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Chemical Cleaning of Utafiltration Membrane after Treatment of Oily Wastewater

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Abstract

Oily wastewaters and Oil–in-water emulsions are two of the major pollutants of the environment. Ultrafiltration (UF) membranes play an important role in the treatment and reuse of oily wastewaters. Fouling of UF membranes is typically caused by inorganic and organic materials present in wastewaters that adhere to the surface and pores of the membrane and result in the deterioration of performance with a consequent increase in energy costs and membrane replacement. In the experiments, polyacrylonitrile (PAN) and outlet wastewater of the API (American Petroleum Institute) separator unit of Tehran refinery as membrane and feed were used, respectively. Fouling and cleaning experiments were performed with oily wastewater and selected cleaning agents using a laboratory scale cross flow test unit. The results showed that metal chelating agent (ethylene diamine tetra acetic acid disodium salt-2-hydrate (EDTA)) and an anionic surfactant (sodium dodecyl sulfate (SDS)) were able to clean the fouled UF membrane effectively by optimizing chemical (pH) and physical (cleaning time, cross flow velocity (CFV) and temperature) conditions during cleaning. Flux recovery and resistance removal were found to improve with increasing CFV, temperature, pH, cleaning time and concentration of the cleaning chemicals. In this paper, the cleaning mechanism is also investigated.

Keywords: *Oily Wastewater, Cleaning, Ultrafiltration, Fouling*

1- Introduction

UF membranes are widely used in the treatment of oily wastewaters, domestic sewage and industrial applications. The use of UF membranes in advanced oily wastewater reclamation using pretreatment wastewater effluent to produce water for indirect potable (industrial applications) use has also increased over the past few years. However, a major problem in the application of UF membrane technology for the treatment of oily wastewaters and domestic sewage reclamation is membrane fouling [1-3]. Control of fouling is of utmost importance [4, 5], and depends on the foulants available in the feed stream. Oily wastewater contains some potential membrane fouling categories, lubricants, cutting liquids, heavy hydrocarbons (tars, crude oils, grease and diesel oil), and light

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hydrocarbons (kerosene, jet fuel and gasoline), microbial (bacteria, viruses, etc.), and inorganic (minerals) contents [2, 3]. Depending on the physicochemical properties of the membrane, the composition of the feed solution and the process conditions, the membrane loses its performance with time. Fouling not only decreases the permeation flux but also changes the rejection of solutes [2]. Foulants, or fouling layer, are general terms for deposits on or into the membrane that adversely affect filtration. Fouling can involve several distinct stages, desirable or undesirable, reversible or irreversible [3, 5]. Although several techniques are involved such as the pretreatment of feed, which reduces particulate density and membrane regeneration, chemical cleaning operation conditions such as temperature, trans-membrane pressure (TMP) and CFV can be considered to reduce fouling [2, 3, 6].

Developing strategies for fouling control has always been a major challenge in membrane research. However, despite the many preventive strategies, fouling is inevitable. A long term solution would be to remove the foulant deposited on membrane surfaces via chemical cleaning [7]. Membrane cleaning is performed when there is a significant drop in permeate flux or salt rejection, or when there is a need to increase the TMP significantly to maintain the desired water flux [7, 8].

There are two strategies that are usually employed to minimize the effect of fouling. The first group includes minimizing fouling by using adequate feed pretreatment, membrane treatment and membrane modification. The second group involves membrane remediation by chemical cleaning which is carried out to restore membrane fluxes [9, 10].

An important technique for membrane regeneration is the chemical cleaning of fouled membranes [6]. Many substances, mostly chemicals, and different procedures are used for cleaning membranes. Chemical cleaning means removing impurities by means of chemical agents. However, cleaning consumes time and money. In general, around 5–20% of the operating cost is the cost of cleaning. This shows the importance of continuous research in this field [6, 11, and 12].

In this paper the chemical cleaning of UF membranes fouled with oily wastewater is investigated. The fouled membranes were washed with chemical agents such as acid, alkali and surfactant. The type of chemical agent and process conditions i.e. concentration of the cleaning solution, cleaning time etc., affect cleaning efficiency. The effects of these parameters on cleaning efficiency as well as the cleaning mechanism are discussed.

2- Material

2.1- Membrane

In all the experiments, polyacrylonitrile (PAN) from the Sepro membrane was used as the UF membrane. Characteristics of the membranes are presented in Table 1.

2.2- Process feed

Outlet of the API separator unit of Tehran refinery was used as the feed. The feed was taken daily and used immediately. Analysis of the feed taken from the wastewater of the API separator unit is presented in Table 2.

Table 1. Characteristics of the polymeric membrane

Membrane				Recommended operating limits		
Series	Name	Material	MWCO	pH range	Pressure range (bar)	Temperature range (°C)
PAN350	PAN	Polyacrylonitrile	20 kDa	1.5 – 10.5	1 - 10	0 - 100

Table 2. Characteristics of the process feed

Parameter	Unit	Feed
Total suspended solids (TSS)	mg/L	60
Total dissolved solids (TDS)	mg/L	2028
Oil and grease content	mg/L	78
Chemical oxygen demand (COD)	mg/L	124
Biological oxygen demand (BOD_5)	mg/L	52
Total organic carbon (TOC)	mg/L	81
Turbidity	NTU	53

3- Experimental

3.1- Experimental method

Fig. 1a shows the experimental set up used in all the experiments. The UF cell was made of two pieces of Teflon (Fig. 1b). These two parts were sealed by O-rings and the rectangular membrane (64 cm^2) was placed between them. It must be mentioned that for each experiment a new piece of membrane was employed. During the experiments, supervision was carefully done to control CFV, TMP, temperature and pH. The permeate was collected for 2.5 h and the pure water permeation flux (L/m^2h) was calculated using the following equation: J=V/At where A is the membrane area, V is the collected permeate volume and t is h. All of the adjustments and measurements for the UF experiments were the same.

3.2- Analysis of samples

Samples for measurements of the feed and the permeate total suspended solids (TSS), biological oxygen demand (BOD_5), chemical oxygen demand (COD), oil and grease content, turbidity, total organic carbon (TOC), and total dissolved solids (TDS) contents were taken as necessary and analyzed by the procedure outlined in standard methods [13]. TOC and turbidity were estimated using a TOC Analyzer (Model DC-190) and turbidimeter (Model 2100A HACH), respectively.

3.3- Fouling and cleaning procedures

Fouling and cleaning were quantified via measurements of the resistance (R) before and after cleaning the membranes. The resistance is due to the formation of a cake or gel layer on the membrane surface. Fouling and cleaning were evaluated using resistance removal (RR) and flux recovery (FR) as follows [6, 10]:

$$FR\ (\%) = [(J_{wc}- J_{ww})/(J_{wi}- J_{ww})]\times 100$$

where J_{wi}, J_{ww} and J_{wc} are the permeation flux of the fresh membrane, that of the fouled membrane, and that of the chemically cleaned membrane, respectively.

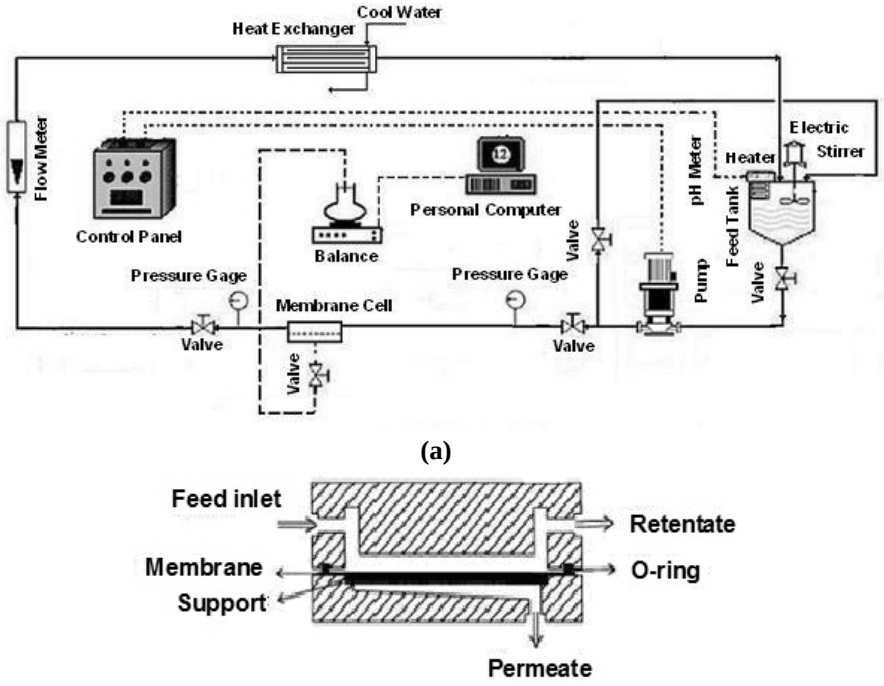

Figure 1. UF experimental set up (a) and UF cell (b)

RR (%) = $[(R_f-R_c)/R_f] \times 100$

where R_C and R_f are the resistance of the chemically cleaned membrane and that of the fouled membrane, respectively.

4- Results and discussion

This section is focused on the types of membrane fouling processes that lead to deterioration of the plant performance and their subsequent recovery using membrane cleaning processes. Different types of inorganic and organic fouling have been addressed in this study. Fouling problems lead to higher operation costs, higher energy demand, reduce the lifetime of the membrane and increase the cleaning frequency. Our work also reveals that it regenerates the membrane performance, even though some cleaning methods have potential limitations.

The success of chemical cleaning methods depends on many factors such as the nature of the foulant, type of cleaning agents, temperature, pH, the concentration of the cleaning chemicals, cleaning time and operation conditions such as CFV. These factors affect the outcome of the cleaning procedure and therefore need thorough investigation in order to establish the optimum cleaning system. Conventional assessment of cleaning by flux measurement has been used in recent decades in order to optimize and evaluate the cleaning procedures. At present there are several modern surface analysis techniques that can assist precisely and rapidly in optimizing the cleaning processes.

4.1- Comparison of cleaning agents

Based on the analysis of the feed, four categories, alkalis, acids, metal chelating agents and surfactants were selected. Fouling of UF membranes in oily wastewater treatment is mostly due to the precipitation of oil and grease, suspended solids, colloidal materials and minerals on the membrane surfaces. The analysis of the feed is presented in Table 2.

To compare the cleaning agents, similar fouled membranes were cleaned with different chemicals. The concentrations of all the cleaning solutions were 5 mM. Fig. 2 showed the cleaning efficiency as FR for various chemicals. Acids were the weakest cleaning agents for the experimental conditions. Alkali had a moderate effect, while EDTA, which is a chelating agent, has a good ability to combine with metals. It is used in special soaps to remove metallic concentration. The effect of SDS (surfactant) can be attributed to the cleaning strength of emulsifiers due to altering the interfacial tension of water. This results in a better separation of fouling materials from the membrane surface. NaoH changes the pH of the solution and provides a better condition for the highest removal of foulants with EDTA and SDS. Cleaning experiments were performed using cleaning solutions containing different concentrations of SDS and / or EDTA, as presented in Figs. 3 and 4, respectively. The results clearly show that cleaning efficiency using SDS and EDTA increases with increasing the cleaning agent concentration. It can be observed that cleaning efficiency increases sharply until a concentration of 4 mM SDS and 30 mM EDTA, after which there is no significant change. Using a more concentrated EDTA cleaning solution can cause the chemical reaction between EDTA and the deposited materials to break down a cake/gel layer network. Cleaning efficiency of the different cleaning solutions (combination of 4 mM SDS and 30 mM EDTA) are compared in Fig.5. The results show that cleaning with a combination of EDTA and SDS is relatively more effective. This is due to the fact that SDS has both hydrophobic and hydrophilic groups, and is semi soluble in both organic and aqueous solvents. Surfactants can solubilize macromolecules by forming micelles around them, and help to remove the precipitated materials from the membrane surface. Also, EDTA can remove divalent cations from complex organic molecules and improve the cleaning efficiency of the fouled membrane. Generally, SDS is more responsible for removing oil and grease, while EDTA removes minerals from the membrane surface.

Table 3. Statistical Analysis for chemical cleaning as response

Trial no.	CFV (m/s)	pH	Temperature (^oC)	Cleaning time (min)	Replicated trials (%)			$\overline{y_u}$	$\sum(y_{ui}-\overline{y_u})^2$	DOF	$t\times S/\sqrt{n}$	$\overline{y_u}\pm t\times S/\sqrt{n}$
					y_{u1}	y_{u2}	y_{u3}					
1	1.25	8	25	10	68.4	70.1	74.3	70.9	18.4	6	4.1	70.9±4.1
2	0.75	10	40	10	61.9	65.8	63.7	63.8	7.6	6	0.1	63.8±01
3	1.25	10	40	30	92.4	95.2	90.7	92.7	10.3	6	2.5	92.7±2.5

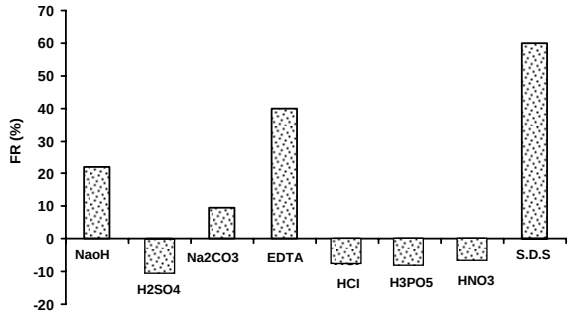

Figure 2. Effect of cleaning agents on FR (cleaning instruction: CFV = 1.25 m/s, temperature = 25 °C, pH = 10, cleaning time = 20 min)

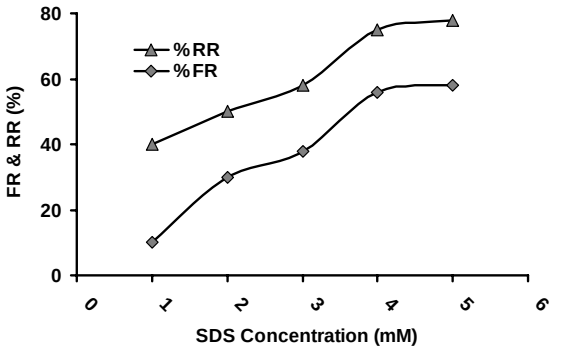

Figure 3. Effect of SDS concentration on FR and RR (Cleaning instruction: CFV = 1.25 m/s, temperature = 25 °C, pH = 10, cleaning time = 20 min)

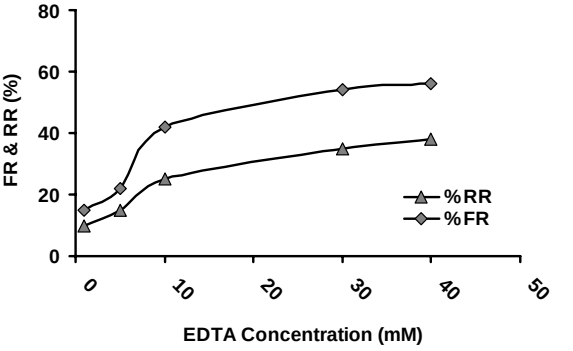

Figure 4. Effect of EDTA concentration on FR and RR (cleaning instruction: CFV = 1.25 m/s, temperature = 25 °C, pH = 10, cleaning time = 20 min)

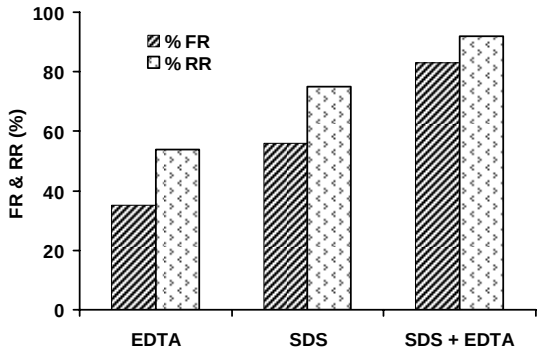

Figure 5. Effect of cleaning agent on FR and RR (cleaning instruction: CFV = 1.25 m/s, temperature = 25 °C, pH = 10, cleaning time = 20 min)

4.2- Factors affecting chemical cleaning efficiency

Cleaning mainly involves the dissolution of the material from the membrane surface and several factors could affect the chemical cleaning process. These are: temperature, pH, cleaning time and operation conditions such as CFV.

4.2.1- Effect of CFV (hydrodynamic shear)

Hydrodynamic cleaning conditions such as CFV play an important role in membrane FR. Lee et al. [14] and Madaeni and Samieirad [2] reported that in an experiment carried out using a fouled UF membrane cleaned with caustic chemical cleaning at low and high CFV, it was found that the cleaning efficiency increased at high CFV compared to low CFV. On the other hand, Bartlett et al. [15] noticed that an increase in CFV while cleaning seems to have a minimal effect on membrane permeation flux fouled with whey protein. The effect of CFV on cleaning efficiency was also investigated, as shown in Fig.6. As can be observed, cleaning efficiency increases with increasing CFV till 1.25 m/s, then remains almost constant. Increasing CFV, which causes higher shear

rates, enhances the mass transfer of the cleaning agent through the deposited materials on the membrane surface, thus increasing the cleaning efficiency. The cleaning efficiency increased with increasing CFV only when the chemical reaction between the foulant and the cleaning agents was high enough to produce a favorable reaction. Otherwise, an increase in CFV which results in an increase in the shear rate does not enhance the mass transfer of foulant in the fouling layer to the bulk solution. It can be calculated that the chemical reaction between the cleaning agent with deposited fouled and associated mass transfer phenomena are quite important in membrane cleaning.

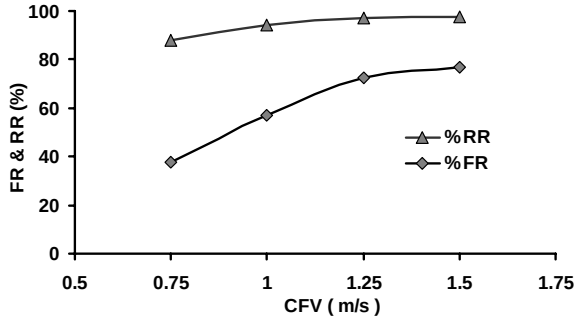

Figure 6. Effect of CFV on FR and RR (cleaning instruction: temperature = 25 °C, cleaning time = 20 min, pH = 10)

4.2.2- Effect of cleaning solution pH

The pH of the cleaning solution affects the recovered permeate flux of the fouled membrane with humic acid. Mänttäri et al. [16] showed that at a lower pH the membrane and humic acid are almost uncharged and that these conditions promote fouling. Li et al. [17] reported that the cleaning efficiency of EDTA depend critically on the pH of the solution as a result of deprotonation of functional groups. Ang et al. [18] and Mohammadi and Kazemimoghadam [10] reported that the influence of solution pH on EDTA cleaning efficiency had a remarkable effect. The effect of pH on the cleaning efficiency of the cleaning agent (a combination of SDS and EDTA) is illustrated in Fig.7. It is shown that cleaning efficiency increases with increasing pH from 8 to 11. The higher chelating ability of EDTA with increasing pH results in a more effective ligand-exchange reaction between ETDA and alginate-metals complexes within the alginate cake/gel layer. Consequently, the alginate cake/gel layer is broken down relatively more easily, resulting in a higher cleaning efficiency. It was concluded that the cleaning solution pH is a governing factor affecting the chemical reaction between deposited fouled and EDTA, whereas the chemical reaction between deposited foulant and SDS is less influenced by cleaning solution pH. The best pH should be selected according to the higher cleaning efficiency and greater chemical stability. Thus, a pH of 10 can be an optimum value.

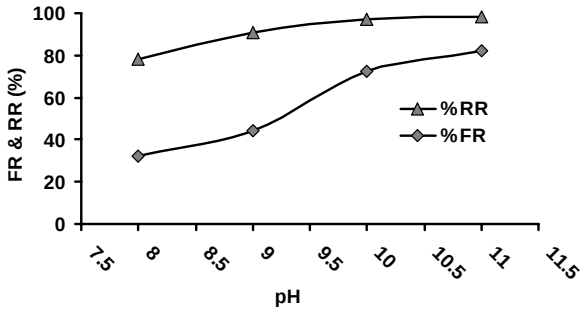

Figure 7. Effect of pH on FR and RR (cleaning instruction: CFV = 1.25 m/s, temperature = 25 °C, cleaning time = 20 min)

4.2.3- Effect of temperature

The temperature of the chemical cleaning solution may also play an important role in the chemical system. Generally, increased temperature increases the cleaning efficiency, presumably by increasing the transport process and the solubility of the material. However, the sensitivity of membrane materials usually prohibits the use of a very high temperature. Generally, membrane manufacturers recommend that chemical cleaning be carried out at a temperature lower than 45°C. Zondervan and Roffel [1] and Li et al. [17] reported that preheating the cleaning solution up to about 40^{o}C had a significant impact on increased FR compared to 25^{o}C, where the increased cleaning temperature was favorable for the desorption of deposits from the membrane surface. Madaeni et al. [6] and Chen et al. [19] noticed that one significant factor which can affect the chemical cleaning of the UF membrane is the cleaning temperature. Ang et al. [18] have shown that, the FR increased dramatically with increasing the temperature from 20 to 40 ^{o}C in the case of reverse osmosis (RO) membrane. The results of the cleaning agent (a combination SDS and EDTA) at different temperatures are presented in Fig. 8. Cleaning efficiency increases dramatically with increasing temperature. This is due to the fact that both the rate of the chemical reaction between the cleaning agent and the deposited materials and the rate of diffusive transport of the deposited materials from the cake/gel layer back to the bulk solution increase as the temperature increases. A cleaning temperature of 45°C can be recommended for the cleaning procedure. Therefore, both the rate of the chemical reaction of EDTA with deposited fouled and the diffusive transport of foulant from the fouling layer to the bulk solution increased as the temperature was increased.

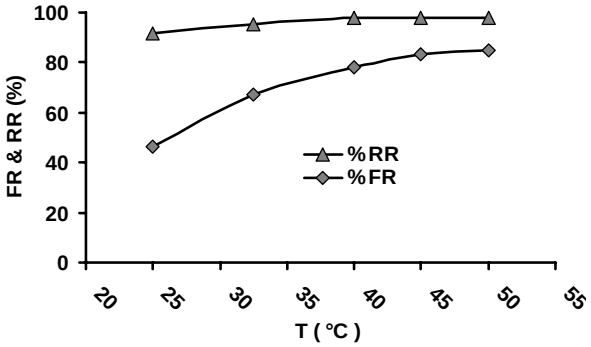

Figure 8. Effect of temperature on FR and RR (cleaning instruction: CFV = 1.25 m/s, pH = 10, cleaning time = 20 min)

4.2.4- Effect of cleaning time

Hydraulic cleaning conditions, such as cleaning time, may play an important role for FR at actual plants. A longer cleaning time at a lower velocity was found to be more effective in removing the fouled (oil and grease, suspended solids, colloidal materials and minerals) from membrane surfaces. In applications, a cleaning time of 15 min is enough, while in other applications of membranes more time is needed for cleaning, about 1 h or even longer to reach their maximal cleaning effect in order to restore the membrane performance. In a recent study, Madaeni and Samieirad [2] and Li et al. [17] noticed that increasing cleaning time from 10 to 20 min for chemical cleaning had greater power on FR. Ang et al. [18] in the case of RO membrane reported that, the cleaning efficiency of EDTA increased dramatically when the cleaning time increased from 15 to 60 min. The cleaning

efficiency of SDS at low concentration with a cleaning time of 15 or 60 min was not effective. The effect of cleaning time on the cleaning efficiency is presented in Fig. 9. According to these results, the longer the cleaning time, the higher the cleaning efficiency. This is due to the favorable chemical reaction between the cleaning agent and the deposited materials in the cake/gel layer which needs some time to proceed. A cleaning time of 30 min can be recommended for the cleaning procedure.

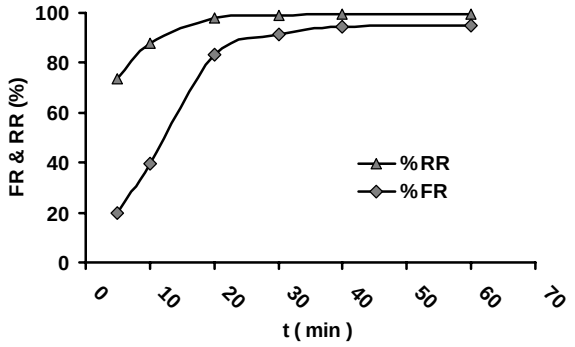

Figure 9. Effect of cleaning time on FR and RR (cleaning instruction: temperature = 45 °C, CFV = 1.25 m/s, pH = 10)

4.3- Cleaning mechanism

Membrane cleaning involves both chemical and physical interactions. These interactions include (i) chemical reaction between the cleaning agent and the fouled in the fouling layer and (ii) the mass transfer of cleaning of the cleaning agents from the bulk solution to the fouling layer and the fouled from the fouling layer back to the bulk solution. A schematic representation of the roles of the chemical and physical interactions for the effective cleaning of the fouled UF membrane is shown in Fig. 10. Effective cleaning can be achieved only when both the chemical and physical interactions are favorable as discussed below.

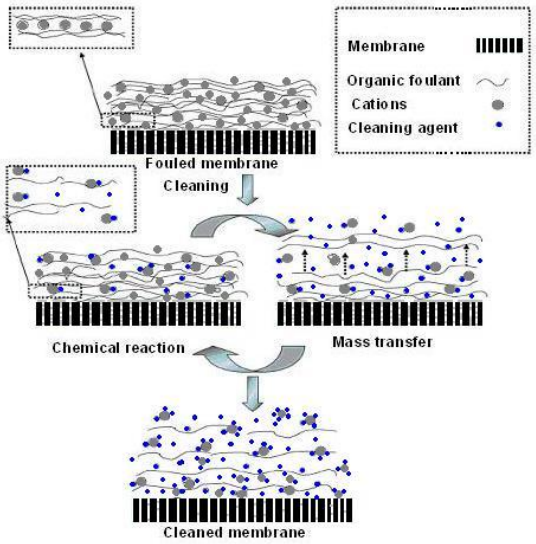

Figure 10. Schematic representation for effective cleaning of organic-fouled UF membranes. A cross-linked fouling layer is formed on the membrane surface in the presence of calcium ions, which bind to organic foulants and form bridges between adjacent foulant molecules. During cleaning, the cleaning agent reacts with the foulants in the fouling layer yielding loosened foulants. These reaction products are removed from the fouling layer to the bulk solution through the hydrodynamics/mass transfer. Thus, Efficient cleaning can be achieved through the coupling between the chemical reaction and mass transfer, along with the optimization of cleaning conditions responsible for the favourable chemical reaction and mass transfer [9, 19].

In the presence of a cleaning solution, chemical reaction will occur between the cleaning agent and the fouled in the fouling layer. In this study, we have seen that the effectiveness of cleaning in terms of chemical reactively depends on the type of cleaning solutions, cleaning solution pH and the foulant chemical composition. When a cleaning agent has a favorable chemical reactivity, the cleaning agent, upon contact

with the foulants, will be able to react with the foulants and weaken the structural integrity of the fouling layer. The physical (hydrodynamic) conditions, which are mainly responsible for the mass transfer of the reaction products, then play an important role in removing the foulants from the fouling layer. The cleaning agent reacts with the attached deposited materials in the fouling layer, yielding to weakening them. The chemical reaction between the cleaning agent and the deposited materials in the fouling layer takes place and then the products diffuse from the membrane surface back to the bulk solution**.** The reaction may be hydrolysis, dissolution or dispersion. These finally result in the removal of the deposited materials from the membrane surface.

4.4- Statistical Analysis

Trial error and experimental error also belong to the group of random errors, so that in estimating their values we use the same approach as for random measurement errors. In determining a measurement error we take into account the number of replicate measurements (u); in a trial error the number of replicate trials (n), and in an experimental error the number of different trials (N). Replication of a trial must not be mixed up with replication of measurements in another trial. When determining a trial error, we estimate the standard deviation of replicated trials. This may be estimated by calculating the standard deviation of several trials whose control factor settings are the same. Ideally, one would set up and run the same trial repeatedly. The small differences in a setup are an important component of the replicate error. The replicate error is made up of two parts: trial error and measurement error. The replicate variance is just the square of the replicate standard deviation. In the case of experimental error we estimate the variance of reproducibility. It is obligatory to include the trial error when comparing values of two different trials, since if the difference of the trials is lower than its error, one may not speak of a better or worse value of the trial. An assumption on the normal distribution of replicated trials enables the determination of the average arithmetic value of response for each trial of experimental and trial error variance [20]:

$$\overline{y_u} = \frac{\sum_{1}^{n} y_{iu}}{n}$$

$$S^2 = \frac{\sum_{1}^{n} \left(y_{iu} - \overline{y_u}\right)^2}{n-1}$$

where:

S^2-is the trial error;

n-is the number of trial replications

y_{iu}-are response (FR in this work) values in the replication of the u-th trial;

$\overline{y_u}$ -is the arithmetic response average in the replication of the u-th trial (outcome of one u-th trial).

S^2 value characterizes the trial reproducibility.

The error square average of a trial has the well-known form[20]:

$$S = +\sqrt{\frac{\sum_{1}^{n} \left(y_{iu} - \overline{y_u}\right)^2}{n-1}}$$

An increase in S^2 or S values characterizes the larger dispersion of the trial outcomes around the average ($\overline{y_u}$). The deviation of the average $\overline{y_u}$ may be estimated from the real value of a trial (y_u) with significance level (α): $t \times S/\sqrt{n}$

$$\overline{y_u} - t \times S/\sqrt{n} \le y_u \le \overline{y_u} + t \times S/\sqrt{n}$$

The operating conditions under which the chemical cleaning UF process was carried out are given in Table 3. Statistical Analysis was applied to calculate response average, trial error and error square average of each run on response (FR). In the present work, 3 runs were selected to evaluate the statistical (error) analysis. Error committed in the measures was inferior to 5%.

5- Conclusions

EDTA and SDS were quite effective in reacting with fouled in the fouling layer formed in the presence of calcium ions, while NaoH cleaning results in poor cleaning efficiency due to its limited reactivity with deposited foulants. Cleaning efficiency with EDTA and SDS was improved by optimizing the cleaning agent concentration and solution pH. However, these chemical factors hardly contributed to improving the efficiency of NaoH cleaning. For favorably reactive cleaning agents, cleaning efficiency can be further improved by enhancing the mass transfer of the reaction product from the fouling layer to the bulk solution. Mass transfer of the foulants from the fouling layer to the bulk solution was mainly controlled by the CFV, whereas cleaning time and temperature affected both the mass transfer and chemical reaction.

The best cleaning agent to enhance the cleaning efficiency of the fouled UF membrane was found to be a combination of SDS and EDTA. A combination of SDS (as a surfactant) and EDTA (as a chelating agent) as a powerful cleaning agent performed very effectively. EDTA is able to combine with metals. The effect of SDS can be attributed to the cleaning strength of emulsifiers due to their ability to alter the interfacial tension of the water. The cleaning efficiency of the recommended cleaning agent was further improved by optimizing the cleaning condition. The results showed that a CFV of 1.25 m/s, a temperature of 45 °C, a cleaning time of 30 min and a pH of 10 are the best cleaning conditions.

The effectiveness of chemical cleaning in terms of chemical reactivity depends on the type of cleaning solution, cleaning solution pH, the cleaning chemicals concentration and the ionic strength, while the mass transfer of the foulant from the fouling layer to the bulk solution is mainly controlled by the CFV, and cleaning time and temperature affect both the mass transfer and the chemical reaction.

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