of the major problems in work of this type is the detection and counting of particles. Until 1989 the detection limit of optical particle counting was 0.3 micron, with membrane filters rated at 0.1 micron being widely used in the semiconductor industry. Since 1989 several optical particle counters have become available with detection limits below 0.1 micron.

Evaluation of 0.1 micron filters by methods other than optical particle counting was considered by the Integrated Circuit Manufacturers' Consortium Filter Evaluation Working Party. Colloidal silica was the test material used to challenge the filters, the efficiency of filtration being determined by measurement of the concentration of silica upstream and downstream of the filter. The concentration was determined by electrothermal atomic absorption.

One of the main disadvantages of this method of testing is that the size distribution of colloidal silica is wide but the detection method is independent of particle size. Furthermore, there is some doubt over the long-term stability of the colloidal silica and it is possible that some of the silica detected is due to dissolved silica rather than particles.

The results of this work showed that the efficiency was dependent on the loading of the filter, that is, on the total solids retained on the filter. Figure 2.1 shows a typical curve for a 0.1 micron membrane filter challenged with colloidal silica with a mean size of 0.05 to 0.1 micron.

After a certain degree of loading breakthrough occurs and the efficiency may fall rapidly from over 95% to less than 50%. Not all the membranes tested showed the same behaviour (see Figure 2.2). In some cases the efficiency was maintained at a high level throughout the test (1 and 2 in Figure 2.2) and in others the efficiency was low at all times (4 in Figure 2.2). It is claimed by some that it is the charged filters which operate
at a constant high efficiency with the uncharged filters showing breakthrough after a certain loading, although it is claimed by others that the reverse is more likely to be true. The effect of charge is a matter of some debate at present.

Attempts have been made to derive a single value for the efficiency or "performance" of a filter in such a situation by calculating the degree of particle loading before breakthrough occurs. However, the time dependence of efficiency and the different
type of response of different types of membranes makes this a difficult task. What the filter user requires in this situation is a model which will enable him to predict when breakthrough will occur.

Similar breakthrough is known to occur with colloidal particles in packed-bed filtration. Adin and Rajagopalan\textsuperscript{48} have developed models to predict the breakthrough curves for packed-bed filtration by comparing it with adsorption/ion-exchange. The nature of these processes can be explained by adsorption/desorption or attachment-detachment models.

Saatci and Sen\textsuperscript{49} have also studied attachment and detachment in fixed beds based on the assumption that there are a limited number of capture sites within a bed, taking into account the effect of occupied sites on the probability of a single particle to attach itself to one of the remaining sites.

A number of other studies of attachment and removal mechanisms have also been carried out.\textsuperscript{52,53,54} Houi and Lenormand\textsuperscript{52} have shown that for particles down to 2 micron frequent movement on the collecting surface occurs and that contact between suspended particles and part of the filter does not necessarily lead to permanent trapping of the particles.

A review of studies aimed at predicting the time-dependent behaviour of deep bed filtration is given by Tien.\textsuperscript{50} It appears that there are definite similarities in the transient nature of deep bed filtration and depth filtration with membranes which may give an insight into the operation of membrane filters.

Although there are problems with the colloidal silica method of testing filters, this work has led to some important findings. Firstly, the efficiency of a membrane filter is not necessarily constant for a given particle size but may vary with particle loading of the filter. Also, when challenged with particles significantly smaller than the rated pore size of the filter, depth filtration may occur, adsorption/desorption processes being in operation.

The fact that the performance of a filter is time-dependent is important because an understanding of the time dependence can lead to further understanding of the
mechanisms of filtration. It can also be important in deciding on the suitability of a filter for a given application to know how the performance of the filter will vary with time. A definition of efficiency which accounts for time-dependence is therefore needed.\textsuperscript{51}

The change in membrane filter efficiency with time (or loading) has been considered by Grant\textsuperscript{55} in conditions where sieving is the only mechanism operating. Grant found that at zero loading membranes challenged with particles of equal size to the filter rating gave log reduction values (LRV's) of 2 to 4, that is, they removed 99% to 99.99% of the challenging particles.

Note, the log reduction value is given by equation (2.1).

\[
LRV = \log_{10}(U/D) \tag{2.1}
\]

where \( U \) = upstream concentration and \( D \) = downstream concentration.

However, as the loading of the filter increased the efficiency was reduced owing to redistribution of the water flow through larger pores once the smaller pores had blocked. This reduction in efficiency with loading was found to be the case for 0.1, 0.2 and 0.45 micron rated filters challenged with a range of particle sizes. The following model was derived:

\[
\frac{U}{D} = \frac{(1 - \exp(\lambda_{st} - \exp(\beta\lambda_{st}U(\gamma - 1)))\{\exp(\beta\lambda_{st}U\gamma) - 1\})}{(1 - \exp(\beta\lambda_{st}U(\gamma - 1)))\exp(\beta\lambda_{st}U\gamma)} \tag{2.2}
\]

where \( \lambda_{st} \) is the initial reduced dimensionless filter coefficient \( \beta \) is the flow redistribution parameter \( \gamma \) is the number of pore volumes of fluid passed through the filter media.

The initial reduced dimensionless filter coefficient and the flow redistribution parameter are functions of particle size, filter porosity and pore size distribution.

The flow redistribution parameter also depends on filter loading and is given by equation (2.3).
where $\beta_i$ is the initial flow redistribution parameter $R$ is the flow redistribution parameter decrement parameter.

The number of pore volumes of liquid passed through the filter medium is a measure of the proportion of filter pore volume which is clogged with particles. This is calculated from equation (2.4)

$$\gamma = \frac{V_f}{\alpha_0 d_f}$$

where $d_f$ is the filter thickness, $V$ is the fluid superficial velocity and $\alpha_0$ is the initial bed porosity.

This work shows that the efficiency of microporous membranes is dependent on loading and therefore varies with time. The effect of the pore size distribution is considerable, not only because particles considerably larger than the rated pore size are able to penetrate, but also because for a wide range of particle sizes there is a significant fall in efficiency as the filter becomes increasingly loaded, this fall in efficiency being due to the redistribution of the flow through larger, unblocked pores. For example, a 0.45 micron rated filter was challenged with 0.442 micron particles. The initial LRV was 2 (efficiency 99%) but this fell to considerably less than 1 as the loading increased. The same filter type challenged with 0.605 micron particles had an initial LRV of 6 (efficiency 99.9999%), which fell below 2 (efficiency 99%) as the loading was increased.

The above model for sieving in microporous membranes does not account for any subsequent removal of particles from the filter. Because the mechanism of filtration is sieving it is assumed that particles captured by the filter are retained. However, membrane filters will capture particles by mechanisms other than sieving and in such cases it is possible that release of these particles may occur.
In their work on mechanisms of filtration with membranes, Grant, Liu and Rubow consider the various means by which particles may be captured by and released from membrane filters.

Six mechanisms of particle capture are described, five of which are shown in Figure 2.3. It is assumed that capture occurs when a particle is brought into contact with the filter medium.

*Gravitational settling* occurs when particles leave the fluid streamline because of their large mass and low velocity.

*Electrostatic deposition* occurs when attractive forces exist between charged particles and an oppositely charged filter medium.

*Impaction* occurs when particles leave the fluid streamline as it is diverted around the filter structure. This is most likely to occur for particles of large density and size moving at high velocity.

*Interception* occurs when the fluid streamline brings the particle into contact with the filter medium. This occurs when the minimum distance between the fluid streamline and the filter medium is less than the particle radius.

*Diffusive capture* is due to the Brownian motion of very small particles, the random motion increasing the probability of the particle being brought into contact with the filter medium.

*Sieving* is the sixth mechanism. This occurs when particles are physically too large to pass through the openings of the filter medium.

It is known in gas filtration that the most important mechanisms for sub-micron particles are interception and diffusion. Much theoretical and experimental work has been done in this field. By considering the physical chemistry of solid-liquid interfaces Grant et al show the conditions under which the mechanisms of gas filtration described above operate in liquid filtration. Under favorable chemical conditions the high efficiencies at low particle sizes which can be achieved in gas filtration should also be achievable in liquids. However, when double layer repulsion is significant sieving becomes the only effective removal...
mechanism, the efficiency of removal of particles smaller than the filter pore size being low. Accounts of the effects of the surface forces described above are given by Cintre and Wakeman.

Because of its high resistivity, 18 megohm-cm de-ionised water is one liquid which will result in high double layer repulsion when the particles and filter medium have the same charge. It would therefore be expected that the removal efficiency of particles smaller than the filter pore size will be low.

Although the above discussion makes theoretical sense it does not explain the removal of colloidal silica by 0.1 micron rated membrane filters which is known to be initially high for charged and uncharged filters with particles considerably smaller than the pore size. There is currently some debate over the performance of membranes in the colloidal silica test, particularly regarding the question of the effect of charge.

Having considered the capture mechanisms of filters the removal of particles from filters is now discussed. Consider a particle considerably smaller than the filter pore size which has captured by the filter (see Figure 2.3). The force of adhesion between the particle and the part of the filter to which it is attached must be overcome for the particle to be released.

The force of attraction increases as the size of the particle decreases. The components of this force are discussed in detail by Grant et al. On the other hand, the drag force on the particle exerted by the fluid flow increases with particle size, fluid velocity and viscosity (see equation 2.4). Because the viscosity of liquids are generally considerably higher than gases the drag force on a particle of a given size will be higher for a liquid than for a gas at equal velocities.
Capture of particles smaller than the pore size

\[ F_D = \frac{3\pi \mu d_p \nu}{C} \]  

(2.4)

where \( F_D \) is the Stokes drag force, \( \mu \) is fluid viscosity, \( d_p \) is particle size, \( \nu \) is fluid velocity and \( C \) is a correction factor.

In fact, the particle does not experience all of this drag force because of the presence of a boundary layer on the surface of the filter medium which has the effect of shielding the particle from the full force of the fluid moving past it. In general the thickness of the boundary layer is proportional to the square root of the fluid kinematic viscosity which is generally less for a liquid than for a gas, so that under similar conditions a gas will produce a boundary layer of greater thickness than a liquid. It follows, therefore, that the drag force felt by a particle of a given size on a surface will be higher in a liquid than in a gas because the available drag force is higher and the shielding provided by the boundary layer is lower.

Having considered the application of a removal force by high velocity liquid flows, let us consider the increased effect of pulsing liquid flows. It is possible that a pulse in the liquid flow could cause a momentary opening of pores which, together with the high
liquid velocity, results in particle release. There has been a reasonable amount of work done in this area, although most of it has been more concerned with the cleanliness of the filter cartridge itself than with the removal of previously captured particles.

There has been some work done in gas systems with pulsing flows and the indications are that in some cases particles have been removed from filters in the event of a pulse. If removal of particles from membrane filters is possible in gases it is certainly likely to be achievable in liquid streams, given that the viscosity and therefore the available drag force is considerably higher.

Consider a particle which has been captured by sieving, its size being slightly greater than the rating of the filter. If the introduction of a pulse in the liquid flow causes sufficient flexing of the filter medium, it is possible that the pore may open momentarily and that the particle may be released. Whether or not particles may be released by such a process and whether they are subsequently re-captured in the depth of the filter medium or manage to penetrate the filter, is clearly dependent on the particle size, filter pore size distribution, filter thickness, force of attraction between particle and filter, liquid velocity and viscosity and the nature of the pulse in the liquid flow.

Nonaka's work on kinetics of membrane microfiltration considers three mechanisms of filtration: intrapore diffusive deposition, surface blocking and thin cake formation. For particles smaller than the pore size it is suggested that intrapore diffusive deposition dominates. By disregarding surface deposition and axial diffusion of particles, filtration is modelled by accounting for radial diffusion of particles and the flow velocity in a pore. It is assumed that the intrapore diffusion process is first order, that is,

\[- \frac{dn}{dt} = k_0 n \quad (2.5)\]

where \( n \) is the concentration of particles and \( k_0 \) is the rate constant for diffusive deposition.

For particles larger than the pores the process is surface blocking, which is again considered to be a first order process:
\[ \frac{dM}{dt} = k_b M \] (26)

where \( M \) is the number of open pores per unit area and \( k_b \) is the rate constant for surface blocking. It has been shown that these first order models describe the behaviour of the filter well, that is, that before cake formation occurs, whether the particles are smaller or larger than the pore size first order processes operate.

The work which has been reviewed in this chapter shows that filter efficiency may vary with time. It seems that in cartridge filtration the level of knowledge regarding the transient nature of filtration is limited and there is a need for further research in this field.
This literature review is limited by the lack of theoretical publications relating to the mechanisms of cartridge filtration. Much of the work which is published in this field is in relation to filter evaluation rather than mechanisms of filtration.

From the work which has been reported it is clear that cartridge filtration of sub-micron particles is a transient process whether surface or depth filtration dominate. With particles smaller than the pore size there are similarities in the transient nature of filtration between membrane filtration and deep bed filtration. When sieving is the dominant mechanism, that is for particles larger than the filter pore size, the efficiency of filtration decreases as the loading of the filter increases. The reason for this is that even for membrane filters there is a distribution of pore sizes, the smaller pores blocking before the larger ones so that the flow is redistributed through the larger pores as the small ones block.

Under certain conditions it is possible that captured particles may be removed from the filter adding further to the variation in filter performance with time.

It became clear that the understanding of the transient nature of filtration was limited, particularly cartridge filtration and the capture mechanisms which operate. It was therefore decided that a programme of experimental work be undertaken to consider the long term performance of cartridge filters under steady and pulsed flow conditions with a range of challenge particle sizes and concentrations. The aim was not only to understand the transient nature of cartridge filtration but also to determine mechanisms of capture and penetration of particles.
CHAPTER 3

DEVELOPMENT OF FILTER TEST RIG AND EXPERIMENTAL PROCEDURES

3.1 Introduction

3.2 Development of experimental rig

3.3 Test procedures

3.4 Particle counting in steady and pulsed water flow conditions

3.5 Summary of experimental programme
3.1 INTRODUCTION

It was decided that there was a need for evaluation of cartridge filters in de-ionised water which would enable the transient nature of filtration to be determined. As well as the capture of particles, subsequent removal of particles from the filter was to be considered.

The most critical filtration step in an ultrapure water system is the point of use, so it was decided that the first phase of testing should be concerned with point of use membrane filters. A test rig and procedures were developed to determine the performance of these filters under steady and pulsing water flows.

The limitation of the work was the sensitivity of the available optical particle counters, which until 1989 was 0.3 micron. As the membrane filters of interest were 0.1 and 0.2 micron rated very few particles were counted downstream of the test filters under steady state water flows. However, when the flow was pulsed more interesting results were recorded. In some cases filters which were more than 99.99% efficient at the test particle size under steady state conditions released significant numbers of particles when the flow was pulsed. This discovery led to the decision to consider filters of larger rating under steady and pulsed flow conditions and the effect of particle size and challenge concentration. Filters rated at 0.4 and 0.45 micron were tested over a range of particle sizes using the 0.3 micron optical particle counter.

Two membrane filters and one prefilter were tested. The prefilter was selected because it was designed to behave in a manner intermediate between that of a membrane filter (designed for sieving retention) and a filter designed to operate by depth filtration. It was expected that comparison of this filter's behaviour with that of the membrane filters would not only identify the differences in performance between membranes and depth filters, but also that it would give further insight into the mechanisms of filtration with membranes.
3.2 DEVELOPMENT OF EXPERIMENTAL RIG

There are several possible ways in which the particle removal efficiency of a cartridge filter can be measured. The type of process used must be determined by the requirements of the experimental programme. In this case the requirements were to test filters with a range of ratings down to 0.1 micron in order to determine the variation in efficiency with time, particle loading, challenge particle size and concentration and the effect of pulsing water flows. In order to achieve these goals continuous monitoring of particle counts either side of the test filter is essential, together with a controllable rate of particle challenge.

A recirculating test rig was assembled in PVDF (polyvinylidene fluoride), a plastic which has very good particle shedding characteristics which is essential if the very clean conditions necessary for testing high efficiency filters are to be achieved. A diagram of the test rig is given in Figure 3.1.

All the filters in the rig (F1, F2, F3) are 10 inch cartridges of pleated membrane with Millipore fittings unless otherwise stated.
Deionized water is provided by a Milli-Q system. It is essential that deionized water is used for the filter evaluation because the performance of liquid filters is dependent on the chemical nature of the liquid. The Milli-Q system has a facility for measuring the resistivity of the water leaving the Milli-Q or in the filter test rig. The ideal resistivity is 18 Meg-ohm centimetre.

The reservoir is filled with approximately 100 litres of water which is then pumped through two prefilters (F1, F2) before the particle injection point. F1 is a 0.2 micron rated membrane filter, F2 is a 0.1 micron rated membrane filter. These prefilters are essential because the deionized water from the Milli-Q system is not sufficiently clean for filter testing. Also, the pump is a continuous source of contamination and particle levels suitable for point of use membrane filter evaluation would not be possible without filtration after the pump.

A pressure of at least 30 psi downstream of the test filter (F3) is maintained by the diaphragm valve, D2. The reason for this is to prevent air bubble formation which would result in particle counts being recorded.

The test flowrate is constant at 10 litres per minute. This is set with the diaphragm valve, D1, on the pump by-pass line. The flow is pulsed by manual operation of ball valves B4 and B5 such that the water is diverted away from the test filter through the return line to the reservoir and then immediately restoring the flow to the filter.

Particles are injected into the rig using a Havard Apparatus infusion pump. This was modified to withstand the full rig pressure of 50 psi. A syringe is filled with a dilute suspension of latex particles and this is injected at a rate controlled by the infusion pump. The latex particle suspension is injected into a 6 mm o.d. flexible tube which runs from the upstream side of prefilter F2 (see Figure 3.1) to a point immediately upstream of valve B3. The pressure drop across the filter F2 is just sufficient to provide a flow of water which carries the particle feed into the main flow of the rig. The turbulent flow of water in the rig and the two bends between the injection point and the upstream particle sensor provide adequate mixing so that particle concentrations upstream of the filter can be measured accurately.
be maintained reasonably constant if required. This concentration can be rapidly adjusted by changing the settings on the infusion pump so that a controlled change or variation in upstream concentration is possible.

A Particle Measuring Systems (PMS) Laser Liquid Particle Spectrometer - X with two sensors sensitive to 0.3 micron is used for particle counting. The particle size calibration of the instrument has been a problem so that all the experimental work was carried out with monosized feeds, the instrument being used as a counter rather than a sizer. A sample flow of 100 ml per minute is taken from a position upstream of the test filter through the sensor and returned to the reservoir. The same arrangement exists on the downstream side of the filter. It is very important on the downstream side that the sample point is upstream of the diaphragm valve D2 so that the minimum pressure of 30 psi is maintained avoiding air bubble generation. It is also important that there are no potential contamination sources between the downstream side of the test filter and the sample point.

The reason for the cooling coil is that the temperature must not be allowed to rise above room temperature as a result of the heat generated by the water pump. This is one of the measures against bacterial growth in the test rig which can be a major source of contamination. The other major control is to regularly sanitize by recirculating a dilute hydrogen peroxide solution through the rig.

The whole test rig and the Milli-Q water system are enclosed in a clean cabinet. The housing for F3 is immediately below a large HEPA filter so that contamination of the test rig or the filter should not occur when a new filter is placed in the rig. Clean room clothing including face masks and gloves is worn when working in the clean cabinet.
3.3 TEST PROCEDURES

Because of the difference in efficiency between the point of use membranes and the filters of larger rating which were tested, different test procedures were followed for the different types of filter.

3.3.1 Test Procedure for Point of Use Membrane Filters

The procedure followed for testing a point of use membrane filter rated at 0.1 or 0.2 micron is summarised below.

1. Fill the rig with de-ionised water from the Milli-Q and circulate through the prefilters recording particle counts until both sensors read very low particle counts (<5 per 100 ml greater than 0.3 micron).*

2. Divert the flow away from the test filter housing (F3) through the return line to the reservoir and insert the filter to be tested into its housing. The plastic surrounding the test filter should be broken at the last possible moment and after the plastic has been broken the body of the filter cartridge kept within the plastic while the head of the filter is located in the head of the housing. The plastic is then removed and the bowl of the housing is connected. These precautions are to ensure minimum particle contamination of the test filter before it is inserted into its housing.

3. Restore the flow to the test filter by opening B5 and closing B4 and record particle counts upstream and downstream of the test filter until the system is clean (<5 particles per 100 ml greater than 0.3 micron).*

4. Pulse the flow to the test filter several times by operation of B4 and B5, recording particle counts upstream and downstream of the filter before and after each pulse. Between pulses the system must be allowed to return to a clean level.*

5. Allow the system to clean to zero particle counts downstream of the test filter.*
6. Commence injection of 0.36 micron latex spheres, recording particle counts upstream and downstream of the test filter. The filter is to be challenged for 24 hours at each of the following particle concentrations: 5,000, 10,000, and 15,000 particles per 100 ml.

7. If necessary, allow the system to return to low particle counts (<5 per 100 ml).*

8. Pulse the flow to the test filter several times, recording particle counts upstream and downstream of the test filter. Ensure the system returns to low particle counts between each pulse.

9. Perform an integrity test. This is a bubble point test performed by a Millipore Integretest-E instrument. (The bubble point test is described in Section 2.3.)

The above tests are performed on two samples of each filter type.

The purpose of the integrity test is to ensure that the filter sample is not damaged and that the cartridge is correctly housed and sealed. The reason why the test is performed at the end of the test procedure rather than at the beginning is that it is possible to introduce a low level of particle contamination by the test and this is therefore best left to the end.

Elsewhere in this thesis item 4 is referred to as the clean pulsing test, item 6 is the steady state efficiency test and item 8 is the dirty pulsing test.

* As the rig sometimes requires several hours or even days to reduce from particle counts of 5 per 100 ml greater than 0.3 micron to less than 1 per 100 ml, a downstream count of 5 per 100 ml is considered adequate before inserting a new test filter or commencing a pulse, as either of these operations generates particle counts orders of magnitude higher than this. However, before commencing the steady state efficiency test much cleaner levels are required as the mean particle counts recorded are often less than 1 particle per
100 ml greater than 0.3 micron. Here, mean particle counts of less than 0.5 micron are required, with a large frequency of 5 or even 10 successive zero counts. The meaning of such particle counts has been considered by Lloyd.70

3.3.2 Test Procedure for 0.4 and 0.45 micron Filters

The procedure for the 0.4 and 0.45 micron filters differed from that described above in that the required level of cleanliness of the test rig before commencing testing was much easier to achieve because the downstream particle counts measured even in the steady state efficiency tests was much higher. The other main difference was that the effect of particle size was considered whereas in the point of use filter tests only one particle size was used.

A further difference is that with some of the filters tested pulsed water flow tests were not employed but pulses and step changes in the upstream particle concentration were introduced.

For those filters which were tested under pulsed water flow conditions the test procedure was as follows:

1. Fill the rig with de-ionised water from the Milli-Q system and circulate through the prefilters, recording particle counts until both sensors read particle counts of less than 100 particles per 100 ml greater than 0.3 micron.

2. Insert the test filter in the same way as for the point of use filter test procedure.

3. Restore the flow to the test filter and record particle counts until the downstream sensor records less than 100 particles per 100 ml greater than 0.3 micron.

4. Perform the clean pulsing test in the same way as for the point of use filter test procedure, allowing the system to clean up between pulses to a level within a few per cent of the downstream particle count registered for the first pulse.

5. Allow the system to clean up to less than 50 particles per 100 ml greater than 0.3 micron.
6. Commence injection of particles of the required size and concentration.

7. Perform dirty pulsing tests in the same way as for the clean pulsing.

8. If the filter is to be tested with other particle sizes repeat steps 5 to 7.

Note, if the pulsing tests are not to be performed on a filter the following procedure is to be followed.

1. No change to above procedure for 0.4 and 0.45 micron filters.

2. No change to above procedure for 0.4 and 0.45 micron filters.

3. As item 3 for above procedure but allow system to clean up to less than 50 particles per 100 ml greater than 0.3 micron.

4. As item 6 in above procedure for 0.4 and 0.45 micron filters.

5. After switching off particle challenge, if another particle size is to be used allow system to clean up to less than 50 particles per 100 ml then repeat item 4.
3.4 PARTICLE COUNTING IN STEADY AND PULSED WATER FLOW CONDITIONS

Particle counting in ultra pure water is a technique which the experienced operator will know to be more complicated than it appears at first sight. Process equipment such as pipework, pumps and valves are all potential sources of particle contamination and the arrangement of the counting system must take account of this. However, the generation of particles by process equipment is not the most difficult problem to be solved as its effect can be eliminated by careful design. Other problems are bacterial growth and air bubble generation as an optical particle counter has no way of distinguishing between solid particles, organisms and air bubbles.

Bacterial growth can be eliminated by careful management of the test rig including temperature control and regular sanitizing. Air bubble generation is not so easily controlled unless the pressure in the sample line to the particle sensor is maintained above 30 psi. With the pulsed water flow tests described in this chapter the flow to the filter is momentarily stopped and the pressure falls to zero. As the flow is restored the pressure upstream of the test filter returns to its normal steady state value. Because of this drop in pressure the particle counting on pulsing must be carefully examined to ensure that the results are not due to air bubble generation.

In the initial experiments performed on 0.1 micron rated membrane filters while developing the test procedures described in this chapter, the differences between filters were very marked. These results were widely questioned for several reasons which led to the procedure for particle counting under pulsed flow conditions being improved.

In the early work the particle counts upstream and downstream of the test filter were continuously monitored from several minutes before the pulse to several minutes after the pulse. Counts were recorded every minute, with more than 90% of the total count following each pulse occurring in the first minute. It was not certain whether the particle counts recorded were caused by actual particles released from the filter or by air bubble generation or even vibration of the sensor due to the pulse.
To provide more information about the nature of the particle counts following a pulse, the particle counts were recorded every 10 seconds. If the particle counts were caused by vibration of the sensor or air bubble generation in the sample tubing, a significant proportion of the total count would be expected immediately after the pulse. On the other hand, if the particle counts were caused by something generated at the filter, a time delay would be expected.

The expected time delay is calculated from the dimensions of the sample tubing and the sample flow rate (see Figure 3.2).

![Diagram of test filter and sample tubing](image)

**Figure 3.2**

Distance travelled from test filter to sensor

The main test rig flowrate, \( Q \) is 10 litres per minute and \( q \), the sample flowrate is 0.1 litre per minute. The distance from the downstream side of the filter to the start of the sample tubing, \( \ell \), is of the same order of magnitude as \( L \), the length of the sample tubing. In calculating the time required for particles to arrive at the sensor from the downstream side of the filter, the time to travel the distance, \( \ell \), is therefore negligible.

Assuming plug flow and instantaneous release of particles from the test filter, the time required for particles to arrive at the sensor will be the residence time in the sample tubing. As the particle concentration is very low and the particles are very small (low Stokes Number) the residence time of the particles will equal the residence time of the water. Equation (3.1) can therefore be used.
\[ t_R = \frac{\pi d^2 L}{4q} \]  

(3.1)

where \( d \) is the internal diameter of the sample tubing (4 mm), \( L \) is 60 mm and \( q \) is 0.1 litres per minute. This gives a value of \( t_R \) of 4.5 seconds.

If equation (3.1) holds the distribution of particle counts with time downstream of the filter following a pulse would be as shown in Figure 3.3.

![Figure 3.3](image)

**Figure 3.3**

Distribution of particle counts with time assuming plug flow and instantaneous release of particles

However, the plug flow model is somewhat idealised and laminar flow is more likely to be in existence (Reynold’s Number is less than 2,000 at 0.1 l/min). Under laminar flow conditions the mean fluid velocity is related to the maximum fluid velocity by equation (3.2)

\[ \bar{v} = 0.5v_{\text{max}} \]  

(3.2)

There will therefore be a distribution of residence times about the mean residence time, \( t_R \), so that the distribution of particle counts with time will be similar to that shown in Figure 3.4.
In Figure 3.4, $t_0$ is the time delay, that is, the time before which no particles are counted, and $t_p$ is the time to reach a peak in the curve. If instantaneous particle release occurs $t_p$ will be the mean residence time from equation (3.1), that is, 4.5 seconds. Given that equation (3.2) also applies:

$$\tau_0 = 0.5 \tau_R$$

Therefore, from equation (3.3) $t_0$ will be 2.3 seconds.

In summary, when testing a filter under pulsed flow conditions, if no particles are counted before 2.3 seconds it is very unlikely that the cause of the counts is sensor vibration or air bubble generation in the sample tubing.

This is checked by setting the particle counter to record counts every 10 seconds and introducing a time lag between the particle counter and the occurrence of the pulse. This technique is demonstrated by Figures 3.5 to 3.8 which show data for pulsing tests on a 0.1 micron membrane filter.

In Figure 3.5 the timing of the particle counter coincides with the occurrence of the pulse, that is, there is no time lag between counter and pulse. The time intervals 1 to 6 therefore represent times 0 to 10, 10 to 20, 20 to 30,... seconds after the pulse. It is clear from Figure 3.5 that with no time lag 90% of the count appears in time interval 1, that is, 90% of the count appears in the first 10 seconds after the pulse.
Figure 3.5
Distribution of particle count with time for no time lag between counter interval and pulse

Figure 3.6
Distribution of particle count with time for three seconds lag between counter interval and pulse
Figure 3.6 shows the situation with a 3 second time lag. Here the time intervals 1 to 6 represent times from 7 seconds before the pulse to 3 seconds after the pulse, 3 to 13 seconds, 13 to 23 seconds, ...and so on. It is clear from Figure 3.6 that very little of the count appears in the first 3 seconds with nearly all the count between 3 and 13 seconds after the pulse.

Figure 3.7

Distribution of particle count with time for four seconds lag between counter interval and pulse

Figure 3.7 shows that 20% of the count appears in the first 4 seconds with over 70% between 4 and 14 seconds after the pulse. From Figure 3.8 50% of the count appears in the first 5 seconds with most of the remainder in the interval from 5 to 15 seconds after the pulse.
The conclusion is that

\[ 3s < t_0 < 4s \]  \hspace{1cm} (3.4)

\[ 4s < t_p < 5s \]  \hspace{1cm} (3.5)

In this case it is clear that the particle counts recorded downstream of the test filter following pulsing were not due to sensor vibration or air bubble generation in the sample tubing. This analysis must be carried out for each pulse performed on each filter tested.

The above method will determine whether or not sensor vibration or air bubble generation in the sample tubing are responsible for the particles counted downstream of a filter on pulsing, but they do not prove that the particle counts are due to release of test particles from the filter. All that can be drawn from the above method is that the cause of the particle counts is generated at the filter. This could be air bubble generation in the filter,
release of test particles from the filter or shedding of filter debris. By comparing the results of pulsing before and after loading with test particles, that is, clean and dirty pulsing, it is possible to determine whether test particles are released from the filter.

3.5 SUMMARY OF EXPERIMENTAL PROGRAMME

A number of 0.1 and 0.2 micron point of use membrane cartridge filters were tested under steady and pulsed water flow conditions. The challenge particle size was 0.36 micron. By pulsing the water flow before and after loading the filter with particles it was possible to determine whether test particles were released from the filter by pulsing the flow. Two samples of each filter type were tested. The test procedure is presented in Section 3.3 of this chapter.

Three filter types of larger particle rating were also tested under steady and pulsed water flow conditions. A range of particle sizes was used and the concentration of the challenge was varied in order to determine the transient nature of filtration. Two of these filter types were membranes, one rated at 0.4 micron, the other at 0.45 micron. The other filter type was a 0.45 micron rated prefilter, designed to operate in a manner intermediate to that of a membrane filter and a depth filter.

The results of the tests performed on these filters are presented in Chapters 4 to 6.
CHAPTER 4

RESULTS OF TESTING FILTERS UNDER
PULSED FLOW CONDITIONS

4.1 Introduction

4.2 Pulsed flow results for point of use membrane filters

4.3 Pulsed flow results for 0.4 and 0.45 micron rated filters

4.4 Conclusions
4.1 INTRODUCTION

This chapter is concerned with the results of testing filters under pulsed flow conditions. 0.1 and 0.2 micron rated point of use membrane filters were tested with pulsed water flows after having been loaded with 0.37 micron latex spheres. It was found that in some cases particles were released by pulsing the flow.

Three filters of larger rating were also tested under pulsed flow conditions. Two of these filters were membranes, one rated at 0.4 micron, the other at 0.45 micron. The other filter was a 0.45 micron rated prefilter, with more depth and a wider pore size distribution than the membrane filters. All three filters showed significant release of test particles in response to pulsing, even for particles significantly larger than the rating of the filter. The pulsed flow results are presented for the 0.4 micron membrane filter and it can be seen that there is a correlation between the number and the size of particles released by pulsing.

Consideration of the time distribution of the particle counts produced by pulsing the water flow shows that for the membrane filters the particles are released instantaneously but that for the prefilter several minutes may be required for some of the particles to be released. This time delay between the occurrence of the pulse and the detection of the particles downstream of the filter is dependent on the particle size.
4.2 PULSED FLOW RESULTS FOR POINT OF USE MEMBRANE FILTERS

The purpose of this section is to show that in some cases particle release can occur from point of use filters under pulsed flow conditions. Pulsed flow test results are shown for two filters only. Figure 4.2.1 shows the results for a 0.2 micron rated filter. The first 9 pulses were performed before the filter was loaded with test particles (clean pulsing). The following 16 pulses were performed after the filter was loaded with test particles (dirty pulsing). After the first few clean pulses the particle count in response to a pulse was low. However, it is clear that the dirty pulsing data show a significant increase in the particles counted after a pulse. The suggestion is that the increase in count from the end of the clean pulsing to the dirty pulsing is due to release of test particles.

A different picture is seen for a 0.1 micron membrane filter supplied by the same manufacturer as the 0.2 micron filter discussed above. Figure 4.2.2 shows that dirty pulsing does not produce an increase in particle count. Clearly there can be no significant release of test particles from the filter in this situation.
Figure 4.2.2
Pulsed flow results for a 0.1 micron membrane filter

The test for the distribution of particle counts with time following a pulse described in section 3.4 was carried out for each of the pulses performed on these two filters. In every case the time distribution of the data was as expected, that is, no particles were counted in the first three seconds following a pulse, with more after 4 seconds and by 5 seconds more than 50% of the total count for that pulse. Sensor vibration or air bubble generation in the sample tubing can therefore not be responsible for the above results.
4.3 PULSED FLOW RESULTS FOR 0.4 AND 0.45 MICRON RATED FILTERS

The purpose of this work was to determine whether or not particles which were captured by these filters under steady water flow conditions could be released from the filter under pulsed water flow conditions. All three filters showed significant particle release even with particles larger than their rating showing that sufficient flexing of the filter medium can occur for penetration to take place.

The results for one of the filters are presented in some detail. The 0.4 micron rated membrane filter was loaded with several particle sizes, pulsed flow tests being performed between each loading. The number of particles loaded on the filter was similar for each particle size. Figure 4.3.1 shows the effect of particle size on the number of particles removed from the filter with each pulse.

It is clear from this graph that there is a correlation between the particle size and the number of particles released when the flow is pulsed. The 0.312 micron particles were released in by far the largest amount with the two tests with 0.37 micron particles (0.37 and 0.37 rep)* showing fairly similar results. It is not surprising that the larger the particle, the fewer particles are released by pulsing as the flexion of the membrane would have to open the pores to a larger extent to remove more larger particles. The most important result is that particles which are captured under steady water flow conditions are released when the flow is pulsed.

*Note, 0.37 rep was a repeat of the original test with 0.37 micron particles.
Effect of particle size on pulsing for 0.4 micron rated membrane filter

The time distribution of the pulsing data agreed with the predicted distribution, that is, very few particles were counted in the first three seconds after a pulse with most having been counted after about 5 seconds. This was also the case for the pulsing results for the 0.45 micron membrane filter. However, with the 0.45 micron prefilter, the time distribution of the pulsing data was different. Neither sensor disturbance nor air bubble generation in the sample tubing could be the cause of the particle counts because no particle counts were recorded in the first three seconds. However, considerably longer was required for the peak in the count to be reached. With the membrane filters tested, nearly all of the particles were counted in the first minute after each pulse, with none counted after about three minutes. With the prefilter it often required 20 minutes for the particle count to have returned to the background level achieved before the flow was pulsed. It is clear that with the prefilter, particles which are released take considerably longer to penetrate the filter and be detected downstream.
Examples of the time distribution data for counts following pulsing are shown in Figures 4.3.2 to 4.3.17. Figure 4.3.2 shows the time distribution of the particle counts following the first pulse performed on the 0.45 micron prefilter after it had been challenged with 0.46 micron particles. Although it appears from this figure that nearly all the particles are counted in the first thirty seconds after the pulse, Figure 4.3.3 shows that significant counts are still being recorded after 4 minutes and that the total count in each 10 second period is reducing very slowly at this time.

**Figure 4.3.2**

Time distribution of particle counts following the first pulse performed on the 0.45 micron prefilter after challenging it with 0.46 micron particles

Figures 4.3.4 and 4.3.5 show a similar pattern for the second pulse performed on the 0.45 micron prefilter after challenging it with 0.46 micron particles. Most of the particles are counted in the first thirty seconds (Figure 4.3.4) but significant counts are still recorded after more than three minutes (Figure 4.3.5).
Figure 4.3.3
Significant particle counts minutes after the first pulse performed on the 0.45 micron prefilter after challenging it with 0.46 micron particles.

Figure 4.3.4
Time distribution of particle counts following the second pulse performed on the 0.45 micron prefilter after challenging it with 0.46 micron particles.

Comparison of Figures 4.3.2 and 4.3.4 show that the total count for the second pulse is less than that for the first pulse. Figures 4.3.3 and 4.3.5 show that the delay in particles being detected following a pulse is more significant for the first pulse than for the second pulse.
Significant particle counts minutes after the second pulse performed on the 0.45 micron prefilter after challenging it with 0.46 micron particles.

Figures 4.3.6 to 4.3.9 show the time distribution of the particle counts for the first two pulses performed on the prefilter after challenging it with 0.76 micron particles. In each case nearly all the particles are counted between 3 and 13 seconds after the pulse (see Figures 4.3.6 and 4.3.8) and the particle counts are approaching the level recorded after 3 seconds within 1 minute of the pulse being performed. Clearly, the delay in 0.76 micron particles reaching the sensor after a pulse is much less significant than for 0.46 micron particles.
Figure 4.3.6
Time distribution of particle counts following the first pulse performed on the 0.45 micron prefilter after challenging it with 0.76 micron particles

Figure 4.3.7
No significant particle counts from 1 minute after the first pulse performed on the 0.45 micron prefilter after challenging it with 0.76 micron particles
Figure 4.3.8
Time distribution of particle counts following the second pulse performed on the 0.45 micron prefilter after challenging it with 0.76 micron particles

Figure 4.3.9
No significant particle counts from 1 minute after the second pulse performed on the 0.45 micron prefilter after challenging it with 0.76 micron particles

Figures 4.3.10 to 4.3.13 show the time distribution of the particle counts for the first two pulses performed on the 0.4 micron membrane filter after loading it with 0.37 micron particles. Nearly all the particle counts are recorded between 3 and 13 seconds after each pulse (see Figures 4.3.10 and 4.3.12) with particle counts approaching the level recorded after 3 seconds within 1 minute (see Figures 4.3.11 and 4.3.13).
Figure 4.3.10
Time distribution of particle counts following the first pulse performed on the 0.4 micron membrane filter after challenging it with 0.37 micron particles

Figure 4.3.11
No significant particle counts from 1 minute after the first pulse performed on the 0.4 micron membrane filter after challenging it with 0.37 micron particles
Figure 4.3.12
Time distribution of particle counts following the second pulse performed on the 0.4 micron membrane filter after challenging it with 0.37 micron particles.

Figure 4.3.13
No significant particle counts from 1 minute after the second pulse performed on the 0.4 micron membrane filter after challenging it with 0.37 micron particles.

After challenging the 0.4 micron membrane filter with 0.31 micron particles the time distribution of the particle counts following a pulse show some delay in particles being counted, but much less than for the 0.45 micron prefilter after challenging it with 0.46...
micron particles. In fact, Figures 4.3.14 to 4.3.17 show that no significant particle counts are recorded after the first minute following a pulse. However, the delay is significant compared with that for 0.37 micron particles on the 0.4 micron membrane filter.

![Figure 4.3.14](image)

**Figure 4.3.14**
Time distribution of particle counts following the first pulse performed on the 0.4 micron membrane filter after challenging it with 0.31 micron particles

![Figure 4.3.15](image)

**Figure 4.3.15**
No significant particle counts from 1 minute after the first pulse performed on the 0.4 micron membrane filter after challenging it with 0.31 micron particles
Time distribution of particle counts following the second pulse performed on the 0.4 micron membrane filter after challenging it with 0.31 micron particles.

No significant particle counts from 1 minute after the second pulse performed on the 0.4 micron membrane filter after challenging it with 0.31 micron particles.
4.4 CONCLUSIONS

In some cases point of use membrane filters will release particles under conditions of pulsed water flows, even at particle sizes considerably larger than the rating of the filter.

The 0.4 and 0.45 micron filters tested all showed significant particle release when the flow was pulsed. For the 0.4 micron membrane filter it is clear that there is a correlation between particle size and the number of particles released from the filter by a pulse.

The particles released from the membrane filters were detected downstream of the filter within one minute of the pulse being performed. However, for the prefilter there was a delay between the occurrence of the pulse and detection of some of the particles released from the filter. Most of the particles released are detected in the first minute after the pulse but there are still significant numbers of particles being detected several minutes after pulsing. This effect is a function of particle size. The delay for 0.76 micron particles was considerably less than that for 0.46 micron particles.

There was also evidence of a small time delay in particles being detected after pulsing the flow to the 0.4 micron membrane filter. This is also dependent on particle size, a more significant delay being recorded for 0.31 micron particles than for 0.37 micron particles. However, the delay is much less than for the prefilter.
CHAPTER 5

RESULTS OF TESTING THE 0.45 MICRON RATED PREFILTER

5.1 Introduction

5.2 First 0.45 micron prefilter sample

5.3 Second 0.45 micron prefilter sample

5.4 Third 0.45 micron prefilter sample

5.5 Conclusions
5.1 INTRODUCTION

In Chapter 4 the results of testing cartridge filters under pulsed flow conditions were presented. In many cases particles which were captured by filters under steady water flow conditions were released when the flow was pulsed. All of the 0.4 and 0.45 micron rated filters showed significant particle release on pulsing. It was shown for the 0.4 micron membrane filter that the extent to which particle release occurs is dependent on particle size.

For the membrane filters there was very little time delay between the occurrence of a pulse and the detection of particles downstream of the filter. However, for the 0.45 micron prefilter, a considerably longer time was required for some of the particles released by a pulse to be detected. This effect was found to be a function of particle size. For the 0.4 micron membrane filter there was some delay observed, this also being a function of particle size.

It was decided that the retention of particles by the prefilter under steady water flow conditions should be considered and compared with that of the membrane filters. In this chapter the results of the steady state water flow tests on the prefilter are presented. Three samples of the prefilter were tested with a range of challenge particle sizes and concentrations. It was found that the efficiency of filtration and the change in efficiency with time or loading was dependent on the challenge particle size. For particles of similar size to the rating of the filter it was found that some of the particles penetrate the filter instantaneously, whereas others experience a residence time within the filter, resulting in a time delay between upstream and downstream concentrations. In fact, two types of delay are observed, a short term delay of minutes and a long term delay which may be of hours.
For particles considerably smaller or larger than the rating of the filter none of the particles which penetrate the filter experience a residence time within the filter, that is, all of the penetration is direct.

Under certain conditions, such as high challenge concentration or high filter loading, a shift from delayed to direct penetration was observed, resulting in an overall increase in penetration. This change may be temporary in the case of high challenge concentration or permanent in the case of high filter loading.
5.2 FIRST 0.45 MICRON PREFILTER SAMPLE

The first sample of the prefilter was tested with two particle sizes: 0.46 and 0.65 micron. The results for each particle size are presented below.

5.2.1 Challenge with 0.46 micron particles on the first prefilter sample

The first prefilter sample was tested with 0.46 micron particles for approximately 1300 minutes. Figures 5.2.1 to 5.2.3 show the variation in upstream concentration, downstream concentration and penetration for the first 102 minutes of loading with 0.46 micron particles. The data are recorded every minute. The downstream concentration immediately before commencing the challenge was zero.

Note: penetration is defined as the ratio of downstream concentration to upstream concentration, and is expressed in these figures as a percentage.

![Figure 5.2.1](image)

**Figure 5.2.1**

Variation in upstream concentration with time for first 102 minutes of the challenge with 0.46 micron particles on the first prefilter sample
A comparison of Figures 5.2.1 and 5.2.2 for the first 20 minutes of the test shows that there is a relationship between upstream and downstream concentrations but that this is not proportional. While the upstream concentration oscillates about its ultimate level, the downstream concentration gradually increases to its steady state level, suggesting that there is a time delay between upstream and downstream concentrations. However, the large peak in the upstream concentration after two minutes produces a simultaneous
peak in the downstream concentration although the magnitude of the downstream peak is less significant than that upstream. It appears from these early results that part of the downstream concentration is determined directly by the upstream concentration at that time, but that part of it is determined by the upstream concentration a few minutes earlier, that is, there is a time delay between upstream and downstream concentrations.

Figure 5.2.3 shows that the penetration is increasing over the first part of the test in what appears to be an exponential manner. After 98 minutes the upstream concentration is suddenly reduced to less than half its previous level. The penetration rises sharply, suggesting that the decrease in the downstream concentration occurs at a different rate to that of the upstream concentration, again suggesting the presence of time delay between upstream and downstream concentrations.

Figure 5.2.4
Variation in upstream concentration up to 1200 minutes of the challenge with 0.46 micron particles on the first prefilter sample
Figure 5.2.5
Downstream response (refer Figure 5.2.4)

Figure 5.2.6
Detail of fall in upstream concentration
Figures 5.2.4 and 5.2.5 show the upstream and downstream concentrations from 159 to 1239 minutes after the start of the test with the data recorded every 10 minutes. Figure 5.2.4 shows a fall in the upstream concentration from 159 to 239 minutes which produces a corresponding fall in downstream concentration (see Figure 5.2.5). However, Figures 5.2.6 and 5.2.7 show that the reduction in downstream concentration continues for at least 30 minutes after the upstream concentration has reached its minimum level. During this fall in upstream and downstream concentrations the penetration is initially constant but then falls showing that the upstream and downstream concentrations are reducing at different rates (see Figure 5.2.8). For the remainder of the time shown in Figures 5.2.4 and 5.2.5 the downstream concentration appears to vary independently, and from about 600 minutes onwards there is a steady increase in downstream concentration while the upstream concentration remains constant. This is shown by a small increase in the penetration from 600 minutes onwards (see Figure 5.2.8).

The indication from the data in Figures 5.2.4 to 5.2.8 is that there is a time delay between upstream and downstream concentrations.
The next period of loading is represented by Figures 5.2.9 to 5.2.11. The large increase in upstream concentration is matched by a corresponding increase in downstream concentration. It is clear that at the higher upstream concentration the filter is considerably less efficient (see Figure 5.2.11).

**Figure 5.2.8**
Variation in penetration (refer Figures 5.2.4 and 5.2.5)

**Figure 5.2.9**
Step change in upstream concentration of 0.46 micron particles on the first prefilter sample
When the upstream concentration is reduced after the step change a time lag between upstream and downstream concentrations is observed. The upstream count reduces to very low levels in just a few minutes, unlike the downstream count which takes considerably longer to fall to a level approaching that observed before commencing the tests on this filter. Figure 5.2.10 shows that the downstream count falls in proportion to
the upstream count for the first ten minutes. In fact, the downstream level has fallen by more than 80% in this time. However, after this the downstream level takes several hours to reduce to 600 particles per 100 ml (see Figures 5.2.13 to 5.2.15), a level which is still orders of magnitude higher than the downstream concentration before testing on the filter was started. The relationship between upstream and downstream levels immediately after reducing the challenge is illustrated in Figure 5.2.12. For about 100 minutes after the upstream concentration was reduced to zero the downstream concentration was recorded every minute (see Figure 5.2.13). Figure 5.2.14 shows the rest of the data which was recorded every 10 minutes and the whole decay time is shown in Figure 5.2.15.

![Graph showing upstream and downstream particle counts over time]

**Figure 5.2.12**
Reduction in upstream and downstream concentrations on switching off the 0.46 micron challenge on the first 0.45 micron prefilter sample
Figure 5.2.13
First 100 minutes of decay after the 0.46 micron challenge on the first 0.45 micron prefilter sample has stopped

Figure 5.2.14
Further decay time
5.2.2 Challenge with 0.65 micron particles on the first prefilter sample

A short test with 0.65 micron particles was performed on the first sample of the prefilter. The results are shown in Figures 5.2.16 to 5.2.18.
Figure 5.2.17
Downstream response to the challenge with 0.65 micron particles on the first prefilter sample (refer Figure 5.2.16)

Figure 5.2.18
Variation in penetration for the 0.65 micron challenge on the first prefilter sample (refer Figures 5.2.16 and 5.2.17)

A comparison of Figures 5.2.16 and 5.2.17 shows no apparent time delay between upstream and downstream concentrations. This is clear from the rise in upstream...
concentration after 36 minutes which causes a corresponding rise in downstream concentration and no change in penetration (see Figure 5.2.18), showing that the upstream and downstream concentrations are changing at the same rate.

Figure 5.2.18 shows a relatively high value for the penetration at the time of the peak in upstream and downstream concentrations, that is after 2 minutes. The high values following this are not due to increased levels of penetration but to the rapid fall in upstream and downstream concentrations such that the fairly high downstream residual concentration causes a high ratio of downstream to upstream concentration. Once the upstream concentration rises again, the downstream residual concentration becomes less significant and the calculated penetration falls.

5.2.3 Summary of findings for the first 0.45 micron prefilter sample

With 0.46 micron particles there is an element of the penetration which is direct, that is, there is no time delay between upstream and downstream concentrations. There is also an element of penetration which is delayed, that is, a change in the upstream concentration is not observed downstream until after a certain time delay.

The response of the prefilter to switching on the particle feed was what appeared to be an exponential increase in penetration to about 6%, the delayed penetration being shown by the change in penetration with time. The time delay was also seen at the end of the test when the particle feed was switched off. The downstream concentration decayed in what appeared to be an exponential manner, significant concentrations being recorded 4 hours after the upstream concentration was reduced to zero.

In the event of a step increase in upstream concentration to more than 700,000 particles per 100 ml the penetration increased from 7% to 15%.

With 0.65 micron particles there is no time delay between upstream and downstream concentrations, that is, all the penetration is direct.
One result which was of interest for the 0.65 micron particle challenge was the response of the prefilter to the peak in the upstream concentration which resulted in a peak downstream and an increase in penetration. At the time of the peak the penetration was 3% compared with less than 1% at all other times.
5.3 SECOND 0.45 MICRON PREFILTER SAMPLE

The second prefilter sample was tested with two particle sizes: 0.94 micron and 0.55 micron. The results for each particle size are presented below.

5.3.1 Challenge with 0.94 micron particles on the second prefilter sample

The challenge with 0.94 micron particles lasted for over 600 minutes. The first 35 minutes of the test are shown in Figures 5.3.1 to 5.3.3. There is no apparent time delay between upstream and downstream concentrations.

Figure 5.3.3 shows what appears to be a varying penetration. However, the high calculated penetration values in the first 12 minutes are due to the effect of the relatively high residual downstream concentration when the upstream concentration is low. In the event of the large peak in the upstream concentration after 13 minutes, the penetration is low and remains low (about 0.1%) while the upstream concentration is significant. Note, there is no increase in penetration as a result of the peak in upstream concentration. This contrasts with Figure 5.2.16 to 5.2.18 for 0.65 micron particles when a peak in the upstream concentration resulted in a momentary increase in penetration.

Figure 5.3.1

Variation in upstream concentration for the first 35 minutes of the challenge with 0.94 micron particles on the second prefilter sample
Figures 5.3.4 to 5.3.6 show that there is no time lag between upstream and downstream concentrations and no increase in penetration at high upstream concentration. The large variation in penetration shown in Figure 5.3.6 is due to the relatively large variation in downstream concentration from 15 minutes onwards. This downstream variation is within that which would be expected for a Poisson distribution where the standard
deviation is the square root of the mean value. In this case the mean downstream concentration is approximately 100 particles per 100 ml so that the standard deviation given that the distribution is Poisson would be 10 particles per 100 ml. Most of the variation in Figure 5.3.5 is therefore within two standard deviations of the mean value.

Figure 5.3.4
Decrease in upstream concentration following the peak at 13 minutes

Figure 5.3.5
Downstream response to decrease in upstream concentration (refer Figure 5.3.4)
Figure 5.3.6
Variation in penetration (refer Figures 5.3.4 and 5.3.5)

Figure 5.3.7 to 5.3.9 show the effect of an increase in the upstream concentration, maintenance of the concentration for nearly 400 minutes and a reduction in the upstream concentration to zero.

Comparison of Figures 5.3.7 and 5.3.8 do not show any time delay between upstream and downstream concentrations but the particle concentrations shown in these figures are average values over 10 minute intervals so that any slight time delay would be hidden.

In contrast to the behaviour with 0.46 micron particles there is no gradual decay in downstream concentration when the upstream concentration is reduced to zero. In fact, the downstream concentration is a similar level to the residual concentration before commencing tests on this filter sample within 10 minutes of the upstream concentration being stopped. This is shown in more detail in Figures 5.3.10 and 5.3.11.

The penetration is constant at 0.09%. The increase in the values shown in Figure 5.3.9 after 627 minutes is due to the upstream concentration having reached very low levels so that the effect of the downstream residual concentration becomes significant.
Figure 5.3.7
Increase in upstream concentration of 0.94 micron particles on the second prefiler sample, followed by reduction to zero

Figure 5.3.8
Downstream response (refer Figure 5.3.7)
Figure 5.3.9
Variation in penetration (refer Figures 5.3.7 and 5.3.8)

Figure 5.3.10
Reduction in upstream concentration at the end of the challenge with 0.94 micron particles on the second prefilter sample
5.3.2 Challenge with 0.55 micron particles

After challenging the filter with 0.94 micron particles similar tests were carried out using a challenge of 0.55 micron particles for more than 1800 minutes.

Figures 5.3.12 to 5.3.14 show the results for the first 35 minutes of the test with 0.55 micron particles. Examination of Figures 5.3.12 and 5.3.13 shows that there is a small time delay (less than 5 minutes) between the upstream and downstream concentrations. Figure 5.3.14 shows that the penetration slowly increases with time from an initial value of 0.55% to approximately 0.8%, confirming the presence of a time delay.
Figure 5.3.12
Variation in upstream concentration with time for first 35 minutes of the challenge with 0.55 micron particles on the second prefilter sample

Figure 5.3.13
Downstream response for the first 35 minutes of the challenge with 0.55 micron particles on the second prefilter sample (refer Figure 5.3.12)
Figures 5.3.15 to 5.3.17 show the effect of reducing the challenge concentration from 600,000 particles per 100 ml to 100,000 per 100 ml. Again there is the indication of a slight delay between upstream and downstream concentrations. The delay is shown by Figure 5.3.17, where the penetration rises while both upstream and downstream concentrations are falling, indicating that the downstream concentration is reducing more slowly than upstream. The penetration then reduces while the downstream continues to fall slightly at constant upstream concentration.
Figure 5.3.15
Reduction in the upstream concentration of 0.55 micron particles on the second prefilter sample

Figure 5.3.16
Downstream response (refer Figure 5.3.15)
Figure 5.3.17
Variation in penetration (refer Figures 5.3.15 and 5.3.16)

The challenge with 0.55 micron particles was continued for a further 1700 minutes (see Figures 5.3.18 to 5.3.20). The data were recorded on a 100 minute basis so that any short time delay between upstream and downstream concentrations will not be visible.

From 160 to 1260 minutes the upstream and downstream concentrations fall, apparently in step with each other. However, Figure 5.3.20 shows that the penetration is rising slightly during this time, suggesting that the fall in downstream concentration is slower than the fall in upstream concentration.

It is clear that while the dominant feature of this period of testing is direct penetration there is also another effect causing a delay between upstream and downstream concentrations.
Figure 5.3.18
Upstream concentration to the end of the test with 0.55 micron particles on the second prefILTER sample

Figure 5.3.19
Downstream response (refer Figure 5.3.18)
5.3.3 Summary of findings for the second prefilter sample

There is no delayed penetration for the prefilter with 0.94 micron particles. There is no change in efficiency with high upstream concentration or peaks in the upstream concentration.

The penetration for 0.94 micron particles is 0.1%.

There is delayed penetration for the prefilter with 0.55 micron particles, the penetration at the start of the test rising in what appears to be an exponential manner to its steady state level. The steady state penetration achieved after the start of the challenge with 0.55 micron particles was 0.8 to 1%. There is evidence of some long term delay between upstream and downstream concentrations being in operation, the penetration rising slowly, but significantly over a number of hours.
5.4  THIRD 0.45 MICRON PREFILTER SAMPLE

The results of testing the first two prefilter samples indicate that for certain particle sizes a time delay between upstream and downstream concentrations exists and that in the event of a peak in the upstream concentration an increase in penetration occurs. Of the particle sizes used in these experiments the one for which the above effects are most prominent is 0.46 micron. The third prefilter sample was therefore only tested with 0.46 micron particles in order to determine the mechanisms in operation which result in the changes in filter performance mentioned above.

5.4.1 First challenge with 0.46 micron particles on the third prefilter sample

The first challenge with 0.46 micron particles on the third prefilter sample is represented in Figures 5.4.1 to 5.4.3. At first sight these figures suggest that a time delay between upstream and downstream concentrations is occurring. This is particularly noticeable from the nature of the downstream response to the step increase in upstream concentration occurring after 217 minutes.

![Figure 5.4.1](image)

Figure 5.4.1

Variation in upstream concentration for the first challenge with 0.46 micron particles on the third prefilter sample
Figures 5.4.2 and 5.4.3 show the results for the first challenge with 0.46 micron particles on the third prefilter sample (refer Figures 5.4.1 and 5.4.2).

Figures 5.4.4 to 5.4.6 show the results for the first 40 minutes of the test. It is clear that the peak in the upstream concentration after 3 minutes causes a peak in the downstream concentration and in the penetration. The large penetration values after this peak are due to the effect of the relatively high downstream residual concentration at low upstream concentration.
Figure 5.4.4
Variation in upstream concentration for the first 40 minutes of the first challenge with 0.46 micron particles on the third prefilter sample.

Figure 5.4.5
Downstream response to the first 40 minutes of the first challenge with 0.46 micron particles on the third prefilter sample (refer Figure 5.4.4)
Figure 5.4.6
Penetration during the first 40 minutes of the first challenge with 0.46 micron particles on the third prefilter sample (refer Figures 5.4.4 and 5.4.5)

The response of the filter to the sharp increase in upstream concentration after 26 minutes results in what appears to be an exponential increase in penetration (see Figure 5.4.7) from 2% to 3.5%, showing that there is a time delay between upstream and downstream concentrations.

Figure 5.4.7
Variation in penetration at increased concentration (refer Figures 5.4.4 to 5.4.6)
The next period of the first challenge with 0.46 micron particles on the third prefilter sample is shown in Figures 5.4.8 to 5.4.10. The time delay between upstream and downstream concentrations is shown by the exponential shape of the penetration curve (see Figure 5.4.10). In addition, the peaks in the upstream concentration after 54 and 100 minutes produce peaks in the downstream concentration and the penetration.

![Figure 5.4.8](image)

**Figure 5.4.8**

Variation in upstream concentration up to 110 minutes of the first challenge with 0.46 micron particles on the third prefilter sample

![Figure 5.4.9](image)

**Figure 5.4.9**

Downstream response (refer Figure 5.4.8)
Following the fall in upstream concentration shown at the end of the data in Figure 5.4.8, the upstream concentration was maintained constant for approximately 120 minutes, after which a step increase in upstream concentration was introduced (see Figure 5.4.11). The downstream response and the penetration are shown in Figures 5.4.12 and 5.4.13. The time delay between upstream and downstream concentrations is shown by the exponential shape of the increase in downstream concentration. It is also interesting to note that the penetration following the step increase in upstream concentration reaches 10% compared with 4% before the step change (see Figure 5.4.13).
Figure 5.4.11
Step increase in upstream concentration during the first challenge with 0.46 micron particles on the third prefilter sample

Figure 5.4.12
Downstream response to the step increase in upstream concentration during the first challenge with 0.46 micron particles on the third prefilter sample (refer Figure 5.4.11)

The particle feed to the prefilter was switched off after 363 minutes. The reduction in upstream and downstream concentrations is shown in Figures 5.4.14 and 5.4.15. By the time the upstream concentration had reduced to zero the downstream concentration was still more than 10,000 particles per 100 ml, compared with the residual count before
commencing the challenge which was less than 50 particles per 100 ml. The downstream concentration was still more than 5,000 particles per 100 ml after another 5 minutes but the decay was not monitored beyond this point.
Figure 5.4.15
Downstream response at the end of the first challenge with 0.46 micron particles on the third prefilter sample (refer Figure 5.4.14)

Summary of findings for the first challenge with 0.46 micron particles on the third prefilter sample

The time delay between upstream and downstream concentrations is shown by the apparently exponential increase in penetration in response to a step increase in upstream concentration. Further, when the particle feed to the filter is switched off, reducing the upstream concentration to zero, there is a gradual decay in downstream concentration.

In the event of peaks in the upstream concentration there is an instantaneous response downstream and a marked increase in penetration.

The steady state penetration reached after approximately 100 minutes was 4% compared with 10% in response to the step increase in upstream concentration after 220 minutes.
5.4.2 Second challenge with 0.46 micron particles on the third prefilter sample

The second challenge with 0.46 micron particles on the third prefilter sample is summarised in Figures 5.4.16 to 5.4.18. It is clear that the peak in the upstream concentration at the start of the challenge results in a peak in the downstream concentration and the penetration. The penetration at this time is 13%. There is no evidence of a time delay between upstream and downstream concentrations at the time of the upstream peak.

Figure 5.4.16
Variation in upstream concentration for the second challenge with 0.46 micron particles on the third prefilter sample
After the upstream peak, both upstream and downstream concentrations fall, resulting in a penetration of 4% after 22 minutes. The penetration does not fall immediately to 4% but decays over several minutes, suggesting that the upstream and downstream concentrations are reducing at different rates, that is, a time lag between upstream and downstream concentrations exists. Examination of Figure 5.4.19 shows that the
penetration falls rapidly from 13% to 6%, after which the rate of decay in the penetration is considerably slower. The suggestion is that the time delay between upstream and downstream concentrations only becomes apparent 2 minutes after the peak in the upstream concentration.

Figure 5.4.19
Detailed decay in penetration after the peak in upstream concentration

The end of the second challenge with 0.46 micron particles on the third prefilter sample is shown in Figures 5.4.20 and 5.4.21. The particle feed was switched off after 376 minutes and it is clear from a comparison of these two figures that the rate of decay of the downstream concentration is much less than that of the upstream concentration.
The decay in downstream concentration is shown in more detail in Figure 5.4.22. The upstream concentration falls from over 300,000 to less than 200 particles per 100 ml in 5 minutes but Figure 5.4.22 shows that the downstream concentration is still more than 2,800 particles per 100 ml at this time and is still more than 1,500 particles per 100 ml 60 minutes later.
Figure 5.4.22
Detail of decay in downstream concentration at the end of the second challenge with 0.46 micron particles on the third prefilter sample

Summary of findings for the second challenge with 0.46 micron particles on the third prefilter sample

The time delay between upstream and downstream concentrations is shown by the difference in the rate of decrease in concentration after the peak occurring at the start of the challenge. Also, when the particle feed to the filter is switched off the downstream concentration decays in what appears to be an exponential manner.

In the event of a peak in the upstream concentration there is an instantaneous downstream response and a large increase in penetration. As the upstream concentration reduces following the peak, the delay between upstream and downstream concentrations returns.

The steady state penetration of 0.46 micron particles is 4%, compared with a penetration at the time of the upstream peak of 13%.
5.4.3 Third challenge with 0.46 micron particles on the third prefilter sample

Having examined the effect of steady state challenge concentrations and step changes in challenge concentration, the third set of tests on this prefilter sample was designed to determine the effect of peaks, or impulses in the challenge concentration.

The third challenge with 0.46 micron particles on the third prefilter sample is summarised in Figures 5.4.23 to 5.4.24. Six upstream impulses were introduced in the first 200 minutes, each resulting in a corresponding peak in the downstream concentration. Following the sixth impulse the upstream concentration was maintained constant for a period of time after which a step increase in upstream concentration was introduced. The reason for the period of constant upstream concentration and the step increase in upstream concentration was to verify that the behaviour of the prefilter in response to these conditions had not changed since the earlier tests were performed. A seventh upstream impulse was introduced after the step increase in upstream concentration.

![Figure 5.4.23](image)

**Figure 5.4.23**

Variation in upstream concentration for the third challenge with 0.46 micron particles on the third prefilter sample
Some of the impulses are considered more detail in the following figures. The first impulse is shown in Figure 5.4.25. Comparison of this figure with Figure 5.4.26, shows that there is no apparent time delay between the upstream and downstream concentrations in reaching their peak values. Inspection of Figure 5.4.27 shows that the penetration is 12% at the time of the impulse but falls immediately to 6% as the upstream and downstream concentrations fall. However, from 40 minutes onwards the upstream and downstream concentrations are both changing slowly and the penetration rises from below 6% to almost 8%, indicating that some delay between upstream and downstream concentrations exists.
**Figure 5.4.25**
Variation in upstream concentration for the first impulse

**Figure 5.4.26**
Downstream response to the first impulse (refer Figure 5.4.25)
The second impulse is summarised in Figures 5.4.28 to 5.4.30. The downstream concentration again rises sharply to reach its maximum at the same time as the upstream concentration. The penetration at this time is 12% compared with approximately 4% immediately before and after the impulse. There is no indication of any time delay between upstream and downstream concentrations.

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The third impulse is shown in Figures 5.4.31 to 5.4.33. The downstream response is split over two minutes because the upstream impulse occurs just before the end of the first one minute counting period. This leads to the very high calculated penetration of 45% at time 133 minutes (see Figure 5.4.33), corresponding to the peak in the downstream count which occurs one minute after that in the upstream count. A more
realistic value for the penetration is obtained by taking the ratio of the sum of the
downstream counts for times 132, 133 minutes to the upstream count at time 132 minutes.
This gives a penetration of 22%.

Figure 5.4.31
Variation in penetration for the third upstream impulse

Figure 5.4.32
Downstream response to the third upstream impulse (refer Figure 5.4.31)
The sixth impulse is shown in Figures 5.4.34 to 5.4.36. The peaks in upstream and downstream concentration again occur simultaneously, with a penetration at this time of over 30%. As upstream and downstream concentrations decay the penetration gradually falls, indicating that some time delay between upstream and downstream concentrations is again occurring. However, the penetration falls to 12% immediately after the impulse before gradually decreasing.
After several upstream impulses had been performed a step change in challenge concentration was introduced (see Figure 5.4.37). The downstream response follows the same pattern as was seen for step changes in challenge concentration in earlier tests, that is, there is an apparently exponential rise in the downstream concentration (see Figure 5.4.38) and the penetration (see Figure 5.4.39).
Figure 5.4.37
Variation in upstream concentration for the step increase after six upstream impulses

Figure 5.4.38
Downstream response to the step increase in upstream concentration (refer Figure 5.4.37)
Figure 5.4.39
Penetration in response to the step increase in upstream concentration
(refer Figures 5.4.37 and 5.4.38)

After maintaining the high upstream concentration after the step increase for approximately 35 minutes the particle feed was switched off (see Figure 5.4.40) The downstream concentration gradually reduced after an initial sudden fall (see Figure 5.4.41) This response is similar to that seen in earlier tests when the particle feed was switched off.

Figure 5.4.40
Variation in upstream concentration after switching off the particle feed
The third challenge with 0.46 micron particles on the third prefilter sample was concluded with a final upstream impulse (see Figure 5.4.42). The downstream response shows a peak at the same time as the upstream concentration and no significant time delay between upstream and downstream concentrations until the upstream level has reduced to zero.
Summary of findings for the third challenge with 0.46 micron particles on the third prefilter sample

Each time the filter experiences an impulse in the upstream concentration there is an instantaneous response downstream of the filter with a corresponding increase in penetration. As the upstream concentration reduces after an impulse the delay between upstream and downstream concentrations becomes apparent once more.

In response to a step increase in upstream concentration the penetration increases in what appears to be an exponential manner, indicating that there is a time delay between upstream and downstream concentrations. This also shown by the gradual decay in downstream concentration once the particle feed to the filter is switched off.

The penetration at the time of each impulse is not constant, the range being 12% to 33%.
5.5 CONCLUSIONS

Three samples of the 0.45 micron rated prefiltre have been tested with a range of particle sizes under conditions of varying challenge concentration. It was found that for 0.46 micron particles there is a significant time delay between upstream and downstream concentrations. Of those particles which penetrated the filter, a proportion did so with no time delay, (direct penetration) while others experienced a residence time within the filter resulting in a delay between upstream and downstream concentrations (delayed penetration).

The time delay between upstream and downstream concentrations was particularly noticeable in response to a step increase in upstream concentration. The downstream concentration in response to such a change upstream would increase in what appeared to be an exponential manner suggesting that for those particles which are delayed in penetrating the filter there is a distribution of residence times within the filter.

The time delay was also noticeable when the particle feed was switched off. The downstream concentration decayed at a much slower rate than the upstream concentration, often requiring hours to reach the residual concentration achieved before commencing testing on the filter, compared with minutes for the upstream concentration.

In the event of impulses in the upstream concentration no delayed penetration was observed and there was an increase in the level of penetration. Delayed penetration was observed after the upstream concentration had started to reduce after the impulse.

Tests on the prefiltre were also performed with 0.55, 0.65 and 0.94 micron particles. No time delay between upstream and downstream concentrations was observed for 0.65 or 0.94 micron particles. There was evidence of some time delay with 0.55 micron particles, but less than that for 0.46 micron particles.

The long decay time for the downstream concentration on switching off the particle feed was only observed for 0.46 micron particles.
The increase in penetration in the event of upstream impulses was observed up to 0.65 micron but not for 0.94 micron particles.

It is clear that there is a relationship between penetration and particle size but it is not possible to quantify the relationship because penetration is not a constant for each particle size. These tests were designed to examine the time dependence of filter performance rather than the effect of particle size on steady state filter efficiency. In order to correlate efficiency with particle size identical tests would have to be performed for each size so that the time delay and the effect on penetration of challenge concentration can be accounted for.
CHAPTER 6

RESULTS OF TESTING THE 0.4 AND 0.45 MICRON RATED MEMBRANE FILTERS

6.1 Introduction

6.2 Results of tests on the 0.45 micron membrane filter

6.3 Results of tests on the 0.4 micron membrane filter

6.4 Conclusions
6.1 INTRODUCTION

In Chapter 5 the results of the steady water flow tests on the 0.45 micron rated prefilter were presented. In this chapter, the results of the steady water flow tests on 0.4 and 0.45 micron rated membrane filters are presented.

Two filters were tested. One was rated at 0.45 micron and the other at 0.4 micron. Two samples of the 0.45 micron rated filter were tested but only one of the 0.4 micron rated filter.

The purpose of these tests was to compare and contrast the performance of the prefilter with that of the membrane filters in an attempt to determine the mechanisms of operation of all three filters.

It was found that for both membrane filters most of the penetration was direct, that is, there is little delayed penetration at any particle size. Also, there is none of the long term delayed penetration such as was observed for the prefilter with 0.46 micron particles when the particle feed to the filter was switched off.

At most particle sizes there was not any delayed penetration for either of the membrane filters tested. The exceptions were for the 0.45 micron filter with 0.46 micron particles and for the 0.4 micron filter with 0.31 micron particles.

In some cases, the occurrence of impulses in the upstream concentration results in increased penetration for the membrane filters. However, the increase in penetration is less significant than that observed for the prefilter and the increase is not observed as consistently as for the prefilter.
6.2 RESULTS OF TESTS ON THE 0.45 MICRON MEMBRANE FILTER

Two samples of the 0.45 micron rated membrane filter were tested with a range of particle sizes and challenge concentrations. The results for each sample are presented below.

6.2.1 First 0.45 micron membrane filter sample

This sample of the filter was tested with three different particle sizes. The first loading of the filter was with 0.37 micron particles followed by 0.31 micron and finally 0.46 micron.

Challenge with 0.37 micron particles on the first 0.45 micron membrane filter sample

The start of the challenge of this sample with 0.37 micron particles is shown in Figures 6.2.1 to 6.2.3. It is clear that very few particles penetrate the filter (Figure 6.2.2) and that those particles which do penetrate the filter do so without any significant time delay.

![Figure 6.2.1](image)

**Figure 6.2.1**
Upstream concentration at the start of the challenge with 0.37 micron particles on the first 0.45 micron membrane filter sample
Figure 6.2.2
Downstream response (refer Figure 6.2.1)

Figure 6.2.3
Penetration at the start of the challenge with 0.37 micron particles on the first 0.45 micron membrane filter sample (refer Figures 6.2.1 and 6.2.2)

The large downstream variation (see Figure 6.2.2) is due to the low concentration. The relatively high calculated penetration in the first 27 minutes (see Figure 6.2.3) is due to the low upstream concentration, resulting in the downstream residual concentration having a more significant effect on the ratio of downstream to upstream concentration.
than would be the case at high upstream concentration.

Figure 6.2.4 shows the variation in upstream concentration for the remainder of the challenge with 0.37 micron particles. This is shown more clearly for the first 400 minutes in Figure 6.2.5.

![Upstream concentration graph](image)

**Figure 6.2.4**
Variation in upstream concentration for the remainder of the challenge with 0.37 micron particles on the first 0.45 micron membrane filter sample

![Upstream concentration detail graph](image)

**Figure 6.2.5**
Detail of the upstream concentration up to 400 minutes (refer Figure 6.2.4)
The downstream response is shown in Figure 6.2.6 and the penetration in Figure 6.2.7. Because of the very low downstream concentration the variation appears to be high. The calculated penetration is relatively high during the first 400 minutes until the upstream concentration rises sharply, at which point the calculated penetration falls as the effect of the downstream residual concentration is reduced.

Figure 6.2.6
Downstream response (refer Figures 6.2.4 and 6.2.5)

Figure 6.2.7
Variation in penetration (refer Figures 6.2.4 to 6.2.6)
Challenge with 0.31 micron particles on the first 0.45 micron membrane filter sample

The start of the challenge with 0.31 micron particles on the first 0.45 micron membrane filter sample is shown in Figures 6.2.8 to 6.2.10. Inspection of Figures 6.2.8 and 6.2.9 suggests that there is no time lag between upstream and downstream concentrations. The appearance of Figure 6.2.10 suggests a time delay between upstream and downstream concentrations may be present because of the slightly exponential shape of the penetration curve. However, if the relatively large values between times 20 and 22 minutes were not present the curve would appear more like a step change, indicating no time lag.

In fact, Figure 6.2.9 shows that at this time the downstream count is varying widely while the upstream is falling slightly. This variation downstream may be sufficient to distort the picture such that the penetration curve appears to indicate a slight delay, or it may be that there is some delay between upstream and downstream concentrations.

![Graph](image)

**Figure 6.2.8**

Variation in upstream concentration at the start of the challenge with 0.31 micron particles on the first 0.45 micron membrane filter sample
Figure 6.2.9
Downstream response (refer Figure 6.2.8)

Figure 6.2.10
Variation in penetration (refer Figures 6.2.8 and 6.2.9)

The remainder of the challenge with 0.31 micron particles on the first 0.45 micron membrane filter is shown in Figures 6.2.11 to 6.2.14. Figures 6.2.11 and 6.2.12 do not indicate any time delay between the upstream and downstream concentrations, but it should be noted that the data represent mean values every 10 minutes so that any slight delay would be hidden.
Figure 6.2.13 shows the penetration relating to the first part of the data shown in Figures 6.2.11 and 6.2.12. From this, and from Figure 6.2.14 for the last part of the data in Figures 6.2.11 and 6.2.12, it is clear that if there is any time delay it is considerably less than 10 minutes.

**Figure 6.2.11**

Variation in upstream concentration for the remainder of the challenge with 0.31 micron particles on the first 0.45 micron membrane filter sample

**Figure 6.2.12**

Downstream response (refer Figure 6.2.11)
The effect of switching off the particle feed is shown in Figures 6.2.14 and 6.2.15. The downstream concentration decays at the same rate as the upstream, indicating that there is no long term delay between upstream and downstream concentrations.
Figure 6.2.15
Reduction in upstream concentration on switching off the particle feed

Figure 6.2.16
Downstream response to switching off the particle feed (refer Figure 6.2.15)
Challenge with 0.46 micron particles on the first 0.45 micron membrane filter sample

The first 28 minutes of the challenge with 0.46 micron particles on the first 0.45 micron membrane filter sample is shown in Figures 6.2.17 to 6.2.19. Comparison of the upstream and downstream concentrations does not indicate any time delay, and this is supported by the penetration curve. However, it does appear that the penetration is higher at high upstream concentration.

Figure 6.2.17
Variation in upstream concentration for the first 28 minutes of the challenge with 0.46 micron particles on the first 0.45 micron membrane filter sample
The remainder of the challenge with 0.46 micron particles on the first 0.45 micron membrane filter sample is shown in Figures 6.2.20 and 6.2.21. Although the data are presented as 10 minute averages, it is clear that there is no significant time delay between upstream and downstream concentrations, both decreasing at the same rate when the particle feed is switched off.
Figure 6.2.20
Variation in upstream concentration to the end of the challenge with 0.46 micron particles on the first 0.45 micron membrane filter sample

Figure 6.2.21
Downstream response (refer Figure 6.2.20)
6.2.2 Second 0.45 micron membrane filter sample

The second sample of the 0.45 micron membrane filter was only tested with 0.46 micron particles. The tests were split into two separate challenges.

Figures 6.2.22 to 6.2.25 show the start of the first challenge with 0.46 micron particles. The peak in the upstream concentration is matched by a corresponding peak downstream with no sign of any time delay. The downstream response after the peak is shown in Figure 6.2.24. It is clear that the downstream concentration rises significantly above the residual concentration only at the time of the upstream peak.

The upstream peak produces a peak in the penetration (see Figure 6.2.25). The high penetration levels in Figure 6.2.25 from 10 to 40 minutes correspond to low challenge concentrations and are therefore due to the effect of the downstream residual concentration. When the upstream concentration rises towards 300,000 particles per 100 ml the penetration is less than 0.05%, compared with almost 0.5% at the occurrence of the peak.

![Graph](image)

**Figure 6.2.22**

Variation in upstream concentration for the first 80 minutes of the first challenge with 0.46 micron particles on the second 0.45 micron membrane filter.
Figure 6.2.23
Downstream response (refer Figure 6.2.22)

Figure 6.2.24
Downstream response with scale adjusted (refer Figure 6.2.23)
Figures 6.2.26 to 6.2.28 show the remainder of the first challenge with 0.46 micron particles on the second sample of the 0.45 micron membrane filter. As the upstream rises from 600,000 to more than 1,300,000 particles per 100 ml the downstream concentration does rise, but to a much lesser extent than at the start of the challenge (compare Figures 6.2.23 and 6.2.27). The penetration at the time of the peak is less than 0.01% compared with nearly 0.5% at the time of the upstream peak at the start of the test, and only rises once the upstream concentration falls and the effect of the downstream residual concentration becomes dominant. The downstream concentration is only significantly higher than the residual concentration at the time of the peak in Figure 6.2.26. Even at 600,000 particles per 100 ml upstream of the filter the downstream concentration remains at the residual concentration.
Figure 6.2.26
Variation in upstream concentration to the end of the first challenge with 0.46 micron particles on the second 0.45 micron membrane filter sample

Figure 6.2.27
Downstream response (refer Figure 6.2.26)
The second challenge with 0.46 micron particles on the second sample of the 0.45 micron membrane filter involved a number of impulses in the upstream concentration. Figure 6.2.29 shows an upstream impulse to 1,200,000 particles per 100 ml, similar to that shown in Figure 6.2.22. A peak in the downstream concentration is again observed with no apparent time delay. The penetration curve shown in Figure 6.2.31 shows that at the time of the upstream impulse the penetration is almost 0.1%, significantly higher than that recorded in Figure 6.2.28 after a gradual increase in upstream concentration. Comparison of Figures 6.2.30 and 6.2.23 shows that the downstream response to the impulse is of a similar order of magnitude to that performed at the start of the first challenge with 0.46 micron particles on this filter sample. The high calculated penetration values after the peak in Figure 6.2.31 are due to the effect of the downstream residual concentration after the upstream concentration has reduced to low levels.
Figure 6.2.29
Variation in upstream concentration for the first impulse of the second challenge with 0.46 micron particles on the second 0.45 micron membrane filter sample

Figure 6.2.30
Downstream response (refer Figure 6.2.29)
The second and third impulses in the second challenge with 0.46 micron particles on the second 0.45 micron membrane filter sample are shown in Figures 6.2.32 to 6.2.35. The penetration curves are not included as these do not show the increase in penetration in the event of each impulse because of the effect of the high residual downstream concentration. However, Figures 6.2.32 and 6.2.33 show that the upstream impulse does result in a downstream peak of similar magnitude to that shown in Figure 6.2.30. In this case the penetration is 0.12%.
Figure 6.2.32
Variation in upstream concentration for the second impulse of the second challenge with 0.46 micron particles on the second 0.45 micron membrane filter sample

Figure 6.2.33
Downstream response (refer Figure 6.2.32)

The third impulse is shown in Figures 6.2.34 and 6.2.35. The upstream concentration at the peak of the impulse is less than that in previous impulses and the corresponding downstream response is also less. In fact, the penetration is only 0.05% which is considerably higher than that recorded for the more gradual increase to 1,300,000
particles per 100 ml upstream in Figures 6.2.26 to 6.2.28, but less than that for the other impulses, suggesting that the concentration at the peak of the impulse determines not only the downstream concentration, but also the degree of penetration.

Figure 6.2.34
Variation in upstream concentration for the third impulse

Figure 6.2.35
Downstream response (refer Figure 6.2.34)
6.2.3 Summary of findings for the 0.45 micron membrane filter

There is little evidence of any time delay between upstream and downstream concentrations for the 0.45 micron membrane filter although it is possible that a slight delay was in operation with 0.31 micron particles. If there is any delay it is insignificant when compared with that observed for the prefilter.

The efficiency of the filter was shown to be dependent on the challenge particle size but it is not possible to quantify the relationship from the data presented here as efficiency has also been shown to be dependent on challenge concentration, and the tests were not all carried out using the same concentrations.

The work on the second sample of this filter considered the effect of concentration on efficiency (or penetration) for 0.46 micron particles. It was found that not only did the concentration effect the efficiency, but also the manner in which the concentration was arrived at.

Several impulses in the upstream concentration were introduced to the filter in the same way as for the third sample of the 0.45 micron prefilter (see Chapter 5). In every case an upstream impulse resulted in a significant increase in penetration compared with the same upstream concentration at steady state. The degree of penetration is dependent on the concentration achieved by the impulse.
6.3 RESULTS OF TESTS ON THE 0.4 MICRON MEMBRANE FILTER

Only one sample of this filter was tested. Three challenge particle sizes were used: 0.37, 0.46 and 0.31 micron. The results for each particle size are presented below.

6.3.1 Challenge with 0.37 micron particles

The challenge with 0.37 micron particles on the 0.4 micron membrane filter is summarised in Figures 6.3.1 to 6.3.3. Comparison of Figures 6.3.1 and 6.3.2 does not indicate the presence of any time delay between upstream and downstream concentrations, although it should be noted that the data are 10 minute averages. The penetration (see Figure 6.3.3) also gives no indication of any time delay.

![Figure 6.3.1]

**Figure 6.3.1**

Variation in upstream concentration for the challenge with 0.37 micron particles on the 0.4 micron membrane filter
The decrease in upstream and downstream counts on switching off the particle feed is shown in Figures 6.3.4 and 6.3.5 respectively. As far as can be judged, there is no time delay between upstream and downstream concentrations.
6.3.2 Challenge with 0.46 micron particles on the 0.4 micron membrane filter

The 0.46 micron challenge is summarised in Figures 6.3.6 to 6.3.8. It should be noted that the large and varying penetration values after 40 minutes in Figure 6.3.8 are due to the downstream residual concentration once the particle feed has been switched off.
Figure 6.3.6
Variation in upstream concentration for the 0.46 micron challenge on the 0.4 micron membrane filter

Figure 6.3.7
Downstream response (refer Figure 6.3.6)
The first part of the challenge is shown in more detail in Figures 6.3.9 to 6.3.11. It is clear that there is no time delay between upstream and downstream concentrations. The peak in the penetration (see Figure 6.3.11) does not correspond to the peak in the challenge concentration but is due to the background level of particles before commencing the challenge.

Figure 6.3.9
Variation in upstream concentration for the first 30 minutes of the challenge with 0.46 micron particles on the 0.4 micron membrane filter
The step change in upstream concentration after 33 minutes is shown in detail in Figure 6.3.12 and it is clear by comparison of this with Figure 6.3.13 that there is no time delay between upstream and downstream concentrations. However, the increase in challenge concentration does produce an increase in the level of penetration (see Figure 6.3.14).
Figure 6.3.12
Variation in upstream concentration for the step increase in concentration during the challenge with 0.46 micron particles on the 0.4 micron membrane filter

Figure 6.3.13
Downstream response (refer Figure 6.3.12)
Figure 6.3.14
Variation in penetration (refer Figures 6.3.12 and 6.3.13)
6.3.3 Challenge with 0.31 micron particles on the 0.4 micron membrane filter

The 0.31 micron challenge on the 0.4 micron membrane filter is summarised in Figures 6.3.15 to 6.3.17.

Figure 6.3.15
Variation in upstream concentration for the 0.31 micron challenge on the 0.4 micron membrane filter

Figure 6.3.16
Downstream response (refer Figure 6.3.15)
The first 100 minutes of the challenge with 0.31 micron particles are shown in detail in Figures 6.3.18 to 6.3.20. The large peak at time 5 minutes in Figure 6.3.18 produces a peak in the downstream concentration (see Figure 6.3.19 which appears to be superimposed on a gradual increase to a steady state downstream concentration. Clearly, once the upstream concentration has reached steady state, the downstream is still increasing, which is shown by the shape of the penetration plot (see Figure 6.3.20). This suggests that there is some time delay between upstream and downstream concentrations.
Figure 6.3.18
Variation in upstream concentration for the first 100 minutes of the challenge with 0.31 micron particles

Figure 6.3.19
Downstream response (refer Figure 6.3.18)

Figure 6.3.21 shows a drop in challenge concentration to almost zero, followed by a step increase. The shape of the downstream plot (see Figure 6.3.22) again indicates that a small time delay may be present. Note that in Figure 6.3.23 the penetration between 145 and 182 minutes has been plotted as zero as the upstream is very low and the downstream represents the residual concentration.
Figure 6.3.20
Variation in penetration (refer Figures 6.3.18 and 6.3.19)

Figure 6.3.21
Variation in upstream concentration from 120 to 220 minutes of the challenge with 0.31 micron particles on the 0.4 micron membrane filter
In Figures 6.3.24 to 6.3.26 there is no indication of any time delay when the upstream concentration is increased or decreased. In addition, the penetration shows no indication of a time delay (see Figure 6.3.26). Note again that the penetration has been plotted as zero when the upstream is zero.
Figure 6.3.24
Variation in upstream concentration from 260 to 300 minutes of the challenge with 0.31 micron particles on the 0.4 micron membrane filter

Figure 6.3.25
Downstream response (refer Figure 6.3.24)

Figure 6.3.27 examines the downstream response to the drop in upstream concentration occurring after 281 minutes (see Figure 6.3.24). It is clear from this figure that some delay is present as the downstream concentration at 284 minutes is well above the downstream residual concentration, which is of the order of 20 per 100 ml. However, the proportion of particles experiencing delay, and the length of the delay are small.
Figure 6.3.26
Variation in penetration (refer Figures 6.3.24 and 6.3.25)

Figure 6.3.27
Decay in downstream concentration when the upstream concentration is reduced to zero, for 0.31 micron particles on the 0.4 micron membrane filter (refer Figures 6.3.24 and 6.3.25)

Figures 6.3.28 and 6.3.29 show the upstream and downstream concentrations when the particle feed is switched off. Again there is the indication of a small time delay between upstream and downstream concentrations.
Figure 6.3.28
Variation in upstream concentration on switching off particle feed

Figure 6.3.29
Downstream response to switching off particle feed (refer Figure 6.3.28)
6.4 CONCLUSIONS

Neither of the membrane filters tested in this programme of work showed the long term delay between upstream and downstream concentrations which was observed for the prefilter. However, there were similarities observed between the operation of the prefilter and the membranes.

There was no evidence of any short term delay between upstream and downstream concentrations for the 0.45 micron membrane filter at any particle size apart from 0.46 micron. At this size it was not possible to be certain whether or not there was any short term time delay but some of the data suggested that some delay may be present.

For the 0.4 micron membrane filter there was no time delay between upstream and downstream concentrations at 0.37 or 0.46 micron but there was significant delay at 0.31 micron. Although this delay was significant it was considerably less than that observed for the prefilter and the proportion of particles experiencing delay was also less.

For both of the membrane filters it was shown that the penetration was dependent on challenge concentration. The efficiency of the 0.45 micron membrane filter showed considerable dependence on challenge concentration for 0.46 micron particles. In addition, in the event of impulses in the challenge concentration it was found that the efficiency was considerably less than under the same steady state challenge concentrations.

For the 0.4 micron membrane filter it was found that with 0.46 micron particles an increase in the challenge concentration led to a decrease in efficiency. No upstream impulses were performed at this particle size. However, for 0.31 micron particles an impulse in the upstream concentration resulted in a momentary decrease in efficiency.
CHAPTER 7

DISCUSSION OF RESULTS

7.1 Introduction

7.2 Analysis of results of tests on the 0.45 micron prefilter

7.3 Summary of findings for the 0.45 micron prefilter

7.4 Summary of findings for the membrane filters

7.5 Conclusions
7.1 INTRODUCTION

In Chapters 4 to 6 the results were presented with some explanation. In this chapter the results of the tests on the 0.4 and 0.45 micron filters are analysed and discussed in detail.

The filter of most interest is the 0.45 micron prefilter, as it is this filter which shows most evidence of residence time distributions for particles within the filter. This filter is considered first, followed by comparison of its operation with the 0.4 and 0.45 micron membrane filters. The main findings regarding the operation of the prefilter which are discussed in this chapter are:

*For 0.46 micron particles:*

1. There is a distribution of residence times for particles within the filter.
2. The penetration is a function of time.
3. The response of the filter to a step increase in upstream concentration is that of a first order dynamic system.
4. The time constant for this first order response is 5 to 10 minutes.
5. When the upstream concentration is maintained constant the downstream concentration continues to increase, even after several hours. This increase is clearly not caused by the process in (2) above.
6. When the challenge to the filter is stopped the downstream concentration decays over a considerable time. This decay can be represented by two first order processes, the time constant for one being considerably longer than that for the other. These two processes are the ones described in (2) and (5) above.
7. At any time, t, the downstream concentration will be a combination of the following:
   (a) direct penetration of particles challenging the filter at time t
   (b) delayed penetration (short term) due to the first order response of the filter
(time constant 5 to 10 minutes)
c) delayed penetration (long term) due to a first order response of considerably longer time constant.

8. Under certain conditions, particularly the presence of a large peak in the upstream concentration, there is a shift from delayed penetration ((b) above) to direct penetration ((a) above), resulting in an increase in the total penetration.

Some of the above effects were also observed for other particle sizes, but they were most apparent at 0.46 micron.

*For the 0.4 and 0.45 micron membrane filters:*

9. For certain particle sizes a small degree of short-term delayed penetration is observed with the membrane filters. However, no long-term delayed penetration is observed with the membrane filters at any particle size.

10. In some cases the membrane filters showed an increased penetration in the event of an impulse in the upstream concentration, but to a much lesser degree than with the prefilter.
7.2 ANALYSIS OF RESULTS OF TESTS ON THE 0.45 MICRON PREFILTER

Three samples of this filter were tested. The results for each sample are analysed separately with a summary of the main findings in section 7.2.4.

7.2.1 First 0.45 micron prefilter sample

The first sample of the prefilter was tested with two particle sizes. The test results for 0.46 micron particles were presented in Chapter 5, Section 5.2, Figures 5.2.1 to 5.2.15.

Figure 7.2.1 shows the change in penetration over the first 45 minutes of loading with 0.46 micron particles. Consideration of this figure together with the results in Figures 5.2.1 and 5.2.2 shows that there is a residence time within the filter for some of the particles which penetrate the filter, the others penetrating immediately.

![Penetration for first 45 minutes of loading](image)

**Figure 7.2.1**
Penetration for first 45 minutes of loading

The shape of the curve in Figure 7.2.1 is similar to the response of a first order dynamic system to a step change in forcing function.\(^\text{72}\)

If a first order dynamic system experiences a step change in forcing function the response is given by equation (7.1).
where $Y(t)$ is the deviation from its steady state value of the output of the system at time $t$, $A$ is the magnitude of the step change and $\tau_c$ is the time constant for the rise in concentration.

In the case of a filter $A$ is not the magnitude of the increase in upstream concentration because over 90% of the increase in upstream concentration does not penetrate the filter. In this case $A$ is the achieved steady state increase in downstream concentration, that is the value of $Y(t)$ as $t$ approaches infinity.

Figure 7.2.2 shows a comparison between the experimental data for the increase in penetration with time and calculated values for a first order system with a time constant of 5 minutes.

![Figure 7.2.2](image)

**Figure 7.2.2**

First order response for first 45 minutes ($\tau_c = 5$ minutes)

It is clear from Figure 7.2.2 that the experimental data and the calculated values for a first order system are in close agreement.
It is important that an understanding of the whole life of the filter be gained, not just the initial performance. Figure 7.2.3 shows a comparison between the same first order curve and the data for the penetration up to 98 minutes after the start of loading.

![Figure 7.2.3](image)

**First order response for first 98 minutes**

The figure shows that after about 50 minutes the data no longer fits the curve because the penetration is still increasing. It is clear that the response of the filter cannot be completely described by the first order process with a 5 minute time constant but that this description is adequate for the first 50 minutes.

During the last few minutes of the results shown in Figures 5.2.1 to 5.2.3 there is a reduction in upstream concentration (a negative step change) which produces a reduction in the downstream concentration. However, it is clear from the shape of the penetration plot at this time that the upstream and downstream counts are not falling at the same rate. In fact, in the time that the upstream concentration falls by 65%, the downstream falls by 45%. Again there is evidence of an immediate response in the downstream concentration to the upstream, but also some delay.

A similar situation is seen in Figures 5.2.4 to 5.2.8 where the upstream and downstream concentrations are reducing. The downstream concentration takes 50 minutes longer than the upstream to arrive at steady state.
Figures 5.2.9 to 5.2.11 show a step increase in upstream concentration. The data are plotted every 10 minutes, and on this scale no delay between upstream and downstream is visible. However, it is noticeable that the penetration at this time increases, unlike the other changes in concentration recorded for this filter (except the increase from zero challenge) where the steady state penetration was the same for each upstream concentration. Clearly, with a change in penetration and a lack of time delay between upstream and downstream concentrations, a change in performance has occurred.

After this step increase in concentration the challenge was reduced to zero. From Figures 5.2.12 to 5.2.15 it can be seen that the downstream concentration takes considerably longer than the upstream to reduce to zero.

The downstream decay is considered here in more detail. Figure 7.2.4 shows what appears to be an exponential decrease in downstream concentration.

If the downstream decay is exponential, that is, first order, equation (7.2) would apply.

\[
\frac{D(t)}{D(0)} = \exp(-t/\tau_d)
\]  
(7.2)
where $D(t)$ is the downstream concentration at time $t$, $D(0)$ the downstream concentration immediately after switching off the challenge and $\tau_d$ the time constant for the decay.

$$\ln\{D(t)\} = \ln\{D(0)\} - \frac{t}{\tau_d}$$  \hspace{1cm} (7.3)

The plot of the logarithm of the downstream concentration against time is shown in Figure 7.2.5. Note that for the first few counts the upstream concentration was still fairly high, so the downstream concentration used for Figure 7.2.5 is given by equation (7.4):

$$D'(t) = D(t) - p_s U(t)$$  \hspace{1cm} (7.4)

where $D'(t)$ is the value of downstream concentration used in Figure 7.2.5, $p_s$ the steady state penetration before switching off the challenge and $U(t)$ the upstream concentration at time $t$.

![Figure 7.2.5](image)

**Figure 7.2.5**

Natural logarithm of downstream decay

Figure 7.2.5 shows what appears to be a reasonable straight line relationship. Figure 7.2.6 is a plot comparing the decay in the log of the downstream concentration with a regression line. The agreement is acceptable although it would be better if the data for the first 50 minutes were not included as during this time the decay appears to be faster. The calculated value for $\tau_d$ is 102 minutes, but this would be longer if the first 50 minutes
were not included. This decay is clearly not caused by the same first order process as that observed for the step increase in upstream concentration at the start of loading. The suggestion is that more than one process is in operation.

![Image of a graph showing regression to find time constant for decay.](image)

**Figure 7.2.6**

Regression to find time constant for decay

In Figure 7.2.7 an exponential decay with time constant 102 minutes is compared with the decay in downstream concentration. The agreement between the curves is good and would be better still if the regression were carried out from 50 minutes onwards (see Figure 7.2.6). Figure 7.2.7 shows clearly that the rate of decay is faster for the first 50 minutes than after this point in time.
The first prefilter sample was also tested with 0.65 micron particles (see Figures 5.2.16 to 5.2.18). There was a slight indication of delay between upstream and downstream concentrations after the large peak at 2 minutes, the downstream taking a few minutes longer than the upstream to reduce. Furthermore, at the occurrence of the peak in the upstream concentration, the corresponding peak in the downstream concentration causes an increase in penetration. Note, the high penetration after the peak is due to the downstream reducing more slowly than the upstream so that the ratio of downstream to upstream increases. It can therefore be concluded that the peak in upstream concentration causes a momentary increase in the penetration.

For the rest of the test with 0.65 micron particles there was no evidence of any delayed penetration, or any change in the penetration.

**Summary of analysis of first prefilter sample**

There is a distribution of residence times within the filter for particles of the same size, 0.46 micron. Approximately 6% of the 0.46 micron particles in the challenge penetrate...
the filter. Of this 6% a proportion penetrate immediately (direct penetration), that is
with no residence time within the filter. Others require a certain time for penetration
(delayed penetration)

The delayed penetration appears to consist of more than one process. When a step
increase in upstream concentration is introduced the penetration increases in a first order
manner with a time constant of 5 minutes. However, when the upstream concentration
is maintained constant the penetration continues to increase at a rate considerably higher
than that which would be seen if the first order process with 5 minute time constant was
the only process in operation. Further evidence of more than one process of delayed
penetration is provided by the decay in downstream concentration when the challenge
is stopped. The decay appears to be first order with a time constant of over 100 minutes,
compared with 5 minutes in response to a step increase in concentration. Also, the initial
downstream decay occurs at a considerably faster rate than that after about the first
50 minutes.

Throughout the test with 0.46 micron particles there is evidence of direct penetration,
delayed penetration with a short time constant and delayed penetration with a long time
constant.

With 0.65 micron particles as challenge there was little evidence of delay between
upstream and downstream concentrations. One important observation was that the
occurrence of a peak in the challenge concentration resulted in an increase in penetration,
that is, the resulting increase in downstream concentration was proportionately larger
than that in the upstream concentration.
7.2.2 Second 0.45 micron prefiltter sample

The tests on this sample of the prefiltter were designed to determine whether any of the effects noted from the tests on the first sample were apparent at other particle sizes.

This sample was tested with two particle sizes. The first of these was 0.94 micron (see Chapter 5, Section 5.3, Figures 5.3.1 to 5.3.11). All the penetration appears to be direct for this particle size, that is, there is no evidence of a time delay between upstream and downstream concentrations.

It is clear that for this particle size an increase in upstream concentration causes a corresponding increase downstream, but no increase in penetration (see Figure 5.3.3).

The second particle size with which this sample was tested was 0.55 micron. The results for the first part of this test are shown in Figures 5.3.12 to 5.3.20. It is clear from these figures that there is some time delay present between upstream and downstream concentrations. Figure 7.2.8 shows that the increase in penetration at the start of the test is similar to the response of a first order system to a step change in forcing function (see Figures 5.3.12 to 5.3.14). In this case the time constant is only 3 minutes compared with 5 minutes for the 0.46 micron challenge. Not only is the time constant less but the proportion of the particles which penetrate the filter that experience this residence time is considerably less than with the 0.46 micron particles, that is, more of the penetration is direct than delayed.
Figure 7.2.8
First order response for first 35 minutes of loading ($\tau_1 = 3$ min)

Figure 5.3.17 shows the penetration up to 160 minutes. It has been noted above that there is evidence of a short time delay between upstream and downstream concentration but this figure shows no evidence of any long time delay, that is there is no gradual increase in penetration over a long time period. If anything, the penetration is decreasing after 160 minutes. However, this may be due to the long-term reduction in downstream concentration following the reduction of upstream concentration; in other words, the gradual reduction in penetration could itself be evidence of a long-term delay between upstream and downstream concentrations.

This is further supported by Figures 5.3.18 to 5.3.20, where the changes in penetration indicate that upstream and downstream concentrations are varying at different rates.

Summary of analysis of second prefilter sample

There is no evidence of any delayed penetration with 0.94 micron particles. Furthermore, the peak in the challenge concentration did not produce any increase in penetration, that is, the increase in downstream concentration was proportionately the same as the increase in upstream concentration.
With 0.55 micron particles there is some evidence of delayed penetration. The most dominant form of penetration is direct, but a significant proportion of the particles which penetrate experience a distribution of residence times. In fact, the response of the filter appears to be first order, but with a shorter time constant ($\tau$) of 3 minutes, compared with 5 minutes for 0.46 micron particles. This difference in time constants is not the important point. The important conclusion is that some of the particles do undergo delayed penetration, that is they have a residence time within the filter.

There is also some evidence of long-term time delay for 0.55 micron particles. It seems that for 0.55 micron particles the three forms of penetration noted for 0.46 micron particles are in operation but that direct penetration is dominant.
7.2.3 Third sample of the 0.45 micron prefilter

The tests on the first two samples of this filter have shown that the most critical size for delayed penetration to occur is 0.46 micron. The tests on the third sample of the prefilter were designed to determine when the three forms of penetration are most apparent, to quantify them, and to arrive at the mechanisms of filtration in operation. All the tests on this sample were carried out with 0.46 micron particles.

Three separate tests were performed on this sample of the filter. (Chapter 5, Section 5.4).
In these tests the effects of the following on penetration are considered:

1. Step changes in the upstream concentration
2. Impulses in the upstream concentration
3. Maintenance of a constant upstream concentration
4. Switching off the particle feed

First challenge with 0.46 micron particles

The results of the first challenge with 0.46 micron particles are summarised in Figures 5.4.1 to 5.4.3. The aim of the test was to investigate the effect of step changes in the upstream concentration.

The test was carried out in three main parts, the concentration changing between parts in an approximation to a step change.

From Figures 5.4.4 and 5.4.5 it is clear that the early peak in the upstream concentration causes a corresponding peak in the downstream concentration, with no apparent time delay. The penetration curve (Figure 5.4.6) shows a peak at this time of 4%, which appears to be superimposed on a gradually increasing curve.

The period of the test before the first step increase in upstream concentration after 26 minutes is shown in more detail in Figures 7.2.9 to 7.2.11. It is clear from these figures that the peaks occurring at 4 and 15 minutes both cause immediate peaks in the downstream concentration, but the peak at 4 minutes produces a penetration of 4% while the one at 15 minutes does not produce an increase in penetration. After the peak at
4 minutes the upstream concentration is reducing faster than the downstream such that the penetration at this time is increasing. In fact, from the start of the test the penetration appears to increase exponentially, with the upstream peak at 4 minutes causing a peak of 4% superimposed on the curve.

**Figure 7.2.9**
Upstream concentration at the start of the first challenge with 0.46 micron particles on the third prefilter sample

**Figure 7.2.10**
Downstream response (refer Figure 7.2.9)
Figure 7.2.11
Variation in penetration (refer Figures 7.2.9 and 7.2.10)

Note that the high levels of penetration after the upstream peak at 15 minutes and before the rise in upstream concentration at 26 minutes are due to the upstream concentration having reduced to very low levels while the downstream remains high for some time.

The penetration for the first period of this test is considered in more detail in Figure 7.2.12. The penetration is compared with the response of a first order system with a time constant, $\tau$, of 5 minutes. The agreement is good, suggesting that the system is first order, with the exception of the peak in the upstream concentration after 4 minutes which superimposes a peak on the penetration curve. Note that the other peaks which appear to be superimposed on the penetration curve are due to the variation in downstream concentration at low upstream concentration.
Figure 7.2.12

Variation in penetration for first part of test compared with response of a first order system with $\tau_1 = 5$ minutes.

The response to the increase in upstream concentration after 26 minutes is now considered in detail.

The upstream concentration does not increase to its steady state level instantaneously, which would be an ideal step increase in concentration. However, the increase only requires a few minutes to reach steady state and is therefore a reasonable approximation. From Figure 5.4.5 the downstream concentration appears to increase in step with the upstream, although the penetration curve (Figure 5.4.6) shows that some time lag must exist as the increase in penetration with time appears to be exponential.

If no time lag exists and only direct penetration is in operation the penetration will be constant. If the system is first order the response will be exponential.

It has already been shown that the response of a first order system would be

$$\frac{y(t)}{A} = (1 - \exp(-t/\tau))$$  \hspace{1cm} (7.1)
In this case the penetration is rising from an initial value at 26 minutes of 2.05%. The penetration at time \( t \) will therefore be:

\[
p(t) = P(t) + 2.05
\]  

(7.5)

where \( p(t) \) is the penetration at time \( t \) and \( P(t) \) is the deviation from the initial penetration before the step increase in upstream concentration.

Figure 7.2.13 shows a comparison between the penetration data for the period of the step change in upstream concentration and calculated values with a time constant for the increase, \( \tau_c \), of 5 minutes. The agreement between the two curves is good suggesting that the system is first order.

First order response to step increase in upstream concentration (\( \tau_c = 5 \) minutes)

The period of time for which the upstream concentration was maintained at the level reached after the step increase in concentration is now considered in detail.

This period of the test is summarised in Figures 5.4.8 to 5.4.10. In fact, the upstream concentration is not constant. There is an increase in upstream concentration from 48 minutes to 54 minutes, resulting in an increase in downstream concentration. However, at the peak of the increase in upstream concentration (at 54 minutes) there is
a sharp increase in the downstream concentration and a corresponding increase in penetration (see Figure 5.4.10). Again, this peak in penetration appears to be superimposed on an exponentially increasing curve.

The peak at 54 minutes is followed by a fall and subsequent gradual increase in the upstream concentration towards the level of the peak. The downstream concentration increases in a similar manner, the shape of the curve being very similar to that for the upstream concentration. The penetration does increase during this time, but only slightly.

The end of this part of the test shows another peak in the upstream concentration at 99 minutes to a level similar level to the peak at 54 minutes. The downstream concentration also peaks at 99 minutes and there is a significant increase in the penetration. After this peak the upstream concentration experiences a step reduction in concentration. The downstream concentration also falls, but some time delay is evident. This is shown by the penetration remaining high after the peak in upstream concentration at 99 minutes, and only slowly reducing to its steady state level as the downstream concentration reduces to its steady state level more slowly than the upstream.

This reduction in upstream and downstream concentrations is shown more clearly in Figures 7.2.14 and 7.2.15. The downstream continues to decrease in concentration for a few minutes longer than the upstream and the rate of decrease in the first few minutes is less for the downstream than the upstream.
Reduction in upstream concentration during the first challenge with 0.46 micron particles on the third prefilter sample

The next part of this test which is of interest is the step increase in upstream concentration occurring at 218 minutes. Figures 5.4.11 to 5.4.13 show that prior to the step change the upstream, downstream and penetration are all at steady state. The upstream concentration initially falls to a low level before increasing to above the new steady state level. The
The corresponding fall in downstream concentration is more pronounced than the upstream fall, giving rise to a drop in penetration. As the upstream concentration then rises, the downstream concentration appears to rise exponentially.

The upstream and downstream concentrations during this period of increase are shown in greater detail in Figures 7.2.16 and 7.2.17. These figures are a clear example of the combination of a direct penetration and a penetration involving some time lag.

![Figure 7.2.16](image)

**Figure 7.2.16**
Step increase in upstream concentration during the first challenge with 0.46 micron particles on the third prefilter sample

Figure 7.2.18 shows a comparison of the data for the penetration during the period of the step change with a first order process. The time constant used in the figure is 10 minutes. The calculated values from the experimental data agree well with those from the first order model, suggesting that the response of the filter is again first order.
At the end of the test the upstream concentration was reduced to zero in a few minutes. The response of the downstream concentration was considerably slower, although insufficient data were recorded for any attempt to determine whether or not the response is first order (see Figures 5.4.14 and 5.4.15).
Summary of analysis of first challenge on third prefilter sample

Direct and delayed penetration are in operation. The response of the filter to peaks in the upstream concentration appears to be direct rather than delayed but an increase in penetration as a result of a peak does not always occur.

The response of the filter to the start of the challenge is first order with a time constant, \( \tau_a \), of 5 minutes. The response to a step increase in concentration after 26 minutes is again first order, with a time constant, \( \tau_a \), of 5 minutes.

When the upstream concentration is maintained almost constant for about an hour there is a gradual increase in the penetration, evidence of a long-term delayed penetration.

A step reduction in upstream concentration causes a reduction in downstream concentration but with evidence of a delay between upstream and downstream.

A subsequent step increase in upstream concentration produces a first order response in the penetration, the time constant being 10 minutes. The reason for the difference in time constants in response to a step increase between the start of the test (5 minutes) and this later step increase (10 minutes) is not clear. However, the important observation is that the response is first order and the time constants are the same order of magnitude.

Switching off the challenge at the end of the test causes a delayed reduction in downstream concentration, but there is insufficient data to determine the nature of the downstream decay.

Clearly, direct, short-term delayed and long-term delayed penetration are all in operation. It appears that under certain conditions, such as peaks in the upstream concentration, the penetration shifts from being delayed to direct, sometimes resulting in an increase in penetration.
Second challenge with 0.46 micron particles

The results for the second challenge with 0.46 micron particles on the third prefilter sample are presented in Figures 5.4.16 to 5.4.18. The major part of the test involved maintaining the upstream concentration at a steady level for about 6 hours to see whether there was any change in penetration over long time periods.

At the beginning of the test there is a peak in the upstream concentration with a count of over 700,000 particles per 100 ml being registered for just one minute. The downstream response is a peak at the same time with a penetration of 13%. There is no evidence of time delay between upstream and downstream concentrations at this point in time. Following the peak some time delay becomes evident.

Figure 5.4.18 shows that after the peak at the start of the test the penetration remains fairly constant at approximately 4%. However, Figure 7.2.19 shows that the penetration does increase slightly over the time at which the upstream concentration is maintained constant, that is, steady state is not reached.

![Graph showing increase in penetration at constant upstream concentration during the second challenge with 0.46 micron particles on the third prefilter sample](image)

**Figure 7.2.19**

Increase in penetration at constant upstream concentration during the second challenge with 0.46 micron particles on the third prefilter sample
It is clear that some increase in penetration is occurring during this time. In Figure 7.2.20 an attempt at a linear regression is shown. Although the relationship between the penetration and time is not adequately described by the equation of the line shown in Figure 7.2.20, it does indicate the significance of the increase in penetration. In fact, the nature of the increase appears to be exponential, suggesting that a long-term first order delayed penetration is in operation.

Figure 7.2.20
Linear regression for penetration with time

When the challenge was switched off the downstream concentration decayed in the manner noted before with this particle size (see Figures 5.4.20 and 5.4.21).

Figure 7.2.21 shows the downstream decay with the downstream count modified to take account of the upstream concentration at that time using equation (7.4). The decay appears to be exponential. In order to test this the logarithm of the modified downstream concentration is plotted against time in Figure 7.2.22 and a linear regression attempted in Figure 7.2.23.
The straight line in Figure 7.2.23 is a reasonable representation of the downstream decay, suggesting that the process is exponential, that is first order. However, the first part of the decay does not appear to fit the line very well and it is not difficult to see that the process would be better represented by two straight lines, one with a large negative gradient, the other with a small negative gradient.
Figures 7.2.24 and 7.2.25 show regression fits for the first 10 minutes and the following 75 minutes respectively. The fit is considerably better in each case than that of Figure 7.2.23. It is therefore clear that the decay can be represented as two first order processes. From equation (7.3) the time constant for the decay is the reciprocal of the gradient. In this case the two time constants are $\tau_1 = 16$ minutes and $\tau_2 = 144$ minutes, where $\tau_1$ is the time constant for the first 10 minutes of the decay and $\tau_2$ is that for the remainder of the decay.
Summary of analysis of second challenge on third prefILTER sample

The large peak in the upstream concentration at the beginning of the test produces an increase in penetration. There is no time delay evident between upstream and downstream concentrations until the upstream concentration has reduced to a lower value and short-term time delay becomes evident.
The maintenance of a steady upstream concentration does not produce a steady state penetration; in fact, the penetration is still increasing after nearly six hours. This is clearly evidence of long-term delayed penetration which appears to be first order.

Switching off the challenge produces a decay in downstream concentration. This decay can be represented as two first order processes, one of time constant 16 minutes, the other 144 minutes.

There is again evidence of direct, short-term delayed and long-term delayed penetration. The occurrence of a peak in the upstream concentration again led to a shift from delayed to direct penetration.

**Third challenge with 0.46 micron particles**

The third test on this sample of the prefilter was designed to determine the effect of peaks in the challenge concentration. During the earlier tests much work was done on the response of the filter to step changes in the challenge concentration. During these tests it was noticed that when a large peak occurs in the challenge this often produces a peak in the penetration, with no time delay observed between upstream and downstream concentrations.

The earlier tests have shown that the response of the filter to step changes in upstream concentration has been first order with a time constant of approximately 5 minutes. The response of a first order system to an impulse is now considered.

Consider the following function:

\[ f(t) = \begin{cases} 
  1/h & 0 < t < h, \\
  0 & t < 0, \quad t > h 
\end{cases} \quad (7.6) \]

The integral of this function with respect to time will clearly be unity for any value of h. If the value of h becomes infinitesimally small the function becomes \( \delta(t) \), the unit impulse function. The value of this function at all times other than at the origin will be zero and the value at the origin will be infinite. The integral with respect to time will still be unity.
The response of a first order system to $\delta(t)$ is given by equation (7.7).

$$Y(t) = (1/\tau) \exp(-t/\tau)$$  \hspace{1cm} (7.7)

where $Y(t)$ is the deviation from the steady state value of the output of the system and $\tau$ is the time constant of the system. This response is shown in Figure 7.2.26 with a time constant of 5 minutes. Note, in Figure 7.2.26, $T = t/\tau$.

![Figure 7.2.26](image)

**Figure 7.2.26**  
Response of a first order system to a unit impulse ($\tau = 5$ minutes)

In this set of experiments performed on the prefilter an approximation to the impulse function was achieved by increasing the upstream concentration to a high level for a short period of time, after which the concentration returns to steady state. If the filter response is first order, an exponential decay similar to Figure 7.2.26 will be recorded downstream of the filter. In fact, with the limitations of the experimental apparatus, it was not possible to reduce the upstream concentration without some decay, such that the distribution of the upstream concentration with time appears somewhat exponential. Even without any first order response the distribution of the downstream concentration with time would appear exponential under these conditions. In order to determine whether or not the response to an impulse is first order the rate of decrease of the downstream concentration is compared with that of the upstream concentration.
Figure 5.4.23 shows a series of seven impulses in the upstream concentration and one step change. A detailed analysis of the response of the filter is not necessary as the data for each of the impulses shows that the downstream concentration decays at the same rate as the upstream, at least until the upstream concentration has reduced to a level at which the first order performance of the filter is restored. In other words, the occurrence of the impulse causes a change in the operation of the filter; the penetration shifts from being delayed to direct.

Figures 5.4.34 and 5.4.35 show the sixth impulse. The penetration curve, Figure 5.4.36, shows that after the direct penetration at the time of the impulse the penetration gradually decays over about an hour. The reason for this decrease in the penetration is that the upstream concentration has reached steady state while the downstream is still falling. In this case then there is evidence of a time delay between upstream and downstream concentrations, but only once the upstream concentration has reduced considerably following the impulse. This is shown in Figure 7.2.27.

![Figure 7.2.27](image)

**Figure 7.2.27**

Decay in penetration after the sixth impulse
The decay in penetration takes place over a considerable time and is not comparable with the response of a first order system which has a time constant of less than 10 minutes. The important point is that there is a considerable time delay between upstream and downstream concentrations.

In order to check that this lack of first order response in the event of an upstream impulse is not due to the condition of the filter after several tests have been performed on it, a step increase in the upstream concentration was performed. The results are shown in Figures 5.4.37 to 5.4.39. It is clear that the response to this step change is first order with a time constant of similar order of magnitude to that observed in earlier tests, that is less than 10 minutes. After the step change, when the upstream concentration was switched off, the downstream concentration again decayed at a slower rate than for the increase at the moment of the step change (see Figure 5.4.41).

Figure 7.2.28 shows the logarithm of the downstream count modified by equation (7.4), plotted against time after switching off the challenge. The shape of the curve is similar to that of Figure 7.2.22, where it was found that the decay could be represented by two first order processes with different time constants.

Figure 7.2.28
Log plot of decay after step change
Figure 7.2.29 shows the linear regression for the plot of Figure 7.2.28 from 10 minutes onwards. The agreement is very good and the time constant, $\tau_d$, for the decay is 31 minutes. This is not as high as that found for the second set of tests performed on this sample, which had a $\tau_d$ of 144 minutes. The reason for this is not clear but it may be due to differences in the loading of the filter. The important observation is that two first order processes are again in operation.

![Figure 7.2.29](image)

**Figure 7.2.29**

Linear regression of log of downstream decay with time

The seventh impulse, which was performed after the step change in concentration, again showed direct response (not first order response) and an increase in penetration.

**Summary of analysis of third challenge of third prefilter sample**

The response to upstream impulses is not first order, there is no time delay between upstream and downstream concentrations and there is a significant increase in penetration coinciding with the impulse. There is a shift from delayed penetration to direct penetration. If the upstream concentration following an impulse is not reduced to zero immediately, the operation of the filter shifts back to delayed penetration.
The step increase in upstream concentration after the sixth impulse produces a first order response in the penetration, with time constant of similar magnitude to that noted in the earlier tests on this sample.

After maintaining the upstream concentration constant after the step increase in concentration, the challenge was switched off and a gradual decay in downstream concentration observed. As before, the decay can be represented as two first order processes.
7.3 Summary of findings for the 0.45 micron prefilter

There are three types of penetration in operation. These are:

1. **Direct penetration.** The particles downstream of the filter have experienced no residence time within the filter. If this was the only penetration in operation there would be no time delay between upstream and downstream concentrations.

2. **Delayed penetration - short time constant.** The change in penetration as a result of this is first order with a time constant in the range 5 to 10 minutes. If this were the only penetration in operation, after a step change in upstream concentration the penetration would reach steady state after approximately 4 time constants from the time at which the step change was introduced, that is 20 to 40 minutes.

3. **Delayed penetration - long time constant.** When this type of penetration is in operation there is a gradual increase in penetration even hours after the upstream concentration has reached steady state. The process is again first order and the time constant for the process can be greater than 100 minutes.

With a challenge particle size of 0.46 micron all three types of penetration operate. Under certain conditions there is a shift away from delayed penetration to direct penetration, resulting in an increase in penetration. This is observed when there are impulses in the upstream concentration.

The only other particle size for which any delayed penetration was observed was 0.55 micron. However, the increase in penetration coinciding with peaks in the upstream concentration was also observed with 0.65 micron particles.
7.4 Summary of findings for the membrane filters

Two membrane filters were tested, one rated at 0.45 micron, the other rated at 0.4 micron. The results are presented in Chapter 6. Only a brief discussion of these results is included because the importance of this work is that it demonstrates the differences between the membranes and the prefilter such that the mechanisms of operation of the prefilter can be determined.

7.4.1 The 0.45 micron membrane filter

The first sample of this filter was tested with three particle sizes: 0.37, 0.31 and 0.46 micron.

The results for 0.37 micron showed that of the very small proportion of particles which penetrate the filter none do so with any significant time delay, that is all the penetration is direct.

It is not certain from the results of the 0.31 micron challenge whether any short term delay between upstream and downstream concentrations exists. When the particle feed to the filter is switched off there is no gradual decay in downstream concentration, implying that there is no long term delayed penetration.

For 0.46 micron particles challenging this filter, there is no time delay between upstream and downstream concentrations.

The second sample was challenged with 0.46 micron particles. A peak in the upstream concentration causes a corresponding downstream peak with no time lag. There is also a significant peak in the penetration (see Figure 6.2.25). Later in the same test the upstream concentration again rises to the same value as it was at the time of the peak mentioned above. On this occasion there is no increase in penetration (see Figure 6.2.28). The difference between these two situations is that in the first case the high concentration was only recorded for one minute whereas in the second case the high concentration was maintained for longer.
A later upstream peak of similar magnitude and duration as that at the start of this test caused no significant rise in penetration (see Figure 6.2.31).

There was little indication of significant time delay between upstream and downstream concentrations with this test, that is, nearly all of the penetration was direct. However, there was a slight delay in the downstream concentration following some of the impulses, once the upstream concentration had returned to low levels (see Figures 6.2.32 to 6.2.35).

### 7.4.2 The 0.4 micron membrane filter

This filter was tested with three particle sizes: 0.37, 0.46 and 0.31 micron. For 0.37 micron particles there is no time delay between upstream and downstream concentrations, that is, the penetration is direct.

For 0.46 micron particles there is no time delay between upstream and downstream concentrations. Peaks in the upstream concentration did not produce increases in penetration. However, when the upstream concentration was increased and maintained at 2 million particles per 100 ml there was a significant increase in penetration.

For 0.31 micron particles there is a small time delay between upstream and downstream concentrations and what appears to be a first order response when the challenge is started. Also, a peak in the upstream concentration causes a peak in the penetration with no time delay (see Figure 6.320). There is also evidence of a short time delay when the particle feed to the filter is switched off (Figures 6.3.22, 6.3.27 and 6.3.29). However, there is no evidence of any long-term time delay, even after considerable loading on the filter.
7.5 CONCLUSIONS

When challenged with 0.46 micron particles the 0.45 micron prefilter experiences direct and delayed penetration. The delayed penetration may be short-term, which is manifest in the first order response of the filter to changes in the challenge concentration, or long-term, which is manifest by increases in penetration at constant upstream concentration or gradual decay of the downstream concentration when the challenge is switched off.

The direct penetration is always in operation but becomes particularly evident in response to large impulses in the upstream concentration, when there is a shift away from delayed penetration and an increase in total penetration.

The prefilter also shows short-term and long-term delayed penetration with 0.55 micron particles, although the proportion of the penetration which undergoes any delay is less than for 0.46 micron particles.

No long-term delayed penetration is observed for either of the membrane filters at any particle size. However, the 0.45 micron membrane filter showed evidence of a slight time delay after some of the upstream impulses with 0.46 micron particles. Also, the 0.4 micron membrane filter showed more significant delay between upstream and downstream concentrations with 0.312 micron particles.

Both of the membrane filters showed increased penetration coinciding with peaks in the upstream concentration; however, the increases were not as pronounced as for the prefilter and an increase did not always occur. When challenged with 0.46 micron particles the 0.4 micron membrane filter showed an increase in penetration when the upstream concentration was increased and maintained at a very high level.

In Chapter 8 the findings of this chapter will be related to the mechanisms of operation of the 0.45 micron prefilter so that the changes in penetration which occur are explained.
CHAPTER 8

MECHANISMS OF OPERATION OF THE PREFILTER

8.1 Introduction

8.2 A model for the mechanisms of operation of the prefilter

8.3 Explanation of the response of the prefilter to impulses in the upstream concentration

8.4 Relating the performance of the prefilter to the structure of the filter medium and to that of the membrane filters

8.5 Conclusions


8.1 INTRODUCTION

The penetration at any time is as a result of three processes. One of the processes is direct penetration. The other two are first order processes, one with a time constant of a few minutes, the other with a time constant orders of magnitude longer.

Under certain conditions the amount of penetration caused by each of the three processes may change; for example, in the event of impulses in the upstream concentration the penetration shifts from being delayed to direct, with a resulting increase in the total penetration.

In this chapter a model for the relationship between these three penetration processes is presented. The performance of the filter under the following conditions is explained in terms of the model.

1. Step increases in the upstream concentration
2. Maintenance of the upstream concentration at steady state
3. Impulses in the upstream concentration
4. Switching off the particle feed to the filter

Finally, the performance of the filter is explained in terms of the structure of the filter medium, and differences in the performance of the prefilter and the membrane filters are explained in terms of differences in the structures of the media. It is shown that the dominant mechanism of filtration in operation in all of the filters tested is sieving. The residence time of particles through the filter medium is explained in terms of a number of sieving stages in series, particles experiencing a residence time at each sieving stage.
8.2 A MODEL FOR THE MECHANISMS OF OPERATION OF THE PREFILTER

There are similarities between the behaviour of the prefilter and that observed in the filtration of colloidal silica by membrane filters\(^44\) and the fixed bed filtration of colloids,\(^48\) for which adsorption/desorption models have been developed. In each case the efficiency is initially high, but decreases. With the fixed bed and membrane filtration of colloids the concept of a breakthrough time is applicable because the initial high efficiency is maintained until a certain time, when breakthrough occurs, that is, the efficiency falls. For the prefilter, the concept of breakthrough time is not applicable because the initial efficiency is not maintained but starts to decrease immediately. Therefore the adsorption/desorption models do not explain the behaviour of the prefilter.

There are some similarities between Grant’s model\(^55\) for the sieving mechanism of microporous membranes and the observations for the prefilter (see Section 8.4). Grant’s model predicts a gradual increase in penetration with loading but does not explain the residence time distribution of particles within the filter medium.

Consider the three processes by which penetration of the prefilter occurs. Process 1 is direct penetration. There is no time delay between particles arriving at the upstream surface of the filter and penetrating the filter. This process can therefore not be in series with a process which involves a time delay between upstream and downstream concentrations.

Process 2 is delayed penetration with a short time constant. It is not in series with the direct penetration of Process 1 by the argument above. Furthermore, it cannot be in series with Process 3 as this would introduce a long time delay between upstream and downstream concentrations and it is clear from the experimental results that the first order increase in penetration begins as soon as the challenge to the filter is started.

It would seem that the only feasible arrangement for the processes of penetration is that they are all in parallel with each other. It follows then that the concentration downstream of the filter at any time \(t\) is given by equation (8.1)
\[ D(t) = D_1(t) + D_2(t) + D_3(t) \]  \hspace{1cm} (8.1)

where \( D(t) \) is the downstream concentration at time \( t \) and \( D_n(t) \) is the downstream concentration due to the \( n \)th penetration process.

As Process 1 is direct penetration it follows that (8.2) will always be true.

\[ D_1(t) \propto U_1(t) \]  \hspace{1cm} (8.2)

where \( U_1(t) \) is the concentration at time \( t \) of particles attempting to penetrate the portion of the filter which operates by direct penetration.

As the other two processes are first order it is necessary to consider the nature of the upstream concentration before determining the downstream response.

Consider the response of the filter to a step increase in upstream concentration. From equation (7.1) the response of a first order process to a step increase in concentration will be:

\[ D_n(t) = A_n (1 - \exp(-t/\tau_n)) \]  \hspace{1cm} (8.3)

where \( A_n \) is the amount of the step increase in upstream concentration which attempts to penetrate the filter through that portion of the filter which operates by penetration process \( n \), and \( \tau_n \) is the time constant for the \( n \)th penetration process.

Therefore, for a step increase in upstream concentration of \( A \) particles per 100 ml the response of the filter with three processes operating in parallel will be:

\[ D(t) = A_1 + A_2 (1 - \exp(-t/\tau_2)) + A_3 (1 - \exp(-t/\tau_3)) \]  \hspace{1cm} (8.4)

But from the definition of \( A_n \) it follows that

\[ A_n/A = p_{1n} \]  \hspace{1cm} (8.5)

where \( p_{1n} \) is the final steady state penetration for the \( n \)th process, that is.
\[ p_{tn} = p_n(t) \text{ at } t = \infty \] (8.6)

and \( p_n(t) \) is the penetration of the nth process at time, \( t \).

So from equations (8.4) and (8.5)

\[ D(t)/A = p_{t1} + p_{t2} (1 - e^{-t/\tau_2}) + p_{t3} (1 - e^{-t/\tau_3}) \] (8.7)

But from the definition of \( A \)

\[ D(t)/A = p(t) \] (8.8)

where \( p(t) \) is the total penetration at time, \( t \). Combining (8.7) and (8.8)

\[ p(t) = p_{t1} + p_{t2} (1 - e^{-t/\tau_2}) + p_{t3} (1 - e^{-t/\tau_3}) \] (8.9)

Figure 8.1 shows a plot of equation (8.9) with values of \( p_{tn} \) and \( \tau_n \) selected to be similar to those measured in the experiments reported in Chapters 5 and 7. In this figure \( p_{t1}, p_{t2} \) and \( p_{t3} \) are 0.01, 0.04 and 0.01 respectively. The values of \( \tau \) are 5 minutes for the second process and 100 minutes for the third process.

Figure 8.1 shows an initial response which appears to be exponential, but as the time increases steady state is not reached as soon as would be expected if only one first order process were in operation. This is the kind of response which was observed for the prefilter when step increases in upstream concentration were introduced.

In Figure 8.2 the model of three processes in parallel is compared with the experimental data for the start of the challenge with 0.46 micron particles on the first prefilter sample (see Figures 7.2.1 to 7.2.3) The time constant for the second process (short-term delay) is 5 minutes as this was the value calculated for the initial response of the filter (Figure 7.2.2). The time constant for the third process (long-term delay) was selected to fit the model to the experimental data. The value of this time constant is 80 minutes, which is of the same order of magnitude as that calculated for the decay in downstream.
Response of three processes in parallel to a step increase in upstream concentration
($\tau_2 = 5$ minutes, $\tau_3 = 100$ minutes)

concentration when the challenge to the filter was switched off (Figures 7.2.4 to 7.2.7).
The values of $p_{e1}$, $p_{e2}$, and $p_{e3}$ were also selected to fit the model to the data, the values
being 0.01, 0.045 and 0.005 respectively.
The agreement between the model and the experimental data is good, confirming that the model is a fair representation of the operation of the filter. Note, the actual values of the time constants are not important - the important point is that three penetration processes are acting in parallel. In every other case when the prefilter experienced a step increase in upstream concentration with 0.46 micron particles a similar response was observed. The model can therefore be accepted.
8.3 EXPLANATION OF THE RESPONSE OF THE PREFILTER TO IMPULSES IN THE UPSTREAM CONCENTRATION

The model of three penetration processes in parallel describes the response of the prefilter to step increases in the upstream concentration and to maintenance of the steady state upstream concentration after the step increase, but at first sight it does not explain the response of the filter to impulses in the upstream concentration.

The results in Figures 5.4.23 to 5.4.36 show that when there is an impulse in the upstream concentration there is a shift from delayed to direct penetration and an increase in the total penetration. In other words, if the magnitude of the increase in upstream concentration in the event of an impulse is I and the amount of the impulse which attempts to penetrate through that portion of the filter which operates under penetration process is I_n, then the ratio of I_n to I is not the same as the ratio of A_n to A for each of the penetration processes. For an impulse there is much more direct penetration; therefore:

\[ I_1 > I_2 + I_3 \]  \hspace{1cm} (8.10)

The shift from delayed to direct penetration which occurs in the event of an impulse is explained below.

In the event of an impulse the concentration of particles at the upstream surface of the filter is momentarily very high. If the concentration becomes too high there is the possibility of coincidence at the opening of a pore, that is, more than one particle attempting to penetrate through the same pore. The result of this will be blocking of the pore if the size of the pore is not sufficient to allow passage of more than one particle at a time.

As the smaller pores become blocked there will be a redistribution of the water flow through the larger pores. The result will be that more of the particles will pass through the larger pores leading to an increase in the penetration because the capture efficiency of the filter at the larger pores is less than that at the small pores. This redistribution of flow and resulting increase in penetration is the basis of Grant’s model for the efficiency
of microporous membranes. However, unlike the situation in Grant's work, the increased penetration in the event of an impulse is only observed at the time of the impulse. There must therefore be some form of temporary blockage of the pores as coincident particles compete for penetration such that once the blockage has been removed the flow is redistributed and the penetration returns to its previous level.

An explanation of why there is an increase in total penetration has been made but this does not explain why the penetration shifts from being delayed to direct.

From the experimental observations it is clear that delayed penetration is most abundant when the challenge particle size is close to the mean pore size of the filter. In these experiments delayed penetration was observed for the 0.45 micron rated prefilter with 0.46 and 0.55 micron particles but not with 0.65 or 0.94 micron particles. Also, other work has shown that at particle sizes of 0.32 micron all the penetration is direct. With 0.32 micron particles there will be very few pores close to this size and as a result there is no delayed penetration. It follows that the adsorption/desorption mechanisms of filtration are not occurring otherwise there would be a significant increase in penetration after the breakthrough time is exceeded. If the pores close to the size of the particles block temporarily in the event of an impulse and it is through these that delayed penetration occurs, it is easy to understand why there is a shift from delayed penetration to direct penetration and an increase in total penetration.

There is still one question regarding the response to upstream impulses which must be answered. If the concentration in the event of an impulse is sufficient to cause temporary blockage of pores through which delayed penetration would otherwise occur, why don’t these pores block in the event of a step increase in upstream concentration as the magnitude of the step increases recorded in these experiments is similar to that of the impulses? The answer to this question lies in the experimental procedure followed when performing an upstream impulse. The particle counter records counts every minute. However, to produce an impulse the particle injection pump is set to a very high level for just a few seconds. The concentration recorded in the minute during which the
Impulse occurred is therefore the mean concentration for that minute, not the concentration upstream of the filter at the moment of the impulse, which would be considerably higher.

This explanation for the response to upstream impulses is supported by experimental data, which show significant increases in penetration in response to step increases in upstream concentration after the filter has been loaded with particles. Figures 7.2.16 to 7.2.18 show a significant increase in penetration in the event of a step increase in upstream concentration after a few hours of loading at fairly high concentration. Because the filter is already heavily loaded the step increase causes sufficient blocking of pores to bring about a shift in the penetration processes. The response to the step increase in concentration still shows some delayed penetration as well as a significant increase in total penetration, suggesting that the shift in penetration processes is not as large as in the event of an impulse but more than that for a step increase in concentration on an unloaded filter.
8.4 RELATING THE PERFORMANCE OF THE PREFILTER TO THE STRUCTURE OF THE FILTER MEDIUM AND TO THAT OF THE MEMBRANE FILTERS

It is clear that the model of three penetration processes operating in parallel is a fair representation of the performance of the prefilter, with the extent to which each of the processes operates being dependent on the loading of the filter, the upstream concentration and the challenge particle size. The following section relates this performance to the structure of the filter medium.

It has already been stated that the process of direct penetration occurs in pores which are larger than the challenge particle. In such a situation there is no time delay between upstream and downstream concentration.

At particle sizes for which there are a large number of pores of similar size the delayed processes are observed. The short-term delay occurs with the prefilter when challenged with 0.46 and 0.55 micron particles, but not for sizes much smaller or larger than these. The pore size distribution for the prefilter is known to be large and there will therefore be a significant number of pores of similar size to both of these challenge particle sizes.

The short-term delay cannot be primarily due to the depth of the filter medium because there is evidence of short term delay at certain sizes for both of the membrane filters tested. On the other hand the long term delay was only observed for the prefilter and only for 0.46 and 0.55 micron particles.

The short-term delay in penetration for the membrane filters could be due to sieving. In Meloy’s work on sieving it has been shown that particles of a given size and shape have a certain residence time on a sieve. It is therefore conceivable that the sieving mechanism in filtration would introduce a time delay between upstream and downstream concentrations.

With regard to the long-term delays observed for the prefilter, consider the following situation. In Meloy’s work particles of different size or shape are separated based on the residence time of the particles in passing through a series of sieves. As the number
of sieves in series increases the difference in residence time between particles of different shapes increases. It is possible to consider the prefilter, which has a considerable depth, as a series of sieving stages.

Consider a channel through the prefilter such as that shown in Figure 6.3. Particles below a certain size will pass through the channel without being captured at any site and with negligible residence time. Particles above a certain size will be prevented from entering the channel or will be permanently captured at some site between the upstream and downstream openings of the channel. Particles within a critical size range will experience a residence time within the channel in a similar manner to particles passing through a sieve. Depending on the nature of the channel, a particle may experience many sites through which it requires a certain residence time to pass. The magnitude of the residence time at each of these sites will be a function of the particle size and the size and shape of the pore at this site.

![Figure 8.3](image)

**Figure 8.3**
A channel through the filter showing possible sites at which particles are delayed

From the proportion of the particles that penetrate the filter through each of the three penetration processes described by equation (8.9), it is clear that for 0.46 micron particles most of these are delayed few times through the depth of the filter, that is, most of these particles experience short-term delay. As the particle size increases it would be expected that for a larger number of the particles the number of sites at which delay occurs increases, causing long-term delay until the particle size exceeds the maximum which can penetrate through these pores. Penetration of particles above this maximum size will therefore all be through considerably larger pores, in which there is no delayed penetration with the particle sizes used in these experiments.
The delayed penetration processes can therefore be explained by sieving: the more sites at which the channel diameter is such as to cause delay in the particle’s passage, the greater the total delay of the penetration of the particle.

The differences in behaviour between the prefilter and the membrane filters in the event of impulses in the upstream concentration can also be attributed to the pore size distribution of the filter media. For the prefilter there is a considerable shift from delayed to direct penetration with 0.46 micron particles, resulting in a large increase in the total penetration. This is due to the flow being redistributed through the larger pores, for which the capture efficiency is considerably lower. As larger particles are used there is no increase in penetration in the event of an impulse because all the penetration is direct for these particles as there are very few pores close to their size.

For the membrane filters there is a small but significant increase in penetration in response to an upstream impulse. The reason for this is that the pore size distribution of the membrane filters is much narrower so that the lower capture efficiency of the larger pores through which some redistribution of flow occurs is much less. The result is an increase in total penetration, but not to the same extent as for the prefilter.

The time distribution of the pulsing data for the 0.4 and 0.45 micron filters can also be explained in terms of the structure of the filter media. With the prefilter, a considerable time is required for some of the particles which are released by pulsing the flow to penetrate the filter. This is because after a pulse the channels through the filter return to their normal size and the penetration processes described above are again in operation.
8.5 CONCLUSIONS

The performance of the prefilter can be represented as three penetration processes operating in parallel. One of these processes is a direct penetration involving no time delay between upstream and downstream concentrations. This process occurs largely in pores which are significantly larger than the particles.

The other two processes are first order, one with a time constant of a few minutes (short-term delay), the other with a time constant orders of magnitude higher (long-term delay). The short-term delay only occurs with particle sizes for which there are a large number of pores of similar size to the particles, and is not affected by the depth of the filter medium. This penetration process is therefore in operation in membrane filters at certain particle sizes, as well as in the prefilter.

The long-term delay is only in operation in filters which have considerable depth with respect to the size of the particles and only with particle sizes for which there are a large number of pores of similar size to the particles. It is therefore not in operation in the membrane filters.

Both of the delayed penetration processes are caused by the sieving mechanism of filtration. As particles of similar size to the pore attempt to penetrate it they experience a residence time within the pore. In a channel through a depth filter there may be several sites at which a particle may be delayed, experiencing a residence time at each of these sites. The result is a long time delay between particles arriving at the upstream surface and penetrating the filter.

The distribution of pore sizes at the upstream surface or within the depth of the filter will produce a distribution of residence times for particles of the same size.

It is clear that the residence time of particles within the filter is not due to adsorptive mechanisms of filtration but due to sieving at one or more sites through the depth of the filter. The delayed penetration processes of the prefilter can therefore be considered as a number of sieving stages in series, each sieving stage requiring a certain residence time.
for particles to penetrate. In some channels through the filter, particles of a certain size may only be delayed at one site (short-term delay) and in other channels particles of the same size may be delayed at several sites (long-term delay).

As the loading of the filter increases, or under conditions of very high upstream concentration, there is a shift from delayed to direct penetration, resulting in an increase in the total penetration. This is caused by temporary or permanent blockage of the pores through which delayed penetration would occur and the resultant redistribution of the water flow through larger pores. This effect increases with the width of the pore size distribution of the filter medium and is therefore more apparent for the prefilter than for the membrane filters, although it is significant for the membranes.
CHAPTER 9

CONCLUSIONS AND RECOMMENDATIONS

9.1 Introduction

9.2 Mechanisms of filtration for the prefilter

9.3 Implications for cartridge filter evaluation and selection of filters for given process conditions

9.4 Recommendations for future work
9.1 INTRODUCTION

The efficiency of the prefilter, and to a certain extent that of the membrane filters, depends not only on the challenge particle size, but also on the degree of loading, the upstream concentration, the nature of the water flow, and time. Because of this it is unacceptable to attempt to evaluate filters of this type simply by measuring the particle removal efficiency for a given particle size at one point in time. Long-term testing with continuous monitoring of upstream and downstream concentrations is essential.

The operation of the prefilter can be represented by three processes acting in parallel. One of these processes is direct penetration, the downstream concentration displaying no time delay in its response to changes in the upstream concentration. For the other two processes a time delay between upstream and downstream concentrations is observed, the nature of the response in each case being that of a first order dynamic system. The time constant for one of these processes is 5 to 10 minutes, but is orders of magnitude greater than this for the other.

The membrane filters also show some time delay between upstream and downstream concentrations, but only that of a first order system with a time constant of a few minutes. The longer-term delay observed for the prefilter is not in operation for the membrane filters.

The mechanism of filtration in each of the processes described above is sieving. The time delay is introduced by the residence time within the pores of particles of similar size to the pores, in a similar manner to the residence time on a sieve of particles close to the sieve size. The long-term delay between upstream and downstream concentrations is caused by particles experiencing several sites through the depth of the filter at which the pore size is close to the particle size, the total residence time within the filter being the sum of the residence times at each of these sites.
The penetration of panicles through the prefilter can be represented by three processes operating in parallel. Process 1 is direct penetration, Process 2 is short-term delayed penetration and Process 3 is long-term delayed penetration. Both delayed penetration processes can be considered as first order processes, the time constant for Process 3 being considerably longer than that for Process 2.

The dominant mechanism of filtration is sieving. The delay in penetration is caused by the residence time of a particle passing through a pore of diameter close to the particle size. The long-term delay is caused by the particle passing through several sites at which the pore size is close to the particle size.

The prefilter can be thought of as a number of sieving stages in series. The pore sizes of each sieving stage are widely distributed such that there will be a distribution of channels through the filter, some being of large diameter throughout the depth of the filter, some of narrow diameter throughout, and others varying in diameter. In penetrating the filter a particle must travel through a channel. For particles considerably smaller than the rating of the filter there will be a large number of channels in which the particles experience no residence time because the pore size is greater than the particle size at each sieving stage. Hence for particles considerably smaller than the filter rating the penetration is direct.

For particles of similar size to the filter rating there will be some channels which will be sufficiently narrow at one site through the depth of the filter to impart a residence time to the particle. For other channels there will be many sites imparting particle residence times. Hence short- and long-term delays are present. But direct penetration is also present for these particles; hence channels of large diameter throughout the depth of the filter must exist.

The presence of channels of large diameter throughout the depth of the filter is confirmed by the direct penetration of particles as large as 0.94 micron and by the fact that no delayed penetration is observed for particles larger than 0.65 micron.
Although analysis of the filter evaluation data shows that there are three penetration processes operating in parallel, it is clear that they are all caused by the same mechanism of filtration. Sieving occurs at sites through the depth of the filter, either capturing particles or imparting a residence time to the passage of particles through the filter.

The proportion of the total penetration attributable to each of the three processes described above is dependent on the loading of the filter. Channels through which delayed penetration occurs in the unloaded filter become blocked on loading and the resultant redistribution of the water flow produces an increase in the proportion of challenging particles which attempt to penetrate through the wider diameter channels of direct penetration. As the capture efficiency of these channels is lower, not only is there a shift from delayed to direct penetration but there is also a marked increase in total penetration.

The same effect is observed in the event of impulses in upstream concentration. The very high concentration causes a temporary blockage of the channels of delayed penetration due to coincidence of challenging particles, resulting in redistribution of the water flow and an increase in total penetration. Once the upstream concentration has returned to lower levels after the impulse, normal penetration takes place, that is delayed and direct penetration are evident.

With the membrane filters tested the direct penetration and short-term delayed penetration of Process 2 operate. No long-term delay is observed. These filters can therefore be considered as single sieves, rather than as a series of several sieves. The shift from delayed to direct penetration and the increase in total penetration are still apparent, but less significant than with the prefilter because the pore size distributions in the membrane filters are considerably narrower than that of the prefilter.

Pulsing the water flow to all of the filters tested in this work produced significant release of previously captured particles. This can be explained in terms of the sieving model. In the event of a water pulse some of the channels through the filter are momentarily dilated, allowing particles to resume their passage through the filter. This model also explains why the rate of decrease in downstream concentration following a water pulse
is less for the prefilter than that for the membrane filters. With the prefilter a particle released by a pulse may still encounter several sites of delay before finally penetrating the filter, unlike the membrane filters which only operate as a single sieving stage.
9.3 IMPLICATIONS FOR CARTRIDGE FILTER EVALUATION AND SELECTION OF FILTERS FOR GIVEN PROCESS CONDITIONS

Because the efficiency of a filter varies with time, loading and upstream concentration, it is essential that filter evaluation methods allow these factors to be considered. It is not sufficient to quote a value for the efficiency of a filter for a given particle size as this value will be meaningless. The efficiency of the filter must be considered in terms of its dependence on the variables mentioned above. Continuous monitoring of particles either side of the test filter is therefore essential for filter evaluation.

It has also been shown in this thesis that even particles which are captured by the filter can be released from it under conditions of pulsed water flows. In industrial applications, where the flow of water through a filter may vary considerably, the response of the filter to pulsing water flows must be known.

The dependence of filter efficiency on challenge concentration has implications on the suitability of filters in certain process situations. If the concentration upstream of the filter is likely to vary considerably with time then the choice of filter should be governed not only by its steady state efficiency but also by its dependence on upstream concentration. Similarly, if the water flow is likely to vary considerably, selection of a filter which retains its particles under pulsed water flow conditions is essential.
9.4 RECOMMENDATIONS FOR FUTURE WORK

The aim of this work was to determine the time dependence of cartridge filtration. It has been shown that filter efficiency is not a constant for a given particle size, but that it varies with time, filter loading and upstream concentration. However, it has not been possible to determine how the efficiency varies with loading or concentration as the experiments were not designed to consider these variables. Because of the extent to which the efficiency varies with time, it has not been possible to quantify the relationship between efficiency and particle size. In order to do this, the same particle size, concentration, loading, etc would have to be used for each sample of every filter tested.

The behaviour of the filters tested in this work under pulsed flow conditions has only been considered very briefly. The work to date simply shows that particles are released from filters when the water flow is pulsed, and that the number of particles released is dependent on the particle size. For a fuller understanding of filter performance in these conditions the effect of loading on the filter should be considered. This work could also be extended to show the forces which must be overcome in order to release particles from filters by pulsing the flow.

The work in this thesis has concentrated mainly on filters rated at 0.4 to 0.5 micron. In many industrial applications 0.1 micron rated filters are now common and particles even smaller than this are thought to be critical to the process. Because the mechanism of filtration is sieving it is likely that similar transient effects will be observed with 0.1 micron filters in response to challenge with 0.1 micron particles. There are now several instruments available which detect particles smaller than 0.1 micron and this work needs to be repeated at these sizes.

The removal of particles from filters by pulsing the water flow also needs further consideration. Recent work carried out at CERCon has shown that 0.1 micron rated filters which have been challenged with 0.1 micron particles release significant numbers of particles when the water flow is pulsed. Extension of this work will enable knowledge to be gained of the removal forces necessary for particle release from filters and the effects of the degree of loading and challenge particle size.
All of the work considered thus far has been under conditions where sieving is the dominant mechanism of filtration. However, there are cartridge filters designed to capture particles considerably smaller than their pore size. Such filters are designed to operate by other mechanisms, for example, positively charged membrane filters. The retention of particles by such filters under pulsing water flows requires consideration. Where sieving is dominant, opening the pores in the event of a pulse allows particle release. With electrostatic effects on particles considerably smaller than the pore size the opening of a pore would not affect the force of attraction and it is possible that good retention of particles under pulsed flow conditions will result. On the other hand, the high water velocity in the event of a pulse may result in sufficient drag force to remove particles captured in this way.

Some insight into the transient nature of sub-micron cartridge filtration has been gained but the above recommendations should be followed if the mechanisms in operation are to be fully understood.
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