

# Characterization of polymeric nanofiltration membranes for systematic analysis of membrane performance

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Received 28 April 2005; received in revised form 9 August 2005; accepted 17 November 2005

Available online 18 January 2006

## Abstract

Membrane and feed characteristics are very important to understand the phenomenon of fouling in nanofiltration, as it is the interplay between membrane and feed that determines the fouling tendency. This paper describes a fouling study of three nanofiltration membranes: NF-270, NF-90 and BW30XLE.

In a first step, the crucial characteristics of the top layer of the membranes were determined: molecular weight cut-off (MWCO), hydrophobicity, surface charge and surface roughness. A comparison of the three membranes indicates that membrane NF-270 had the largest pores, was the most hydrophilic, had the smoothest surface and the highest surface charge density. Membrane BW30XLE had the smallest pores and the roughest surface, while membrane NF-90 was the most hydrophobic.

These characteristics were used in a second step to explain the observed fouling in the filtration experiments. As fouling depends also on the properties of the feed, several aqueous feed solutions were used containing either uncharged organic compounds, salts (NaCl, Na<sub>2</sub>SO<sub>4</sub> and CaCl<sub>2</sub>) or silica colloids. In all cases, the lowest fouling was observed for membrane NF-270, which can be explained by the hydrophilicity, the negative charge (when filtrating salts) or the smooth surface (when filtrating silica). Membrane NF-90 and BW30XLE have a comparable fouling tendency because the characteristics of these membranes are almost equal. Only when filtrating a high concentration of silica, membrane BW30XLE shows more fouling due to the very rough surface of this membrane.

Colloidal fouling was also studied in the presence of different NaCl concentrations, indicating that fouling increased with increasing ionic strength due to the shielding effect of the membrane and the colloid charge. This enables a more dense packing of the silica on the membrane surface and consequently more fouling was observed for the mixture of NaCl and silica in comparison with NaCl without the silica. However, the opposite was found when filtrating a mixture of silica and NaCl at low and high pH, because in this case the silica colloids cause an extra repulsion force resulting in less pore blocking and hence in less fouling.

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**Keywords:** Nanofiltration; Fouling; Characterization; Colloidal fouling; Salt

## 1. Introduction

Nanofiltration can be successfully applied in drinking water production [1–5], the textile industry [6–8], the food industry [9–12] and other industries [13–15]. However, the main draw-

back of nanofiltration (and membrane processes in general) is fouling, which results in an undesirable flux decline. The origin of this undesirable effect can be found in the interaction between the membrane and the compounds in the feed. A thorough knowledge of both membrane and compound properties is hence very useful to understand the phenomenon of fouling.

Van der Bruggen et al. [16,17] studied the problem of fouling in the case of filtrating aqueous solutions containing organic

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compounds and found that adsorption of organic material on the membrane surface is the major fouling mechanism. It was also shown that adsorption (and hence fouling) is mainly determined by the hydrophobicity of the membrane and the solute [18–20]. These hydrophobic interactions are also important in the fouling mechanism of pulp and paper mill waters, as shown by Manttari et al. [21].

When filtrating aqueous solutions of charged compounds (salts or organic acids), the membrane charge comes into the picture. Electrostatic repulsion and attraction are used in literature to explain the observed phenomenon of flux decline and fouling [22–24]. Hirose et al. [25] showed that an approximately linear relationship exists between the surface roughness of the membrane and the permeate flux, when filtrating a NaCl solution. They explained this effect by considering the unevenness of the top layer as an enlargement of the effective membrane area.

The opposite effect (i.e. a decreasing permeate flux with increasing roughness of the membrane) is, however, observed during filtration of colloidal solutions, which is explained by the fact that particles preferentially accumulate in the ‘valleys’ of rough membranes, resulting in ‘valley clogging’ and hence in a severe flux decline [26–28]. However, experiments by Van der Bruggen et al. [29] seemed to support the assumption that particle fouling is mainly determined by the hydrophobicity of the membranes. Beside the influence of the membrane characteristics, colloidal fouling is also determined by the nature of the solution, since changes in solution ionic strength [30–32] or pH [33] control the magnitude and range of the electrostatic double layer interactions of the colloids.

As shown in literature, membrane and feed characteristics are very important to understand the phenomenon of fouling. In this paper fouling is studied for three nanofiltration membranes. At first some important characteristics of the top layer of the membranes were investigated (molecular weight cut-off (MWCO), hydrophobicity, surface charge and surface roughness). The second part uses these characteristics to explain the observed fouling in the filtration experiments. As fouling depends on the properties of the feed, several feed solutions were used.

## 2. Materials and methods

Three nanofiltration membranes supplied by Dow/FilmTec (Edina, USA) were studied, namely NF-270, NF-90 and BW30XLE. All three membranes were made via the process of interfacial polymerisation. The membrane NF-270, made from piperazine and benzenetricarbonyl trichloride, is a typical nanofiltration membrane with applications in the drinking water production [34]. Benzenetricarbonyl trichloride is also a starting material for the membranes NF-90 and BW30XLE, but instead of piperazine, 1,3 phenylene diamine is used to complete the interfacial polymerisation. Although the latter two membranes are made of the same monomers, they differ in performance because of different processing conditions.

Table 1

Compounds used to determine the molecular weight cut-off together with the molecular weight ( $M_w$ ), the hydrophobicity ( $\log P$ ) and the determination method

	$M_w$	$\log P$	Determination
Methyl ethyl ketone (MEK)	72.12	0.26	Gas chromatography
Ethylacetate (EA)	88.12	0.86	
Isobutyl methyl ketone (BMK)	100.16	1.16	
Xylose	150.13	−1.98	Condensation reaction
Maltose	342.30	−5.41	and UV–VIS
Raffinose	504.42	−6.76	spectroscopy

### 2.1. Characterization of the membrane surface

The molecular weight cut-off of the membranes (MWCO or the molecular weight of the compound with 90% retention) was determined by doing filtration experiments with aqueous solutions of organic compounds with different molecular weights. As the experiments were done at neutral pH, the organic compounds were not ionized. The organic compounds are summarized in Table 1, together with their hydrophobicity.

The hydrophobicity of the membrane surface was analysed by contact angle measurements, performed with a Drop Shape Analysis System DSA 10 Mk2 (Krüss) in a three-phase system consisting of the membrane surface, air and water droplets of 2  $\mu$ l. The sessile drop method was chosen. Each contact angle was measured 10–15 times and an average value was calculated. The contact angle of the wet, clean membrane was determined for all membranes.

Non-contact atomic force microscopy (AFM) was carried out with an AFM system Autoprobe M5 (Park Scientific Instruments, presently VEECO). The cantilever was made out of Si with a spring constant of 3.2 N/m and a nominal tip apex radius of 10 nm. After scanning, the images were flattened with a second order polynomial approximation to remove AFM scanner-induced curvature and slope from the image. After flattening the RMS roughness (the root-mean-squared roughness or the standard deviation of the data) was calculated:

$$R_{\text{rms}} = \sqrt{\frac{\sum_{n=1}^N (z_n - \bar{z})^2}{N - 1}}, \quad (1)$$

where  $\bar{z}$  is the average of the  $z$  values within the given area,  $z_n$  the current  $z$  value and  $N$  is the number of data points within the given area. The roughness was measured for four different scan areas: 0.5  $\mu$ m  $\times$  0.5  $\mu$ m, 1  $\mu$ m  $\times$  1  $\mu$ m, 3  $\mu$ m  $\times$  3  $\mu$ m and 5  $\mu$ m  $\times$  5  $\mu$ m. The first scan was always made for the largest area (5  $\mu$ m  $\times$  5  $\mu$ m) and then this area was reduced till the smallest area of 0.5  $\mu$ m  $\times$  0.5  $\mu$ m was reached. Each measurement was performed three times to obtain a mean value of the RMS roughness. Several scan areas were used in order to improve comparison of surface roughness for different samples. Due to statistical reasons as well as to imperfections of the AFM scanning procedure (non-optimal sampling of the features) the roughness depends on the size of the scan area [35]. The same size scan range should therefore be used to compare the roughness for different samples.

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