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Ultrafiltration membrane cleaning with different chemical solutions after treating surface water

J.M. Arnal^{*}, B. Garcia-Fayos, M. Sancho, G. Verdu

Chemical and Nuclear Engineering Department, Polytechnic University of Valencia, Camino de Vera s/n, 46022 Valencia, Spain Tel. +34 (96) 3879633; Fax +34 (96) 3877639; email: jarnala@iqn.upv.es

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ABSTRACT

In recent years, application of pressure-driven membrane processes as ultrafiltration (UF) has expanded as an alternative promising technology to obtain drinking water for human consumption. Despite the strong potential of membranes, one of the common problems encountered in applications is membrane irreversible fouling. AQUAPOT project faces this common problem in the spiral-wound UF facilities that are located in Ecuador and Mozambique. Lack of chemicals and application of not optimized cleaning protocols have been revealed as the main causes that explain dramatic decrease in permeate flow observed in several installations. A simulation of this situation has been initiated in the Naquera Research Center (Valencia) with the aim of studying the main causes of irreversible membrane fouling due to long-term filtration of surface water. Physical, chemical (static and dynamic test) and physicochemical cleaning techniques have been studied to recover permeate flow of spiral-wound UF fouled membranes. This work describes the experimental procedure performed in chemical dynamic tests and the main results obtained for five different chemical solutions tested at two different temperatures (25 and 40°C). FTIR, SEM and EDX analysis of fouled and cleaned membranes have also been included so as to characterize the nature of membrane fouling in the gel layer and to evaluate cleaning efficiency of the chemical solutions and working conditions tested.

Keywords: AQUAPOT; Ultrafiltration; Chemical cleaning; Surface water; FTIR; SEM-EDX

1. Introduction

In recent years, application of pressure-driven membrane processes as ultrafiltration (UF) has expanded as an alternative promising technology to obtain drinking water for human consumption [1]. Despite the strong potential of membranes, one of the common problems encountered in applications is membrane fouling, which can significantly increase the energy consumption of the process over long-term operations [2].

Fouling is a process resulting in loss of performance of a membrane due to deposition of suspended or dissolved substances on its external surface, at its pore openings or within its pores [3]. Inorganics, biological foulants, suspended solids, colloids, metal oxides, and organics are the main species in the feed that contribute to fouling of membranes [4].

Membrane fouling occurs through one or more of the following mechanisms: (i) accumulation of solute and gradual irreversible changes in the polarised layer (such as cake formation), (ii) surface adsorption: deposition of solutes and (iii) adsorption: deposition of solute within the membrane. There are many factors contributing to fouling including surface properties (chemistry, morphology, etc.), hydrodynamic conditions, ionic strength and solute concentration [5].

^{*} Corresponding author.

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Membrane cleaning is an essential step in maintaining the permeability and selectivity of a membrane process. Cleaning can be defined as a process where material is relieved of a substance which is not an integral part of the membrane material [6]. Nowadays, cleaning techniques for membranes restoration could be broadly categorized into three types: physical, chemical, and physical-chemical methods.

Physical cleaning is based on mechanical treatment dislodges and removes foulants from the membrane surface. Physical methods include hydraulic cleaning, vibration, air sparge, and CO₂back permeation. Hydraulic cleaning methods are often adopted in UF for drinking water treatment [7].

Chemical cleaning depends purely on chemical interactions to remove foulants from the membrane surface. Chemical reactions involved in cleaning include hydrolysis, peptization, saponification, solubilization, dispersion and chelation [8]. There are five categories of chemical cleaning agents: alkaline solutions, acids, metal chelating agents, surfactants, and enzymes. Chemical agents react with deposits, scales, corrosion products and other foulants. The chemical should loosen and dissolve the foulant, keep the foulant in dispersion, avoid new fouling, and not attack the membrane [9].

Actually, not only cleaning is important to prevent fouling, several operating parameters as fouling including flux, concentrate velocity, backwash frequency, and transmembrane pressure have to be chosen depending on the water quality to insure a long-term operation of the membrane.

In the application of membrane technologies for drinking water treatment in remote areas, membrane fouling and cleaning are even more critical. The lack of suitable pretreatment, the difficulty of getting reasonable chemicals, and the application of not optimized cleaning protocols are the main reasons that limit the feasibility of these technologies in those regions [10].

This is the case observed in the AQUAPOT project [11,12]. In the last 3 years, several UF facilities have been installed in Ecuador (South America) and Mozambique (Africa) that allow producing drinking water in rural areas where the population has no access to it. Nowadays, the system represents a viable solution for community based on drinking water needs in the places where it is located.

After long-term operation, a considerable permeate flux decline has been observed as a consequence of not accurate cleaning and maintenance of the installation. To study the consequences that this fact can have over the UF membranes AQUAPOT has initiated a research at the Naquera Research Center (CIN) to investigate affordable and widely available cleaning reagents that allow recovering permeate flow of a UF membrane that suffered from irreversible fouling.

In the CIN, AQUAPOT prepared a spiral-wound UF

membrane that suffered from severe irreversible fouling (85% reduction in membrane permeability) caused by constituents in drinking water source, due to long-term (6 months) membrane filtration [13]. Fouling experiments were performed at a drinking water treatment facility placed in the CIN, similar to those installed in Ecuador and Mozambique. Cleaning protocols planned in the research work include physical, chemical (static and dynamic test) and physico-chemical techniques including the study of the influence of temperature, chemical concentration, pH, pressure, flow and time over membrane cleaning [14].

The present paper studies the effect of cleaning strategies in flux recovery performing dynamic tests over a previously fouled UF membrane. Chemicals used were selected from the best chemical solutions obtained in previous static tests [15]. In these previous experiments, some widespread cleaning chemicals were tested: sodium hypochlorite, sodium hydroxide, hydrogen peroxide, citric acid and cupric acid; as well as one commercial agent, Auxiclean B.13. The best cleaning performance in the static tests was observed with hydrogen peroxide, sodium hypochlorite and Auxiclean B.13, which are some of the chemicals used in the experiments of this work.

The main results obtained from this study will be applied in the installations that AQUAPOT has in Ecuador and Mozambique where fouling is commonly managed by local technicians.

2. Materials and methods

2.1. Feed water characteristics and fouling characterization

A UF spiral-wound membrane placed at the CIN was fouled after six month operation with surface water coming from an irrigation pond. The main parameters of the analytical composition of this raw water are shown in Table 1.

Membranes fouling was reached after a 6 month longterm filtration period of surface water in which membranes worked at a transmembrane pressure of 0.4 MPa and at a feed flow of 1200 L/h with a permeate recovery rate of 60% and operated continuously during 24 h/d. The installation had as a pretreatment a cylindrical screen filter with a 6 mm pore size, a prefilter with a pore size between 3 and 0.8 mm and previous to UF membranes a sand filter of about 200 microns. As described in the previous works on cleaning, backwashing was used by compressed air (0.5 bar), clarified water or both [13].

Before starting the cleaning procedures, an autopsy of the polysulfone fouled membrane was done with the aim of determining the nature of fouling in the gel layer which would help to select the best cleaning protocols to be applied in the experiments. Fouled UF membranes (with a cut-off of 100 KD) were removed from the spiral-wound module and divided into three sections, perpendicular Table 1

Analytical composition of surface water used for fouling a UF membrane

Parameter	Value
Turbidity, NTU	12.4
Conductivity (20°C), µS/cm	385
pH	8.44
Alkalinity (CaCO ₃), mg/L	160
Total hardness, mg/L	88
Nitrate, mg/L NO ₃ Fe	8
Cr (VI), mg/L	< 0.02
Ca, μg/L	<5
Mg, mg/L	57.6
Microbiological, mg/L	14.6
Aerobic (22°C), CFU/1 mL	32,400
Total coliform, CFU/100 mL	890
Escherichia coli, CFU/100 mL	10
Clostridium perfringens, CFU/100 mL	1050

to the feed-water flow direction and cut in rectangular pieces of 30 cm² before being used in the dynamic tests at a pilot plant scale [15].

2.2. Pilot plant description

All the dynamic tests have been performed in a UF pilot plant shown in Fig. 1.

The membrane module (M) comprises four flat membranes with an effective area of 30 cm² each. The membranes are placed in series, so that the retentate of each one turns into the feed of the following membrane. Chemical cleaning solutions are prepared in the cleaning tank (CT) and pumped to the membrane module. The permeate is collected in the ultrafiltrate tank (UFT), while the cleaning solution is returned to the cleaning tank (CT). The temperature remains constant during all the experiments by using heat exchangers (C and H) located in cleaning (CT) and ultrafiltrate tanks (UFT).

The permeate flow was measured through P1, P2, P3 and P4 valves by using a graduated cylinder and feed flow was measured with a flowmeter (5).

2.3. Cleaning procedure in dynamic tests

Experimental procedure consisted in the following three stages:

1) Water permeability before cleaning. Membranes were tested to determine the initial water permeability with deionised water at a transmembrane pressure of 0.2 MPa. All the samples were taken from the same spiral-wound UF module, from different positions in the module representing the whole membrane area. Four membrane samples were tested at the same time. The permeate flow of each sample was measured every 15 min during 1 h. Each experiment was done three times. The average permeate flux was then calculated for each membrane.

2) *Chemical cleaning*. The chemicals used in the experiments (Table 2) were chosen according to the membrane manufacturer's recommendations, bibliography consulted [6,16–18], as well as a consequence of the results obtained in the previous static tests [15]. The different chemical solutions were recirculated during 2 h at a transmembrane pressure of 0.2 MPa and with a feed flow



Fig. 1. Flow diagram of the UF pilot plant used in the dynamic tests. H: Heating system, C: Cooling system, RV: Pressure regulation valve, CT: Cleaning tank, UFT: Ultrafiltrate tank, F: Pump filter, M: Membrane module, M1-M4: Permeate collector, P00: Manometer, T: Thermometer, P0: Manometer, 1,2: Valves, 3: Centrifugal pump, 4: Safety valve, 5: Flowmeter.

Table 2 Chemical solutions used in the cleaning experiments

Solution Concentration pH Auxiclean B.13 (*) 2% (w/v) 11 Auxiclean A1.6 (*) 5% (v/v) 2 Sodium hypochlorite (NaClO) (**) 100 ppm 11 Hydrogen peroxide (H ₂ O ₂) (**) 0.5% (v/v) 7.7 Sodium hydroxide (NaOH) (**) 0.001 M 11			
Auxiclean B.13 (*) 2% (w/v) 11 Auxiclean A1.6 (*) 5% (v/v) 2 Sodium hypochlorite (NaClO) (**) 100 ppm 11 Hydrogen peroxide (H_2O_2) (**) 0.5% (v/v) 7.7 Sodium hydroxide (NaOH) (**) 0.001 M 11	Solution	Concentration	pН
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Sodium hypochlorite (NaClO) (**) 100 ppm 11 Hydrogen peroxide (H_2O_2) (**) 0.5% (v/v) 7.7 Sodium hydroxide (NaOH) (**) 0.001 M 11	Auxiclean A1.6 (*)	5% (v/v)	2
Hydrogen peroxide (H_2O_2) (*) 0.5% (v/v) 7.7 Sodium hydroxide (NaOH) (*) 0.001 M 11	Sodium hypochlorite (NaClO) (**)	100 ppm	11
Sodium hydroxide (NaOH) (**) 0.001 M 11	Hydrogen peroxide (H ₂ O ₂) (**)	0.5% (v/v)	7.7
	Sodium hydroxide (NaOH) (**)	0.001 M	11

(*) Supplier Auxicolor S.A.

(**) Supplier PANREAC Spain

of 160 L/h. The chemical solutions tested were the ones determined as the best ones in the previous static tests [15], as it was mentioned in the Introduction, with the addition of Auxiclean A1.6, another commercial solution from the membrane supplier. The permeate flow was measured every 15 min for 2 h, and it was later used for calculating the permeate flux of each sample.

3) Water permeability after cleaning. After chemical cleaning, water permeability with deionised water was again determined in order to compare it with the initial value and to calculate the degree of flux restoration and the recovery (in percentage) of the membrane permeability. The operation conditions were the same as those in the first stage.

This experimental procedure was carried out twice for each solution at two different temperatures: 25°C and 40°C. Cleaning conditions shown would not cause damage to the membrane since the concentrations examined in this study were within the ranges recommended by the membrane manufacturer. After each experiment, cleaned membranes were replaced for new fouled samples.

2.4. Analytical methods

In this experiment, fouled and cleaned membranes were analyzed using the Fourier transform infrared (FTIR) technique. FTIR spectra were obtained by using FTIR spectrophotometer (Model 710, Nicolet, Madison, USA). Both membrane and fouling layer were simultaneously analysed.

Fouled and cleaned membranes were also analysed with scanning electron microscopy (SEM) technique by using SEM microscope (JEOL-JSM 5410) equipped with an EDX analysis system (Link Isis, Oxford Instruments; conditions were 90 s, 20 kV, 200× magnification) to determine the inorganic components in the gel layer. Data from EDX analysis were processed with the Inca Point & ID software.

3. Results and discussion

3.1. Membrane fouling characterization

3.1.1. FTIR analysis

Fig. 2 shows a representative spectrum of the membrane fouling, in which the following peaks can be emphasized: two peaks of adsorption around 3600 cm⁻¹, which are attributed to stretching of the O–H bond in hydroxyl functional groups, and smaller peaks at 2921 and 2842 cm⁻¹, which are due to symmetric and asymmetric C–H stretching vibrations (non-aromatic, linear CHstructure) [19]. In addition, a sharp peak at 1026 cm⁻¹ can be attributed to C–O stretching of alcoholic compounds which most likely originated from polysaccharides and polysaccharide-like substances or Si–O bonds of silicate materials, respectively [20]. Peaks at 1494 and 1405 can be assigned to aromatic ring stretching vibrations. Another peak appears at 550 cm⁻¹ in the finger print region of the spectrum, confirming the presence of structural units



Fig. 2. The FTIR spectra of the fouled membrane.

of the aromatic sulfone. As it is known, the fouled UF membrane is made of polysulfone and is characterized by repeating units of diphenylene sulfone.

From the FTIR spectra, it can be seen that membrane foulants include polysaccharides and natural organic matter (NOM), very common when treating surface water.

3.1.2. SEM-EDX analysis

SEM photograph (Fig. 3a) reveals that the fouled membrane was covered with a slime gel layer. Once the gel layer was developed, it would become difficult to remove the layer from the membrane surface. Consequently, the formation of the gel layer resulted in the increase of TMP and caused severe irreversible membrane fouling in this study as it was previously explained. Element analysis was further performed on the surface layer in order to identify the chemical components of the layer by EDX analysis. The elements Mg, Fe, Al, K, Ca, S and Si were detected as shown in Fig. 3b.

Si, Fe, Al, K and Ca had appreciative effects on the formation of the gel layer since the relative contents of these inorganic elements were significant.

3.2. Membrane cleaning at dynamic experiments

(a)

The effectiveness of the cleaning procedure in removing fouling and recovering membrane performance was determined by comparing permeate fluxes before and

Fig. 5. Degree of flux restoration in the experimental tests at 40°C.

1.00

1.50

 J_1/J_0

2.00

2.50

3.00

after chemical cleaning. Figs. 4 and 5 show the degree of the restoration of the fouled membranes in terms of the pure water flux by chemical cleaning with a single reagent. The ratio of the pure water flux after chemical cleaning (I_1) to the flux before chemical cleaning (I_0) is used to express the degree of flux restoration:







Fig. 4. Degree of flux restoration in the experimental tests at 25°C.

Auxiclean A 1.6

Auxiclean B.13

NaClO

Water

NaOH

H2O2

0.00

0.50

 $T = 40^{\circ}C$



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Degree of flux restoration =
$$\frac{J_1}{J_0}$$
 (1)

According to the results presented in Fig. 4, at a temperature of 25°C the best chemical solution is the commercial Auxiclean B.13 which shows a degree of flux restoration above 1.5. Sodium hypochlorite and hydrogen peroxide show acceptable flux restoration values of 1.34 and 1.11 on average, respectively; while NaOH showed very low values for degree of flux restoration — around 1.05. A significant effect of Auxiclean A 1.6 was not recognized in this study. This is because it was recommended for removing inorganic fouling and, as it has been shown in the spectrum of Fig. 2, the fouling was mainly caused by organic matter. Thus, it was rejected as a possible chemical solution for cleaning the UF membrane used in the treatment of surface water.

At a temperature of 40°C, as it is shown in Fig. 5, the best chemical solution was also the commercial solution Auxiclean B.13 with a degree of flux restoration of 1.94. In this case, the hydrogen peroxide showed very good performance in permeate flux restoration with a value of 1.76. On the other hand, at this temperature, sodium hydroxide showed poor values of permeate flux recovery, very similar to those obtained at 25°C. Lastly, sodium hypochlorite did not show any permeability recovery with values of permeate flux before cleaning very similar to those after cleaning.

Finally, Table 3 includes the average rate of membrane recovery (*R*) calculated as:

Recovery (%) =
$$\left(1 - \frac{J_0}{J_1}\right) \cdot 100$$
 (2)

Comparing the results of membrane recovery shown in Table 3 for different temperatures, it is clear for the cases of Auxiclean B.13 and H_2O_2 that the increase of tem-

Table 3 Membrane permeability recovery by chemical cleaning

	R (%)	
	25°C	40°C
Auxiclean B.13	35.3	48.7
Auxiclean A.16	-31.7*	-17.1^{*}
NaClO	25.4	-2.2*
H,O,	10.4	43.1
NaOH	4.7	8.2
Water	0.8	-1.1^{*}

*Means no recovery of membrane permeability

perature from 25°C to 40°C improves membrane permeability recovery till values of 48.7 and 43.1% respectively.

After analysing the experimental results, it can be said that commercial solution Auxiclean B.13 is the best chemical agent from the point of view of membrane permeability recovery.

3.3. Analysis of cleaned membranes by FTIR and SEM

FTIR and SEM-EDX analysis was done from the membrane sample which produced the best results on cleaning test done by dynamic tests, Auxiclean B.13.

3.3.1. FTIR analysis

In Fig. 6, adsorption peaks in the spectrum of the cleaned membrane are shown at about 2800, 2900, 3030, 3057 and 3456 cm⁻¹.

In fact, a broad region of adsorption around a peak at 3456 cm⁻¹ has been attributed to stretching of the O–H bond in hydroxyl functional groups by previous researchers [19]. Moreover two peaks at 3030 and 3057 cm⁻¹ ap-



Fig. 6. FTIR analysis of the membrane cleaned with Auxiclean B13.

peared that can possibly be assigned to C–H aromatic stretching vibrations.

The peaks at 2800 and 2900 cm⁻¹ are due to the symmetric and asymmetric C–H stretching vibrations (non-aromatic, linear CH-structure) [19].

Finally, all other peaks below 1500 cm⁻¹ clearly correspond to the structure of the polysulfone membrane.

Comparing Fig. 2 with Fig. 6, it can be observed that not all organic fouling has been removed with chemical cleaning, even though groups attributed to polysaccharides and polysaccharide-like substances present in NOM or silicates and aromatic ring have been effectively removed.

3.3.2. SEM-EDX analysis

(a)

Element analysis was further performed on the surface layer in order to identify the chemical components of the layer by EDX analysis. The elements of Cl, S, Al, Si and Ca were detected and are shown in Fig. 7b.

The main element is S that corresponds to the presence of polysulfone due to its being the main compound of the membrane material. Cl, Si, Al and Ca are the main inorganic foulants detected in EDX analysis of the cleaned membrane with Auxiclean B.13.

Comparison between Figs. 3 and 7 reveals that the cleaning reagent is able to remove inorganic fouling due to Mg, Fe, K and Zn, but adds Cl. The membrane also

maintains fouling due to Al, Si and Ca which could not be removed by using this commercial alkaline reagent.

The results clearly show that not all of the fouling has been removed during cleaning, even though Auxiclean B.13 is the most effective in recovering permeate flow of the fouled membrane, reaching values of 35.3% at 25°C and 48.7% at 40°C.

4. Conclusions

According to the results previously analyzed, the following conclusions can be stated:

- FTIR, SEM and EDX analysis proves that the membrane surface was covered with a compact gel layer formed by organic substances and inorganic elements such as Mg, Al, Ca, Si or Fe coming from the surface water that caused irreversible fouling.
- The commercial solution Auxiclean B.13 is the most effective chemical for cleaning a polysulfone UF membrane used for treating surface water. However, this commercial solution is difficult to get in rural areas of developing countries where AQUAPOT project is being developed.
- Hydrogen peroxide gives satisfactory results at both tested temperatures (much better at 40°C), and it is a wide spread chemical. So, it is selected as the most suitable chemical agent within the tested ones to be used in the cleaning process of UF membranes when treating surface water.

Moreover, further investigation should be carried out with this chemical agent in order to define a proper cleaning protocol to be applied to spiral-wound UF modules. Physical and physical-chemical cleaning protocols will also be implemented to optimize membrane cleaning and remove fouling due to long-term filtration of surface water. Finally, the optimized cleaning protocol will be applied in AQUAPOT facilities located in Ecuador and Mozambique where this project has been implemented.



Fig. 7. (a) SEM photograph of the cleaned membrane surface 200× magnification (b) EDX analysis of the membrane cleaned with Auxiclean B.13.

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