Influence of water compositions on fouling of plane organic membrane in frontal filtration: application to water and wastewater clarification

P. Sridang, P. Wanichapichart and A. Grasmick

ABSTRACT

The aim of this study was to evaluate and quantify the filterability of suspended/soluble organic and suspended inorganic matter in a condition without and with chemical conditioning on membrane fouling using cake filtration model. The experiments were conducted with different feed water concentrations under a given TMP (0.2 to 0.5 bar). The fouling potential was examined and described in terms of resistance coefficient ($\alpha{\cdot}W$) and specific resistance (α). The results showed an increase of $\alpha{\cdot}W$ and α within the concentration of wastewater samples tested. The soluble fractions in wastewater induced fouling and its mechanism was due both to the interaction of soluble organic components and also some of the particular colloids in MLSS, causing irreversible fouling, followed by thin film formation on membrane surfaces with low porosity, dense structure and also internal fouling. This phenomenon promoted the values of $\alpha{\cdot}W$ and α from final treated wastewater 5–20 times higher than in bentonite suspension and on reservoir surface water. Higher pressure than 0.2 bar induced greater hydraulic resistance values than lower applied pressure. The pore size of the porous membrane did not show any difference in the values of $\alpha{\cdot}W$ and α obtained, but they mostly depended on the water composition tested. The hydraulic resistance values appeared largely to minimise when using chemical conditioning because of cake forming as a dynamic membrane that reduced the irreversible fouling phenomena giving a constant filtration rate.

Key words | clarification, filterability, frontal filtration, water and wastewater

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INTRODUCTION

Nowadays, pressure driven membrane processes are widely applied for producing high water quality, whatever the water resource, and for treating wastewater (Nicolaisen 2002; Howell 2004). Micro-Ultrafiltration is a technological development to clarify water and replace conventional water clarification. Also used for external separation and/or immersion inside a biological reactor for wastewater treatment, is an emergent technology, known as the membrane bioreactor system (MBR). Its performance is well known in terms of efficiency of pollutant removal with high physical and chemical qualities and thus favourable for water reuse onsite (Gomez *et al.* 2001; Howell 2004). Micro-Ultrafiltration operates under very flexible filtration modes, including frontal filtration or a tangential mode, when the suspensions contain a large range of compounds obliging specific conditioning to minimise fouling (Lebeau *et al.* 1998; Rautenbach & Voßenkaul 2001; Schäfer *et al.* 2001; Sridang *et al.* 2005).

In water treatment most fouling is caused by suspended solids and colloid fractions deposit on the membrane surface and/or by pore blocking. The organic matters are also important in drinking water fouling. Meanwhile, membrane fouling in wastewater treatment is due to both bacterial suspensions in the MBR system and soluble microbial products from cell lyses reaction, causing

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concentrated polarization layers. It causes cake formation on the membrane surface, steric overloading or progressive pore clogging and interaction of soluble compounds with the membrane material or with the other elements in the suspension (Belfort *et al.* 1994; Ognier *et al.* 2002; Sridang *et al.* 2006*a*). The importance of operational conditions is recommended to limit and control fouling potential, for instance sub-critical conditions or adapted hydrodynamic conditions such as tangential shear stresses increase or back pulsing regeneration, module-system design and suspension conditioning can be defined to avoid macroscopic deposits from building up on the membrane surface (Field *et al.* 1995; Sridang *et al.* 2005, 2006*b*; Yang *et al.* 2006). However, the membrane permeability also decreases slightly during the operation in comparison with clean membranes according to the macromolecules, such as proteins, polysaccharides and organic colloid fractions. These, created from microbial metabolism and its activities in biological suspension with membrane material, are still the major limitations. Then the biological process control not only plays a key role to provide improvement of treatment efficiency, but also enhances the filtration performance in the MBR system (Belfort *et al.* 1994; Chang & Fane 2001; Gomez *et al.* 2001; Ben Aim & Semmens 2002; Howell 2004; Sridang *et al.* 2008).

To predict or evaluate the membrane fouling potential according to the numerous and various compounds implied in membrane fouling, mostly reversible fouling from cake deposits, during industrial filtration scales, is essential as reference information. The objective of this work is to quantify and evaluate the influence of water compositions and the impact of operational conditions on the membrane fouling dynamics by measuring specific hydraulic resistance using the cake filtration model.

MATERIALS AND METHODS

Experimental set up and membrane

To predict or evaluate the membrane permeability decrease according to the numerous and various compounds implied in membrane fouling, the fouling potential of feed suspensions was studied and its filterability was examined in the frontal filtration mode. To evaluate the filterability of the suspension, we supposed that the determinant step reducing the membrane permeability was cake formation due to particle retention on the membrane surface. The experimental set-up, shown in Figure 1, is a lab scale filtration unit with plane organic membranes. The unit consisted of a pressurized filtration cell with a working volume of 150 ml. The characteristics of each membrane used are given in Table 1 and Figure 2. Cake filtration theory was used to examine and explain the effect of the various components in the suspension tested. The methodology consisted in following up the cumulated volume of filtrate during

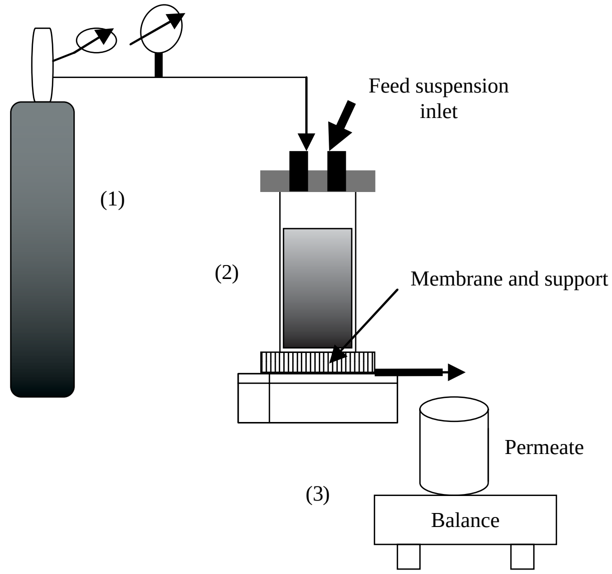

Figure 1 | Schematic diagram of frontal filtration unit set-up. (1) = N_2 tank equipped with pressure regulator, (2) = Pressurized filtration cell and (3) = Balance and permeate receiver.

Table 1 | Membrane characteristics

	GSWP 04700	VMWP 04700
Type	Plane	Plane
Membrane material	Mixed cellulose ester	Nitrocellulose
Dimension (mm, diameter)	47	47
Filtration area (cm^2)	11.9	11.3
Pore size (μm)	0.22	0.05
Porosity (%)	75	72
Thickness (μm)	180	105
Water permeability (20°C, 1 bar) (l. h^{-1} m^{-2})	10800	400
Membrane resistance R_m (m^{-1})	2.5×10^6	0.9×10^{12}

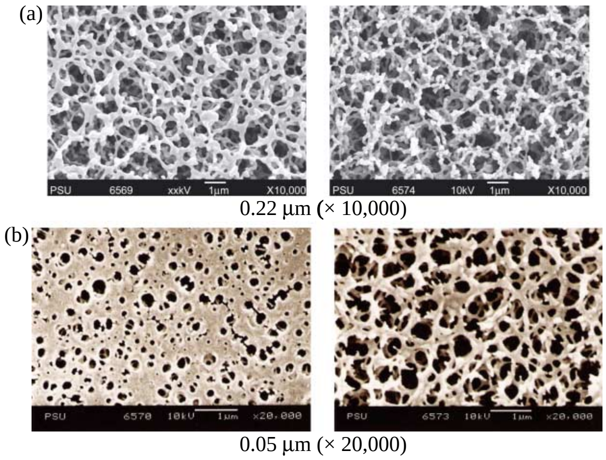

Figure 2 | SEM analysis of GSWP and VMWP 04700 Membrane.

filtration time for given transmembrane pressures (TMP) at 0.5 and 1 bar without any applied turbulence. The changes in the filtered volume can be described by cake filtration theory, in which the ratio t/V is a linear function of V:

$$\frac{t}{V} = \frac{\mu \times \alpha \times W}{2 \times \Delta P \times \Omega^2} \times V + \frac{\mu \times R_m}{\Delta P \times \Omega} \quad (1)$$

where

W	particle concentration (kg m^{-3})
T	time (s)
ΔP	transmembrane pressure (Pa)
α	specific resistance (m kg^{-1})
R_m	initial membrane resistance (m^{-1})
μ	dynamic viscosity (Pa s)
V	cumulated volume of filtrate (m^3)
Ω	membrane area (m^2)

The linear plot of t/V with V was used to calculate the product αW estimated and/or the specific cake resistance α as the quantitative degree of the fouling potential of the suspensions tested.

Studied suspensions and experimental conditions

The type of feed composition and concentration was conditioned by coagulation/adsorption. Real wastewater, from the activated sludge treatment plant in the seafood processing industry, bentonite and surface water reservoir were used (Table 2). The conditioning of clay suspension and surface water with $FeCl_3$ were executed in a lab scale Jar-Test unit, which was expected to provide various mixing conditions, rapid (150 rpm for 1 min) and slow velocity (40 rpm for 15 min) coagulation and flocculation. The effect of adsorption with powder activated carbon was investigated for final effluent in rapid mixing conditions for 12 h, operated by the Jar-Test unit (Table 3–4). The filtration performances were evaluated according to the quality of filtered samples compared to the initial characteristics of feed samples. Different parameters were

Table 2 | Studied suspension characteristics

Type of feed	pH	SS (mg/L), Turbidity * (NTU)	COD (mg/L)	TKN (mg/L)	Protein concentration β-lactoglobulin standard (mg/L)	Mean particle size (µm)
1. Influent wastewater	6.5 ± 0.3	1400 ± 500 500 ± 50*	3800 ± 100	150 ± 100	300 ± 200	Not analysed
2. MLSS	8.0 ± 0.5	600 ± 100	Not analysed	Not analysed	150 ± 100	47 (2.4–1300)
3. Clarified water from sedimentation tank	8.0 ± 0.3	240 ± 20	350 ± 150	120 ± 10	10–300	Not analysed
4. Effluent discharged	8.5 ± 0.5	80 ± 40	200 ± 20	80 ± 50	10–300	Not analysed
5. Bentonite suspension						
SS = 500 mg/L	7–8	78	152	Not analysed	Not analysed	27.74 (0.545–194.2)
SS = 5000 mg/L	7–8	1100	1085			15.95 (0.598–83.89)
6. Surface water	6.8 ± 0.5	27.0 82*	170	0.219	Not analysed	Not analysed

Table 3 | Studied operating conditions in fresh water samples

Type of feed	Type/membrane pore size (μm) and feed concentration (mg/L- SS and chemical conditioning) GSWP 04700, Millipore 0.22 μm (P = 0.2 and 0.5 bar)	VMWP 04700, Millipore 0.05 μm (P = 0.2 and 0.5 bar)
1. Suspended solids of chemical coagulants		
$FeCl_3$ suspension(mg/L)	20, 100	20, 100
Alum suspension (mg/L)	20, 100	20, 100
2. Bentonite suspension (SS-mg/L) and chemical conditioning doses used (mg/L)	500, 5000, 500 + 20 $FeCl_3$, 500 + 20 Alum 500 + 100 $FeCl_3$, 500 + 100 Alum, 5000 + 200 $FeCl_3$, 5000 + 200 Alum 5000 + 500 $FeCl_3$, 5000 + 500 Alum	
3. Surface water (NTU) and chemical conditioning doses used (mg/L)	Real value- NTU Real value-NTU +20 $FeCl_3$, real value NTU +50 Alum Real value-NTU +200 $FeCl_3$, real value NTU +200 Alum	
4. Effluent discharged (COD-mg/L) and chemical conditioning doses used (mg/L)	200, 200 + 20 $FeCl_3$, 200 + 500 $FeCl_3$, 200 + 50 PAC, 200 + 200 PAC 200 + 20 $FeCl_3$ + 200 PAC, 200 + 20 $FeCl_3$ + 1000 PAC 200 + 20 $FeCl_3$ + 2000 PAC	

Remark: the experiments were tested in the same operating conditions for both membranes.

analyzed in the feed samples and the permeate effluent through the important parameters: turbidity, color, COD, TKN, MLSS, NOM-soluble proteins-particle size distribution along with the analytical methods used (APHA *et al.* 2005).

RESULTS AND DISCUSSIONS

Figures 3–5 and Table 5 present the evolutions of t/V vs. V and $\alpha{\cdot}W$ values obtained from three different filtration experiments: influent wastewater, microbial suspended solids (MLSS) and clarified wastewater tested. These were studied with the objective of showing the influence of the composition and concentration of suspension.

The values of $\alpha{\cdot}W$ when filtering all suspensions considered in the same conditions studied increased with the concentration of COD and suspended solids tested. When filtering influent wastewater and raw microbial suspension the $\alpha{\cdot}W$ values from 0.05 μm were lower than, or some of them were equivalent to, the values obtained from 0.22 μm at any TMP applied. The effect of TMP values did not show any negative impact to increase the values of $\alpha{\cdot}W$ and α when using 0.5 bar for influent wastewater and clarified water from the sedimentation tank. This implied that the structure of cake on membrane surface was

Table 4 | Studied operating conditions in wastewater samples

Type of feed	GSWP 04700, Millipore 0.22 μm P = 0.2 bar	GSWP 04700, Millipore 0.22 μm P = 0.5 bar	VMWP 04700, Millipore 0.05 μm P = 0.2 bar	VMWP 04700, Millipore 0.05 μm P = 0.5 bar
	Type/membrane pore size (μm) and feed concentration in SS (mg/L) or COD (mg/L)			
1. Influent wastewater (COD-mg/L)	500	500	500	500
	3000	3000	3000	3000
2. MLSS (mg/L)	500	500	500	500
	1000	1000	1000	–
	2500	–	2500	–
3. Clarified water from sedimentation tank (COD-mg/L)	50 250	50 250	50 250	50 250

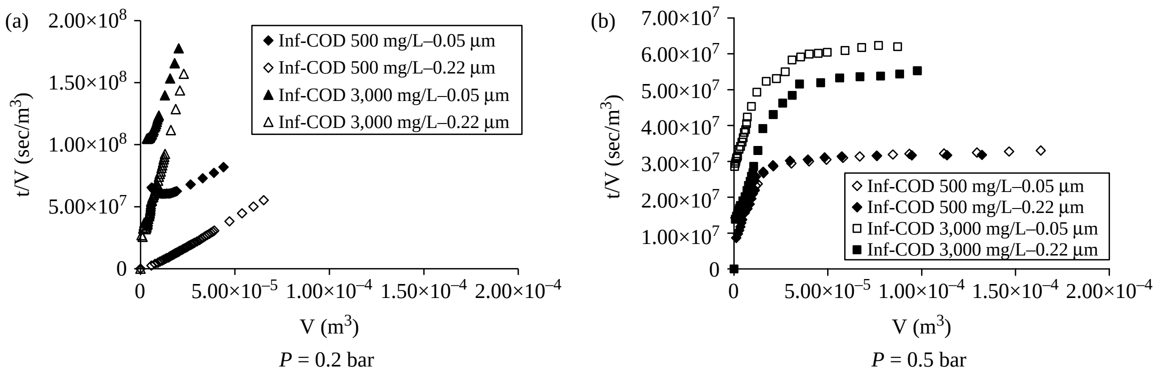

Figure 3 | Evolution of t/V vs. V for influent wastewater samples.

dynamic during filtration and/or that the residual soluble fraction was too small and could pass or penetrate through membrane pores. The values of $\alpha{\cdot}W$ and α obtained from the raw biomass suspension (MLSS, particular and soluble fractions of biomass suspension) with 0.5 bar was higher than values obtained from 0.2 bar for a concentration of 500 and 1,000 mg/L, while using different pore sized membranes was not so different in $\alpha{\cdot}W$ and α values when filtering the biomass suspension at a low TMP of 0.2 bar. In addition, the values of α obtained were independent from the initial concentration of MLSS tested at 0.2 bar for both membranes used. At the same time the filtration of 500 and 1,000 mg/L-MLSS with 0.5 bar and 0.05 µm showed that the value of α increased 2 times with concentration and was 2 times higher than the experiments tested with 0.22 µm. This is because high external fouling occurred because most of the particular fraction could be retained totally on the membrane surface faster than soluble fractions could form or penetrate. When filtering clarified water from the sedimentation tank, the $\alpha{\cdot}W$ and α values from 0.05 µm were higher than, or some of them were equivalent to, the values obtained from 0.22 µm at any TMP and the COD concentration applied. The effect of TMP at 0.2 and 0.5 bar on the values of $\alpha{\cdot}W$ and α was similar when filtration was done at a low COD of 50 mg/L with 0.22 µm membranes tested, while filtration of 250 mg/L-COD with 0.5 bar showed that the values of $\alpha{\cdot}W$ and α were 3–5 times lower than the operation at 0.2 bar for both membranes tested. Since raw water contains a large fraction of inorganic suspended solids, the bentonite suspension was chosen as representative of surface water.

Table 6 presents the evolution of t/V vs. V and $\alpha{\cdot}W$ values when filtering bentonite suspension, 500 and 5,000 mg/L, for different experimental tested conditions, both with and without chemical conditioning. The results showed that the specific hydraulic resistance criteria ($\alpha{\cdot}W$)

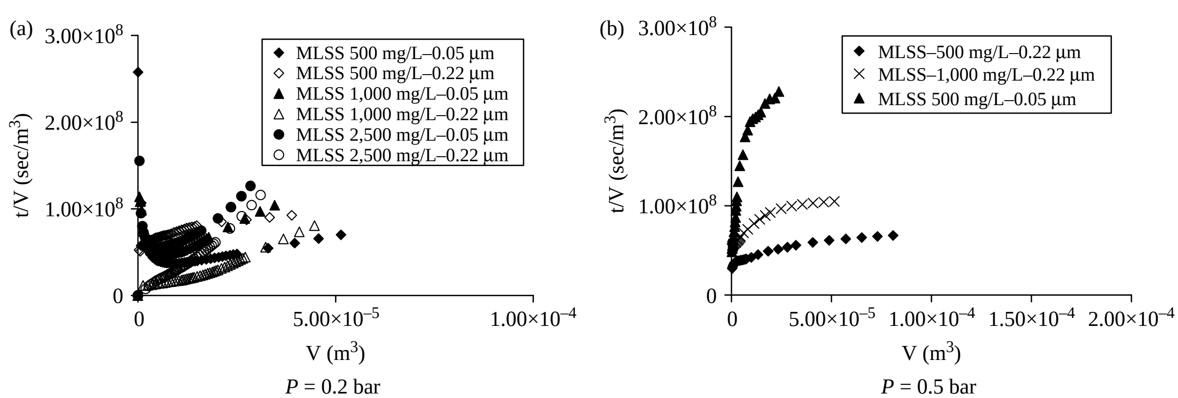

Figure 4 | Evolution of t/V vs. V for Mixed Liquor Suspended Solids (MLSS).

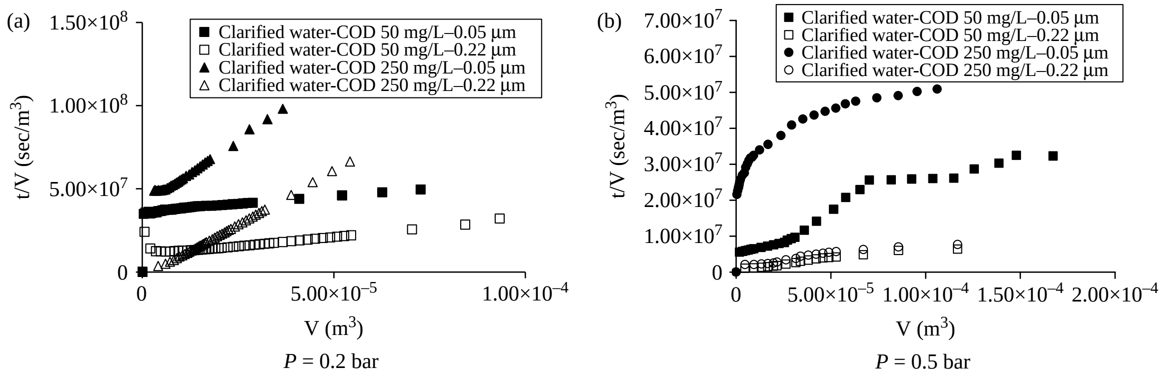

Figure 5 | Evolution of *t/V* vs. *V* for Clarified water from sedimentation tank.

appeared as directly proportional to the suspended particle concentration when filtration was carried out without conditioning, α being proportional to the particle concentration. The small membrane pore size did not have a significant effect on hydraulic resistance values obtained when filtration was done at a low suspended solids concentration of 500 mg/L, while the pressure applied at 0.5 bar for all conditions tested showed the relatively high values of $\alpha{\cdot}W$ and α. After the ferric chloride and alum solutions in the coagulation/flocculation process were performed in bentonite suspension, the result showed the benefit of such conditioning to transfer small particles into the floc suspension which induced a significant reduction of the specific resistance criteria ($\alpha{\cdot}W$ and α) by a factor of 2 to 20 or more when working with high concentrated suspensions of 5,000 mg/L. However, it was found that the relatively small values of $\alpha{\cdot}W$ and α were increased and were significant when there was the presence of large cake porosity and weak deposit compactness in the chosen working conditions. The presence of such a cake deposit on the membrane surface represented a dynamic layer which avoided any entering of small particles into the membrane pores. It had a positive impact of reducing irreversible membrane fouling if it occurred.

When filtering effluent from the Songklanagarind hospital wastewater treatment plant for different tests both with and without ferric chloride and/or PAC conditioning, due to residual COD and SS still present in effluent after

Table 5 | αW and α values obtained for wastewater studied

	αW (10^{12}, m^{-2})/α (10^{12}, $m\,kg^{-1}$)			
	***P* = 0.2 bar**		***P* = 0.5 bar**	
Samples	**0.22 µm**	**0.05 µm**	**0.22 µm**	**0.05 µm**
1. Influent wastewater (COD-mg/L)				
500	51/102	28/56	10/20	14/28 (99)
3000	340/113	220/73	71/24 (280/93)	57/19 (280/93)
2. MLSS (mg/L)				
500	60/120	48/96	57/114	120/240 (2800/5600)
1000	110/110	110/110	140/140	–
2500	170/68	170/68	(280/280)	–
3. Clarified water from sedimentation tank (COD-mg/L)				
50	11/220	11/220	10/200	28/560 (28/560)
250	60/240	110/440	9/36	43/172

Remark: (…) values at beginning run with maximum slope occurred.

Table 6 | αW and α values obtained for fresh water samples studied

Samples	Chemical conditioning doses used (mg/L)	αW (10^{12}, m^{-2})/α (10^{12}, $m\,kg^{-1}$) $P = 0.2$ bar 0.22 μm	0.05 μm	$P = 0.5$ bar 0.22 μm	0.05 μm
1. Floc suspension (mg/L)	$FeCl_3 = 20$	0.23/11.5	2.30/11.50	0.56/28	85/4250
	= 100	0.55/5.50	2.80/28	1.40/14	14/140
	Alum = 20	0.23/11.50	5.10/255	0.56/28	5.60/280
	= 100	0.51/5.10	1.70/17	0.85/85	11.30/113
2. Bentonite suspension (mg/L)					
500	$FeCl_3$, Alum = 0	4.50/9	1.10/2.20	8.50/17	2.80/5.60
	$FeCl_3$ = 20	0.23/0.44	2.80/5.40	0.85/1.60	5.60/10.80
	= 100	0.40/0.66	3.40/5.70	0.85/1.40	4.20/7
	Alum = 20	0.34/0.65	45/86.5	0.56/1.10	56/107.70
	= 100	0.23/0.38	1.10/1.80	0.42/0.70	28/46.60
5000	$FeCl_3$, Alum = 0	110/22	110/22	140/28	280/56
	$FeCl_3$ = 200	5.10/0.98	11/2.10	11.30/2.20	8.50/1.60
	= 500	2.80/0.51	40/7.30	0.056/0.01	5.60/1
	Alum = 200	5.10/0.98	23/4.40	11.30/2.20	99/1.90
	= 500	0.45/0.08	23/4.40	0.99/0.18	8.50/1.60
3. Surface water (mg/L)	$FeCl_3$, Alum = 0	1.70/17	3.40/34	2.80/28	2.80/28
	$FeCl_3$ = 20	0.28/2.30	1.10/9.20	0.56/4.60	1.40/11.60
	= 200	0.55/1.80	3.40/11.30	1.10/3.60	1.40/4.60
	Alum = 50	0.40/2.60	3.40/22.60	0.56/3.70	8.50/56.60
	= 200	2.80/9.30	1.10/3.60	1.40/4.60	7.10/23.60
4. Effluent discharged (COD 200 mg/L, SS 88 mg/L)	$FeCl_3$, PAC = 0	110/5500	5.50/275	140/7000	140/7000
	$FeCl_3$ = 20	34/850	11/275	71/1775	280/7000
	= 500	3.40/6.60	45/86.50	8.50/16.40	42/80.80
	PAC = 50	40/571.40	1.70/24.30	85/1214	5.60/80
	= 200	40/181.80	110/500	85/386.40	42/190.90
	$FeCl_3$ 20 + PAC 200	–	–	113/470.80	99/412.50
	$FeCl_3$ 20 + PAC 1000	–	–	85/81.70	56/53.80
	$FeCl_3$ 20 + PAC 2000	–	–	56/27.50	99/48.50

treatment at a conventional wastewater treatment plant, by the aerated lagoon system, the alternative process using coagulation and adsorption is a classical conditioning process recommended for the improvement of effluent prior to filtration by porous membrane for reclamation purposes. The filterability tests showed that the values of $\alpha{\cdot}W$ were quite different between effluent and influent suspensions (COD 500 mg/L) when the filtration was done for both pressure and membrane pore size tests. The resistance values of effluent discharged was higher by 2–10 times than the values found when filtering influent wastewater. Even its COD and SS were lower than in the influent sample. This implied that soluble fraction inducing adsorption and blocking in the pores play a key role in cake formation. In contrast, the benefit of ferric chloride and powder activated carbon addition could reduce greatly the values of $\alpha{\cdot}W$ and α obtained by a factor of around 2–20 times. Membrane separation showed a very good efficiency of retaining suspended solids and solutes in all suspensions tested.

The physical quality of filtered water was in good to excellent levels with turbidity lower than 0.5 NTU with the COD removal at about 50–90%. The combination of coagulation and adsorption for filtering effluent discharged samples produced a very good quality due to the natural organic matter in terms of UV absorbance 254, and the humic acid concentration was reduced by about 60–70%. The reduction rate of COD was between 50–60% and a total color removal of 90% was achieved. In any case, the membrane in cellulose ester (0.22 μm) presented lower hydraulic resistance values than nitrocellulose which can translate (1) the deposit cake resistance to be higher in comparison with initial membrane resistance and (2) the importance of the membrane material in fouling intensity.

CONCLUSIONS

A laboratory-scale filtration unit with plane organic membranes was used to conduct filtration with different types of feed and concentration under given TMP (ranging from 0.2 to 0.5 bar) without any applied turbulence. Fouling potential and filterability were examined and described by cake filtration law in terms of resistance coefficient ($\alpha \cdot W$) and specific resistance (α). The results showed an increase of $\alpha \cdot W$ and α in the concentration of wastewater samples tested. The values of $\alpha \cdot W$ and α obtained from the total composition of Mixed Liquor Suspended Solids (MLSS) compared to the soluble fraction from MLSS showed similar values. This soluble fraction induced fouling and its mechanism was due both to the interaction of soluble organic components and also some of the particular colloids in MLSS causing irreversible fouling of thin film formation on membrane surfaces with low porosity, dense structure, pore reduction and blocking. The evaluation of the filterability of the bentonite suspension, reservoir surface water and final treated wastewater both with and without chemical conditioning showed an increase in $\alpha \cdot W$ and α when using the concentration of feed water without any conditioning methods. It was found that the values of $\alpha \cdot W$ and α from final treated wastewater were 5–20 times higher than in the bentonite suspension and reservoir surface water tested. Higher pressure than 0.2 bar induced greater $\alpha \cdot W$ and α values than lower pressure applied. The pore size of porous membranes did not show any difference in the values of $\alpha \cdot W$ and α obtained, but they depended on the type of feed water tested. It was observed that high resistance values were in the same range as that for bentonite suspension and reservoir surface water when they were filtered with 0.22 μm and 0.05 μm in both applied pressures. Feed water, bentonite suspension, reservoir surface water and final treated wastewater, conditioned by adding ferric chloride, alum and/or activated carbon, showed similar resistance values. The values of $\alpha \cdot W$ and α were lower than the values obtained from feed water without conditioning because of low porosity and lower density of cake structure forming reversible fouling. The cake layer represented the secondary filter giving a constant filtration rate. Finally, the optimization of the membrane process should be considered and controlled. The significant operating conditions such as biological reaction, water and wastewater conditioning prior to the filtration process can maintain system performances and its efficiency for continuous filtration runs.

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