

Simple laboratory techniques improve the operation of RO pre-treatment systems.

Authors: *Mr. Edward G. Darton, Ms. Silvia Gallego*

Presenter: *Ms. Silvia Gallego* – Genesys Membrane Products - SPAIN

Abstract

Membrane failures are often caused by poor pre-treatment design and/or poor operation. Systems are frequently designed and built without a clear understanding of the chemistry of the feed water to be treated and the physical characteristics of the particulate matter it contains. The larger the system the more likely is some incident of pre-treatment failure, often at a high financial cost to the operator. Such problems are common with medium and large surface water systems, particularly those using sea, lake, river and recycled effluent waters.

It is standard practice to carry out pre-treatment chemical trials (coagulation and flocculation) on-site. However, new techniques such as on-line particle counting and the examination and analysis of SDI papers allow more comprehensive scientific investigation to be made under laboratory conditions. Results from laboratory studies mean that it is often possible to improve pre-treatment operation, even on a well run system.

This paper considers traditional membrane autopsy techniques and looks at some of the new laboratory tools available to improve pre-treatment system performance. The authors discuss some of the laboratory techniques involved such as the use of ‘particle counting’ and the chemical examination of SDI papers and show how they can help pre-treatment systems operate more effectively without the need for costly re-engineering. Real case experiences are used to illustrate this discussion.

I. MEMBRANE SYSTEM FAILURES. THE IMPORTANCE OF PRE-TREATMENT.

Many plant operators and OEMs have seen membrane failures caused by poor pre-treatment design or operation. Poor feed water quality often leads to fouling problems and increased membrane cleaning, meaning higher water production costs, with irreversibly fouling, flux loss, as well as the irreversible increase in salt passage.

In more than 200 autopsies made at the Genesys Laboratories in Madrid, it has been seen that in more than 60% of cases membrane damage can be linked, directly or indirectly, to problems or inefficiencies in pre-treatment processes as shown in [Figure 1](#). This is a significant fact.

These systems are often designed and built without a clear knowledge and understanding of the chemistry of the water and physical characteristics of the particulate matter within it, nor its variability. Failures relating to the dosage of pre-treatment chemicals (coagulants and flocculants) are also important. It was seen that in 12% of cases, episodes of chemical overdosing or under dosing due to water quality variability and inappropriate monitoring of the process, has led to a fall in production. In some occasions irreversible fouling has also been seen.

Particulate (colloidal) matter in feed water forms on both the membrane surface and within the spacer medium, and this is particularly true for membrane elements in the lead position. This particulate matter can be inorganic in nature such as alumino-silicates, metallic oxides, or organic matter such as humic acids, polysaccharides, etc. Micro organisms such as bacteria, fungi, moulds or protozoa, should also be considered as they are invariable present as a biofilm.

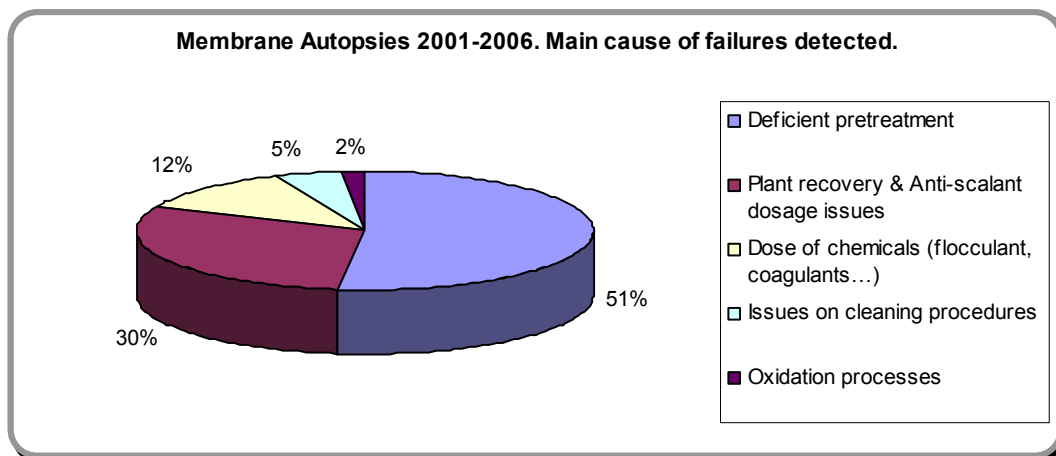


Figure 1. Main causes of membrane failures detected during autopsy (2001-2005). Source: GMP laboratories statistics

Although the membrane position is not known for every element autopsied, [Figure 2](#) shows the main types of foulant found in the first or lead position elements. The main foulants seen on membranes in the first position are alumino-silicates (35%), organic matter (27%) and biofilm (22%). This data ties in reasonably well with data presented by Darton and Fazel [1].

To prevent these deposits from occurring, improvements in pre-treatment design or operation were specifically recommended in all cases for each type of deposit.

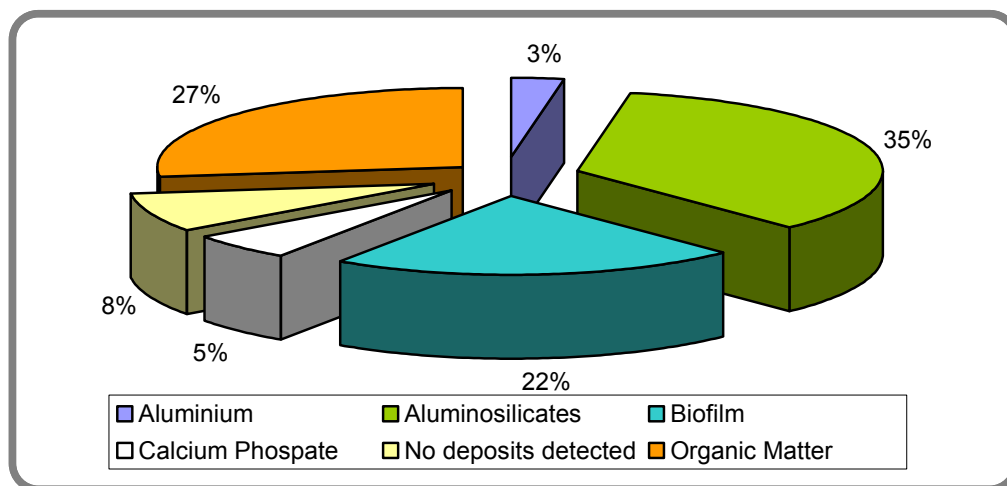


Figure 2. Main types of foulant found on membrane elements in first position during autopsy (2001-2005).
Source: GMP laboratories statistics.

Although membrane autopsy is the standard tool for evaluating and identifying deposits on membrane surfaces, the high cost of analyses and the need for element replacement make this technique expensive for small to medium sized systems. Typically, plant operators will only consider the autopsy option when system performance is dramatically affected and operational costs are rising sharply. For this reason the search for complementary and/or alternative laboratory tools becomes a priority.

Analytical techniques such as scanning-electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX), used in membrane technology and autopsy, or optical particle counting devices now provide technicians with new approaches for characterizing the nature of particulate matter in RO feed water, and then evaluating and improving pre-treatment performance. Both techniques will be described and case studies discussed in the remainder of this paper.

II. ANALYTICAL METHODOLOGY.

2.1. Scanning-electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX).

The SEM-EDAX technique is used to study deposits on membrane surfaces, cartridge and security filters and even SDI papers. The information gained is invaluable when proposing preventative and corrective actions in pre-treatment operation. In many cases the examination of cartridge filters and SDI papers avoids the need for sacrificing membrane elements to autopsy. Examples are described in case studies in section 3.

SEM micrographs of different cartridge filters are show in [Figures 3,4 and 5](#).

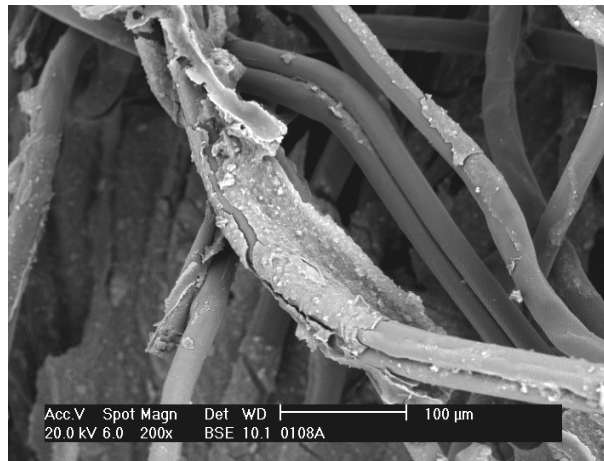


Figure 3. SEM Micrograph. Detail of 5µm cartridge filter surface. Organic deposits and aluminosilicates are partially covering filter fibres.

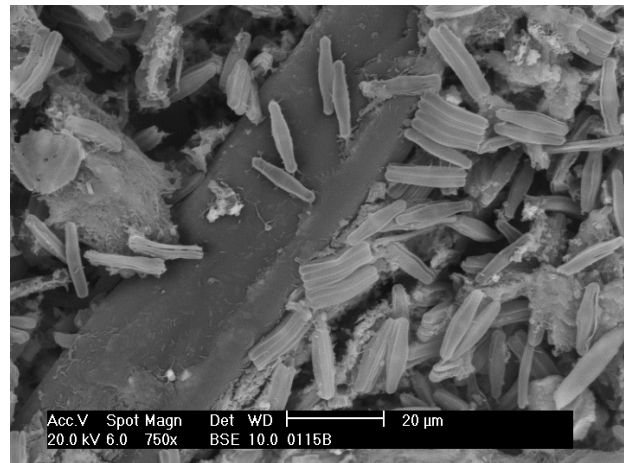
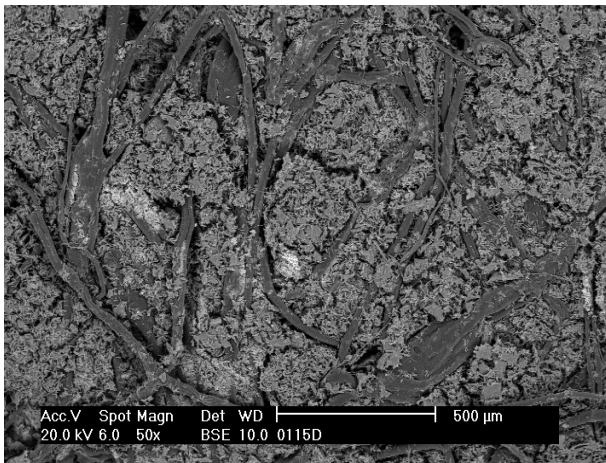


Figure 4&5. SEM Micrographs. General view and detail of 5µm cartridge filter surface. Massive presence of diatoms detected.

2.2. Optical particle counting technology.

Particle counters are rapidly becoming an essential tool for improving pre-treatment design and system operation, not only when evaluating coagulant and flocculant performance, but particularly when evaluating sand filter and cartridge filter performance. Particle size is one of the most important parameters when selecting or designing a filtration system and in many cases is seldom considered. Figure 6. Turbidity has commonly been used as a reference parameter, but turbidity is affected by size, shape, colour and particle refraction index.

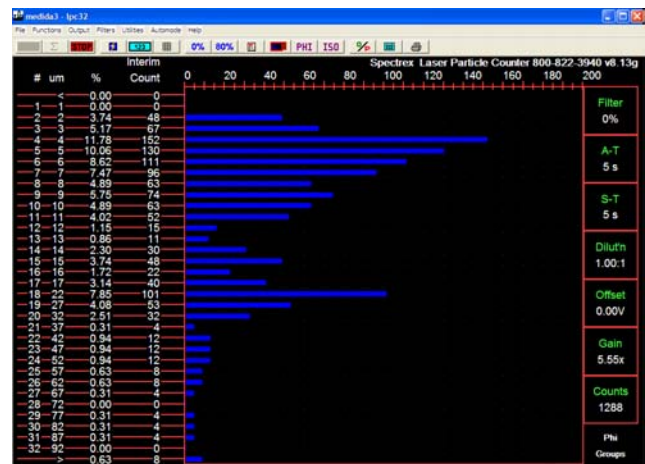


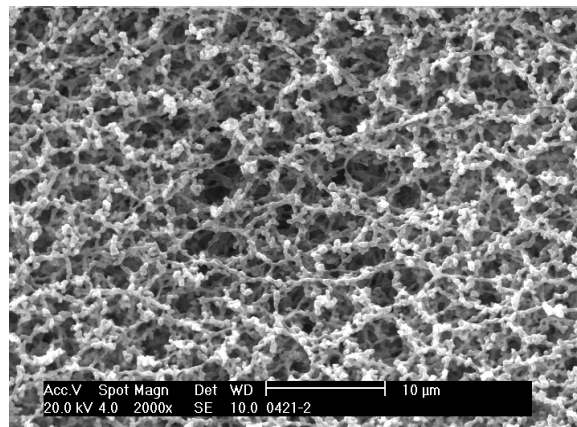
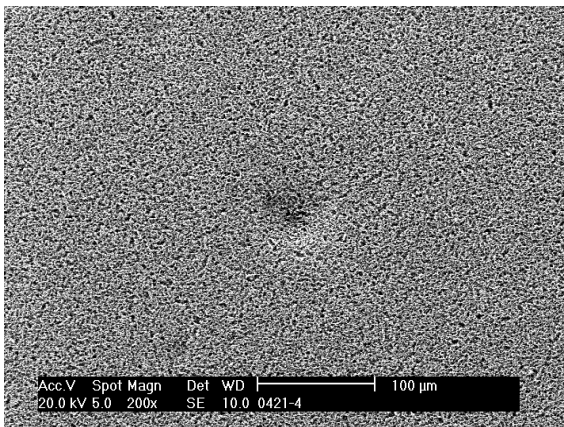
Figure 6. Example of a particle size distribution graph from an RO pre-treatment system.

III. CASE STUDIES

3.1. Membrane cleaning advice based on particulate matter characterisation by SEM-EDX

The decrease in normalized product flow and the increase in differential pressure or permeate conductivity are symptoms of membrane fouling. Membrane cleaning is recommended when variations of between 10-15% in these parameters are observed. Colloidal fouling dramatically affects the flow rate, especially to membrane elements in the first position. Although SDI measurement has been used for many years to determine fouling potential of RO feed water, the SDI measurement cannot provide any scientific information about the nature and type of fouling expected.

When fouling occurs, the nature of the foulant must be identified if an effective cleaning program is to be suggested. An alternative procedure to membrane autopsy is to evaluate colloidal matter retained in an SDI filter paper. RO feed water is passed through a 0.45 μm paper membrane filter, then the filter surface is analyzed by SEM-EDAX and the nature of the colloidal matter is characterized. Some examples of SEM micrographs are shown in Figures 7-12.



Figures 7 & 8. SEM micrograph of a SDI paper (blank sample) at different magnifications.

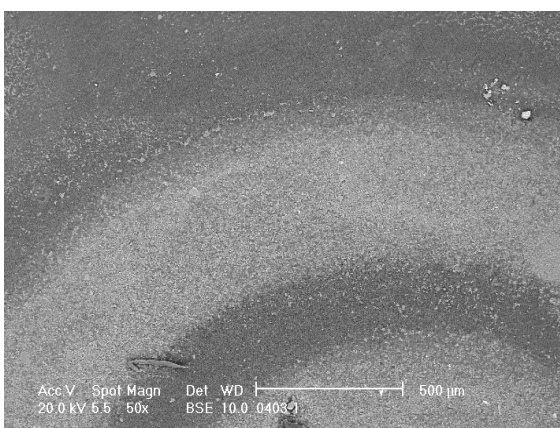


Figure 9. SEM micrograph of an SDI paper after the cartridge filter.

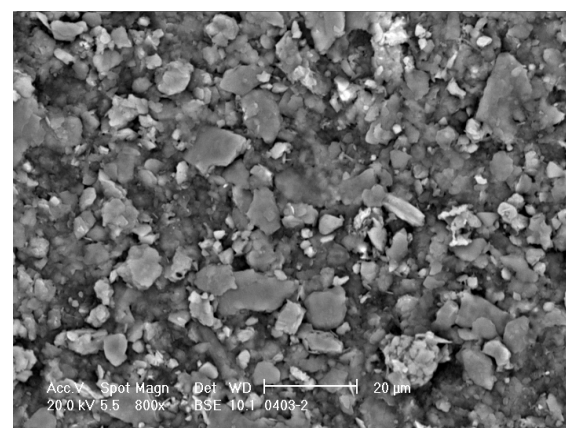


Figure 10. Detail of deposit detected on SDI paper shown in Figure 9 (Alumino-silicates).

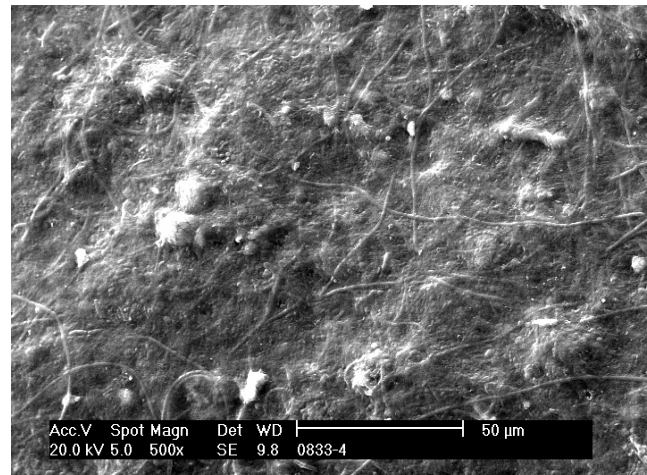
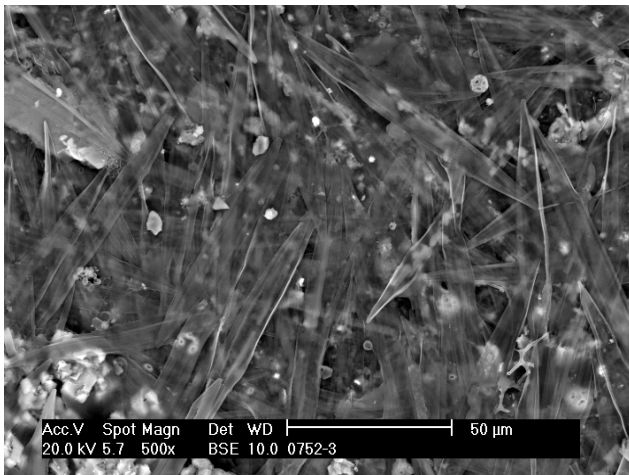


Figure 11 & 12 SEM micrographs of SDI papers. The deposits are organic in nature and micro-organisms have been detected on the surface.

Deposits from the surface of the SDI papers can then be analysed using energy dispersive X-ray analysis which identifies the elemental composition of the deposit as shown in the graph in Figure 13. The predominant elements are silica and aluminium with some calcium and iron. This is indicative of alumino-silicate clay. The results of analysis of these SDI papers allow our technicians to recommend specific cleaning protocols. An effective cleaning recommendation saves in cleaning chemicals and minimizes operation costs, as well as helping to ensure a longer life for the membrane elements.

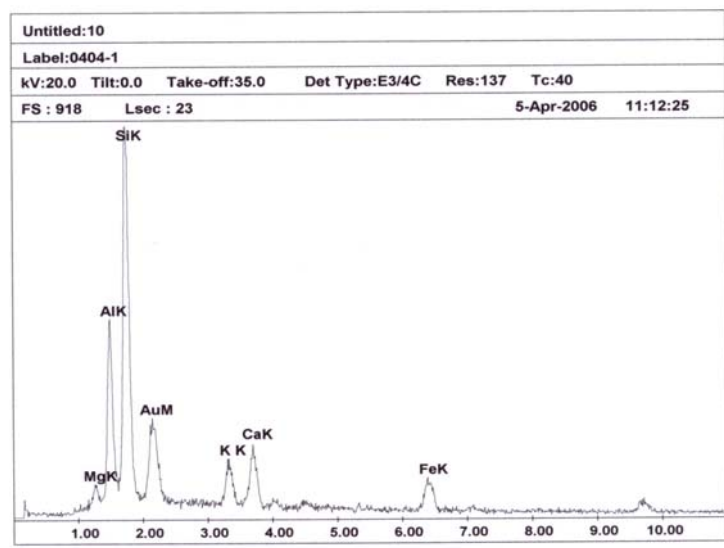


Figure 13. EDAX analysis result (area shown in Figure 10).

3.2. Optimizing coagulant and flocculant dosing using a particle counter.

Coagulation-flocculation assays have largely been used in wastewater treatment systems where the floc formation can be visually seen. However, as the turbidity of RO feed water tends to be significantly lower than that of wastewater, alternative procedures have been developed to study coagulant and flocculant behaviour and evaluate their efficiency.

Particle counters help to decide the best products available and the optimum dosage for improving the performance of existing pre-treatment systems. The objective of coagulation is to aggregate particulate matter to form flocs of sufficient size to guarantee retention by the filtration system. Comparison of particle size distribution under different experimental conditions is a valuable tool to aid decision making. Dose rate data can then be obtained in the laboratory simply using different compounds, with variable dose rates and pH conditions.

Figures 14 and 15 are examples of particle counting usage for coagulation-flocculation assays. After dosing a cationic membrane-compatible flocculant, the particle size distribution curve has been shifted to the right and shows larger flocs, so improvements to the installed filter media are assured. In Figure 15 filtration efficiency has also been tested and shows the efficacy of coagulation/flocculation and the performance of the filtration system.

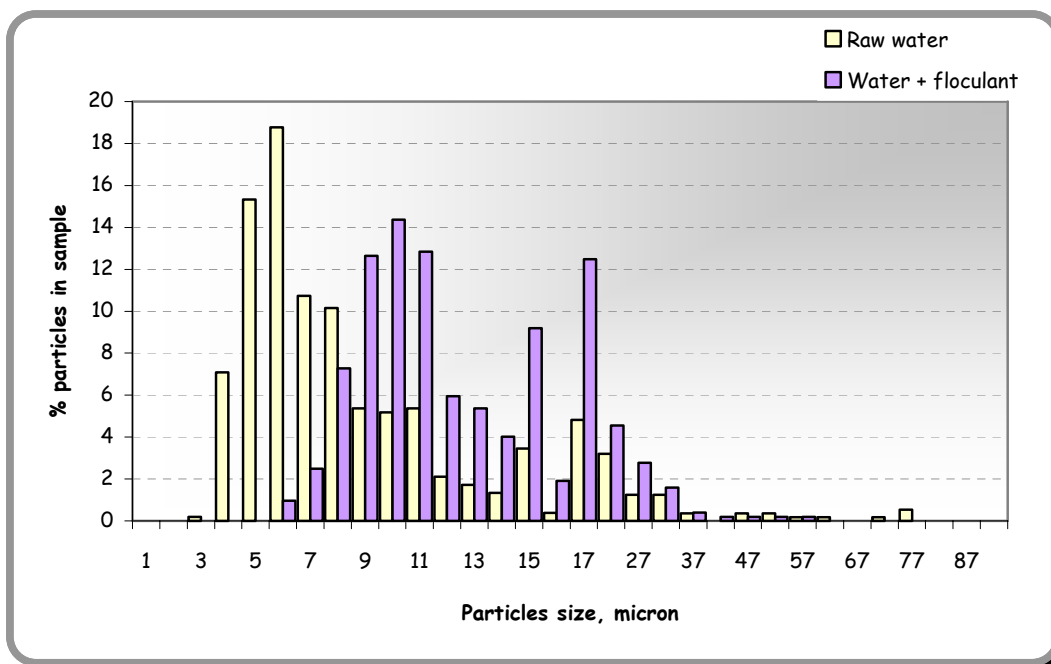


Figure 14. Comparison between particle count results for a RO feed water sample before and after dosing 3 ppm of cationic membrane-compatible flocculant.

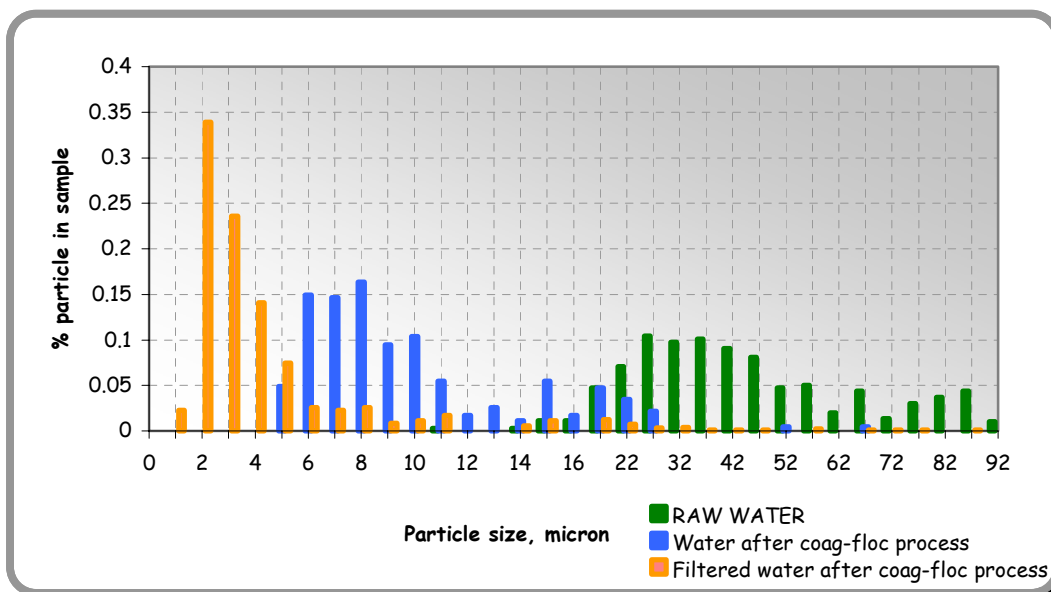


Figure 15. Coagulation-flocculation-filtration (5µm) essay results using a particle counter.

3.3. Particulate matter characterization and sand filtration optimization.

As explained in section 3.1 (membrane cleaning), selecting the best coagulant and flocculant is made easier when the nature of the colloidal and suspended matter is known. This is because the chemical substances used for coagulating small particles to larger flocs are different for organic matter, colloidal silica, clays, etc.

To protect membranes from fouling and to guarantee good performance, the Membrane Manufacturers recommend a Silt Density Index (SDI) value lower than 5. SDI values lower than 3 are highly recommended although it has been suggested [2] that a low SDI value itself is not a guarantee of membrane cleanliness. The variability of feed water quality often makes low SDI values difficult to achieve, especially for surface waters. Dosing membrane compatible flocculants before sand filters can usually be a cost-effective solution to improve particulate matter retention.

For optimizing filtration processes, it is essential to be aware of the nature of all particulate matter. In order to characterize these particulates, the same procedure described on section 3.1 for cleaning can be used. SEM-EDAX analysis of an SDI paper offers a quick and cost-effective tool for analysis.

Figures 16 and 17 show the results of a study carried out in an RO plant in north east Spain. After three months operation without any problems at an SDI <3, the membranes started to block due to intermittent episodes of high SDI. To study the nature of the particulate matter SDI papers used during plant monitoring were analysed by SEM-EDAX (as shown in Figure 16). Laboratory analytical results were used to decide which flocculant should be dosed and tested on site. The optimum dosage was then established by optimising the SDI values. The effectiveness of the proposed treatment was then evaluated by SEM-EDAX analysis of SDI papers used during the first evaluation.

In this way, the effectiveness of a simple and cheap means of filtration enhancement has been evaluated before considering complicated and expensive procedures such as MF or UF.

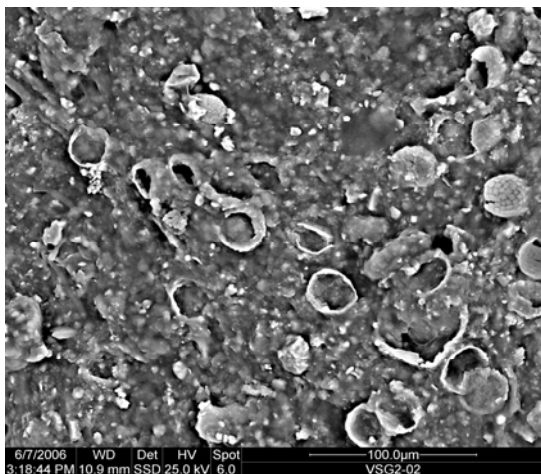


Figure 16. SEM Micrograph. SDI membrane surface after sand filter (SDI >5)

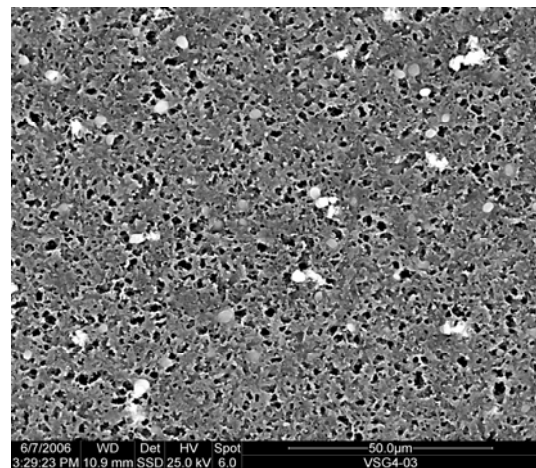


Figure 17. SEM Micrograph. SDI membrane surface in same point after dosing 2,8 ppm of a membrane-compatible flocculant (SDI <2)

3.4. The comparable performance of sand filters

When assessing source water and treated water quality, particle counting gives excellent results in determining process performance and treatment efficiency. This technique is useful for evaluating and comparing the performance of filtration systems, particularly in low turbidity waters.

Sand filters are commonly used in the pre-treatment of RO waters. The design of sand filters include such details as filtration surface, number and depth of filtering layers, granulometry of materials, etc. as well as operating conditions such as filtration velocity and cleaning frequency. The objective in changing one or more of these variables is to improve system performance and particle counting is a way of measuring changes to performance when small changes are made to filter design and operation. [Figure 18 and 19](#) show results obtained from two parallel sand filters from an open intake sea water plant in Chile. Significant differences in performance can be seen when comparing results obtained for both lines, especially for smaller particles (< 10 μm). This demonstrates that two identical systems on a common feed source are performing completely differently; such data enables the plant operator to take remedial action.

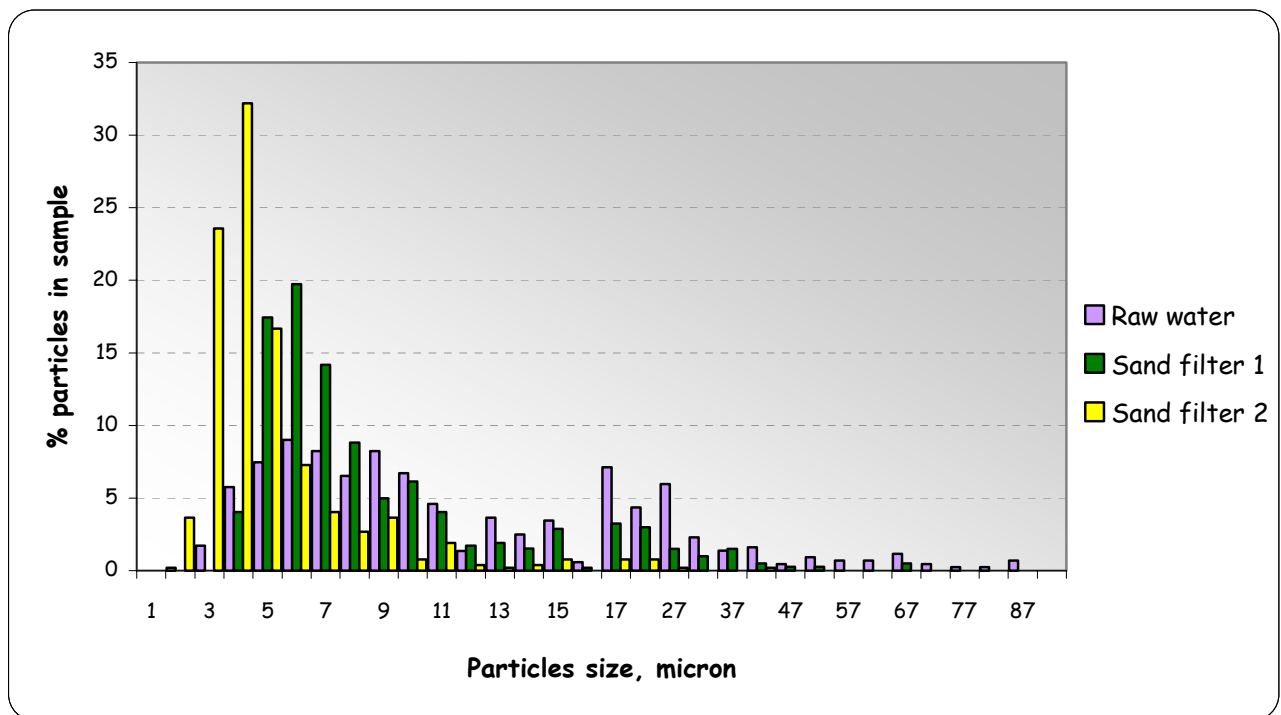


Figure 18. Size distribution graph for two sand filters operating in parallel.

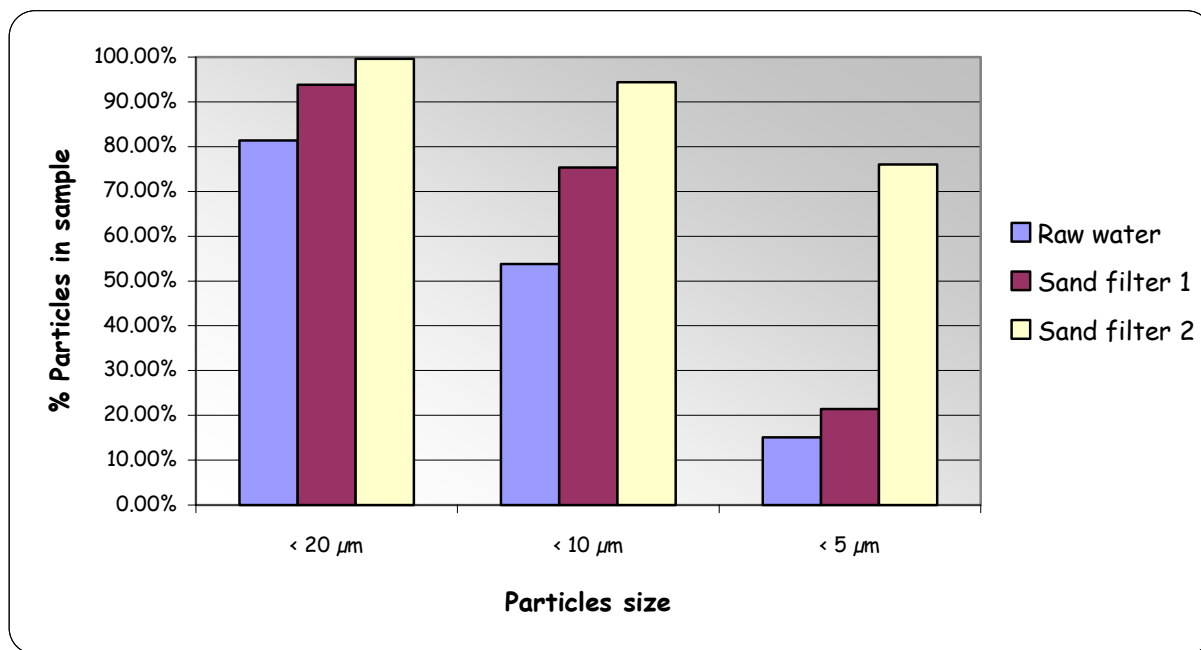


Figure 19. Particle size distribution for example shown on Figure 18

IV. CONCLUSIONS

- The analysis of data from more than two hundred RO membrane autopsies has shown that deficiencies in pre-treatment design and/or operation are the main causes of RO systems performing poorly.
- The nature of particulate matter found in RO feed waters can be widely variable. A statistical review of autopsy results on first position (lead membrane) elements shows the primary foulants to be alumino-silicates, organic matter and micro-organisms (biofilm and biomass).
- The ability to identify the nature and characteristics of these particles and their size distribution is extremely useful in setting up effective membrane cleaning programmes and improving pre-treatment design and operation, so as to eliminate or minimise the risk of fouling.
- Membrane autopsy has become the preferred tool for identifying and characterizing deposits on membrane surfaces. New analytical techniques provide an alternative to membrane autopsy, avoiding the need for sacrificing membrane elements and reducing (or eliminating) the need for plant down-time.
- Scanning-electron microscopy (SEM) and energy dispersive X-Ray analysis (EDAX) techniques have been widely used in membrane autopsy. The same technique can also be used to determine the nature of particulate matter in RO feed waters when SDI papers are analysed. This is a useful tool for plant operation and monitoring by selecting the best coagulation-flocculation system to optimize filtration efficiency as well as improving membrane cleaning.
- Particle counting provides a quick and reliable result when assessing source water quality, treated water quality, unit process performance (especially filtration) and total treatment

efficiency. Particle counting is a most useful tool for RO plant management in three important areas of pre-treatment system operation.

- The planning and design of filtration systems with respect to filter pore size.
- Filtration efficiency control.
- A simple alternative to conventional coagulation-flocculation essays (jar-tests) in low turbidity waters.

It has long been understood that the secret of membrane cleanliness, low cost production and long membrane life lies with the good design and efficient operation of the pre-treatment system. Such systems are frequently designed and built with little understanding of the characteristics of the water to be treated and we often find plant users involved in costly modifications to poorly-performing pre-treatment systems. The use of the techniques described in this paper can improve the performance of many such systems at minimal cost. Even small improvements made to ‘adequately operating pre-treatment’ systems offer the plant user savings in operational costs.

Part 2 of this paper examines some field trial experiences. It will be published in 2008.

References

1. Darton, E. G., & Fazel, M., “A statistical review of 150 membrane autopsies”. 62nd Annual International Water Conference, Pittsburgh, 2001.
2. Darton, E. G., Annunziata, U., Del Vigo, F., Gallego, S.; “Membrane autopsy helps to provide solutions to operational problems”. *Desalination* 167 (2004) 239-245.