

Removal of heavy metals from electroplating wastewater by membrane

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Abstract

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This research was to study the treatment of heavy metals in electroplating wastewater using membranes. Two selected membrane types, cellulose acetate microfiltration membrane with pore size 0.2 μm and polysulfone ultrafiltration membrane with MWCO of 30 kDa were used in this study. Synthetic and factory electroplating wastewater were used as the samples. The experiments were performed by chemical precipitating both synthetic and factory wastewater in the first step and membrane filtrating of supernatant at the pressure of 50, 100 and 200 kPa in the second step. The concentration of chromium, copper, nickel and zinc of treated water were compared with standard values given by the Ministry of Industry (MOI), Thailand.

The experimental results showed that flux was highest at the pressure of 200 kPa and decreased as the pressure decreased. The rejection was highest at the pressure of 50 kPa and decreased as pressure increased. The results from synthetic wastewater were better than those from factory wastewater. The capability of heavy metal removal of microfiltration and ultrafiltration membrane was the same, but microfiltration gave more flux.

The heavy metal removal efficiency of microfiltration of synthetic electroplating wastewater of four processes of chromium, copper, nickel and zinc electroplating, each was higher than that from factory wastewater but slightly lower than the removal efficiency obtained from composite synthetic wastewater.

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The removal efficiency of chromium, copper, nickel and zinc from composite synthetic wastewater was higher than those from composite factory wastewater for both microfiltration and ultrafiltration processes.

The results from the study of membrane surface washing showed little flux increase after washing the membrane by stirring with a propeller at a distance of 2 mm above membrane surface at 400 rpm for 30 minutes.

Key words : membrane, heavy metals, electroplating, wastewater

The electroplating industry is one of the major industries which generates a large portion of wastewater containing heavy metals. Even though conventional chemical treatment processes were suitable for this kind of wastewater, the effluent after treatment still contained too high a concentration of heavy metals over the effluent standard regulated by the Ministry of Industry (MOI), Thailand. It is then necessary to have more treatment prior to discharge into the natural resources. Membrane technology was considered suitable for this final treatment step to improve the quality of the effluents before discharging into the rivers.

Heavy metals are important sources of environmental pollution. Some of them can form compounds which are toxic even in very low concentration. Even though the conventional techniques for organic compounds removal are biological and chemical treatment of the toxic materials degrades them to salts and CO_2 , the metals are permanent. The only way to remove them is to change their chemical and physical states by oxidation/reduction and precipitation. So far, there have been a number of researches that studied physical and chemical treatments.

Membrane technology was one option for a nonpolluting process. The membrane of optimum pore sizes were capable of removing almost all pollutants without using any chemicals. Sludge obtained in the process contained only pollutant constituents in this feed stream. In order to increase the removal efficiency and reduce the operating cost, membrane technology was also used together with other treatment processes.

Heavy metal removal from wastewater has been investigated by many researchers. Mcvaugh

and Wall (1978) studied a chemical treatment system using lime as a coagulant for the precipitation of heavy metals from wastewater of agricultural machinery factories, the optimum pH for the heavy metals was 10.5. Suspended solids, Cr, Fe, Pb and Zn at the initial concentration of 200 mg/l, 26 mg/l, 56 mg/l and 42 mg/l were reduced to 28 mg/l, 1.40 mg/l, 1.59 mg/l, 0.62 mg/l and 2.88 mg/l, respectively.

Kreye (1978) reported effective heavy metals removal by using NaOH and cationic polymer. Total suspended solid was reduced 96%, Zn, Cr and Fe were reduced from 52 mg/l, 5.5 mg/l and 14.5 mg/l to 0.23 mg/l, 0.43 mg/l and 0.31 mg/l, respectively. The difficulty of this process was that Cr in wastewater had to be reduced prior to precipitation. The removal of heavy metals studied by Patterson (1985) showed that different heavy metals had different optimum pH for precipitation with OH⁻. Also Boonyakitsombat (1992) found that optimum pH for heavy metals precipitated by lime were at the same range as caustic soda. The optimum pH for Cr, Cu, Ni, Zn removal were 9-11, 7.5-11, 10.5-11, and 9.5-11, respectively. All heavy metals concentrations in the effluent after treatment except Zn were still higher than the standard effluent of Ministry of Industry (MOI), Thailand.

Parinyapariwat (1989) performed the experiment of Zn and Cr removal by three kinds of bases; NaOH, CaO and polymer. The results showed that lime gave more efficiency of Cr removal than caustic soda, but the effluent after treatment still did not meet the requirement of effluent properties of MOI.

Membrane technology is an alternative method recently applied to improve the quality of

effluent. To reduce the problem of fouling, this technology can be applied after chemical treatment when big flocs have been formed.

Studying the heavy metals from water using membrane, Kosarek (1981) found that efficiencies of removal of As, Cd, Cu, Pb, Hg, Ni, Se and Zn were 75-98%. The results also showed that for water treatment with polymer prior to chemical treatment, the percentage removal increased to 90-99%.

Fane (1992) informed the results from the recovery of heavy metals from wastewater using three systems, nanofiltration, ultrafiltration coupled with ion exchange resin and liquid membrane contractor. Fane found that nanofiltration can remove all heavy metals in one step, the second system gave high efficiency of filtration but was more suitable for batch process, and the last method had the problem of phase leakage.

Kim *et al.* (1993) studied the filtration of Ag using ultrafiltration membrane(UF) of MWCO of 30, 100, and 300 kDa and microfiltration membrane (MF) of pore size of 0.22 μm . The results showed that only UF membranes could reject all Ag. They also reported that washing by agitation over the membrane surface could improve the filtration efficiency. Kim *et al.* (1994) filtrated colloids of gold of two particle sizes, 10 nm and 53.5 nm using GVHP membrane of pore size 0.22 μm . It was shown that gold colloids of particle size 10 nm had lower flux and higher decreasing flux rate compared to those of 53.5 nm. Flux of 10 nm - gold colloids was higher than that of 8.3 nm - silver and showed lower solute resistance. Washing by agitation over the surface of the membrane at 400 rpm gave more efficient performance than doing so at 200 rpm.

Enoch *et al.* (1994) studied the pilot scale for the wastewater treatment of wet lime(stone) – gypsum flue gas desulphurization plant by precipitation with hydroxide and sulphite followed by cross flow filtration. It was found that hydrophilic membrane gave higher flux compared to hydrophobic membrane, and backwash had positive effect on flux. The removal efficiency was satisfactory, except for Cd removal.

This research was aimed to propose a membrane technology to supplement the conventional chemical treatment of heavy metal in wastewater. The results obtained in this work can be applied to the industrial requirement and can also be used as a database for the environmental technology development.

Materials and Methods

The electroplating process has four solutions of chromium, copper, nickel, and zinc with each at different concentrations. The automobile part was rinsed with water after dipping in a solution for a certain time. The four rinsed wastewater samples then contained a large amount of heavy metals and needed to be treated before discharging into the public reservoir. The electroplating process diagram is shown in Figure 1.

In the experiment, we used four rinsed wastewater from electroplating process and four synthetic wastewater containing the same heavy metal concentrations as in the rinsed wastewater. The four synthetic wastewater of chromium, copper, nickel and zinc had the concentrations of 2500 mg/l, 1000 mg/l, 100 mg/l and 10 mg/l, respectively.

The composite rinse wastewater had a ratio of 1:1:1:1 by volume and the composite synthetic wastewater also had the same ratio. The synthetic wastewater was used for comparison with factory wastewater.

The parameters of factory and synthetic wastewater samples, such as heavy metal concentrations, total solids (TS), total suspended solids (TSS), total dissolved solids (TDS), pH, and conductivity were determined by analytical methods referred to methods for the examination of water and wastewater (APHA, AWWA, and WEF, 1992).

The experiments were divided into two parts, the first part was the study of suitable pH for heavy metal removing by precipitation and the second part was the removal of heavy metals in supernatant by membrane filtration, microfiltration (MF) and ultrafiltration (UF).

In the first part of the experiment we found

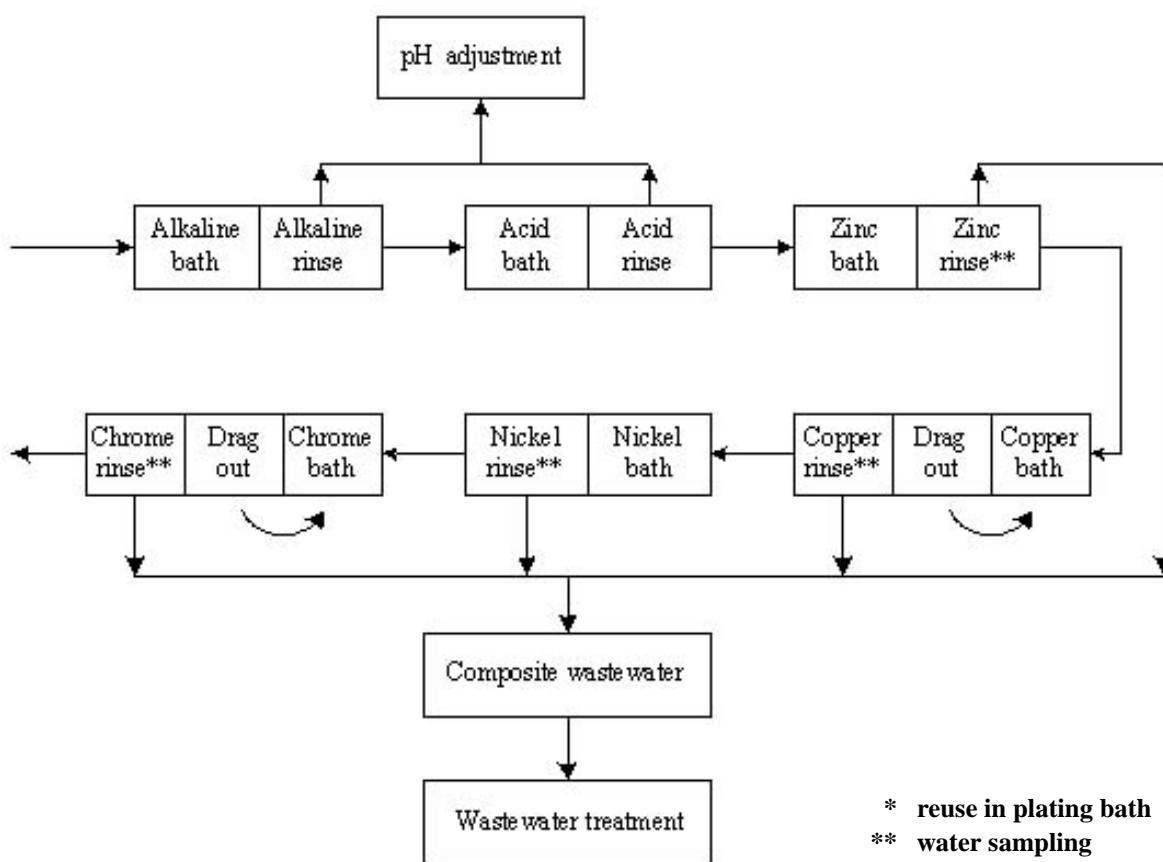


Figure 1. Working process and water sampling

that the suitable pH values for heavy metals in the four rinsed wastewater and the four synthetic wastewater samples to precipitate were in the range of 9.5 – 10.5 with the optimum precipitating time of 90 minutes. The heavy metals removal from the supernatant solution taken from the first part was performed by MF and UF process. Cellulose acetate membranes with pore size of 0.2 μm were used in the MF step and the polysulfone membranes with MWCO 30 kDa were used in the UF step. Both membranes were from Satorious Company, Germany. The filtration by MF and UF processes was carried out at constant pressure of 50, 100 and 200 kPa and the flux was recorded at 1, 5, 10, 20, 30, 40, 50, 60, 70, 80 and 90 minutes.

Deionized water was used in the washing step to decrease fouling after 90 minutes of fil-

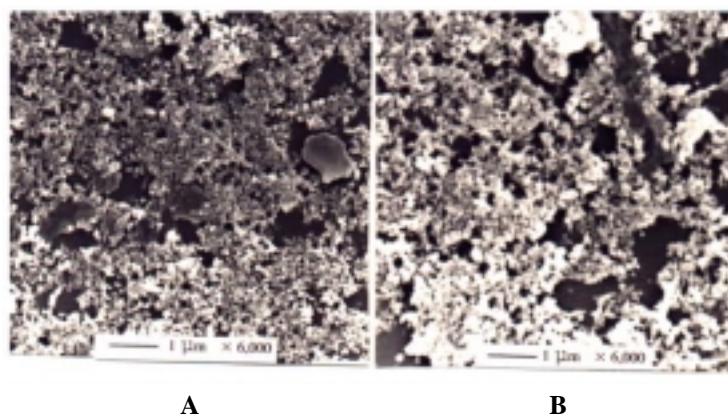
tration. The total filtration time in each experiment was 360 minutes then the membrane was washed four times. The stirring effect during the washing step was studied by operating a magnetic stirrer at 400 rpm for 30 minutes.

Experimental results

The four rinsed wastewater samples taken from washing water baths after electroplating step were clear as almost all solids were soluble. Some soluble salts gave colorful solution. Rinsed water from the Zn electroplating process was neutral and the rest were acidic. By several sampling analysis, it was found that the properties of wastewater from electroplating process varied in wide range depending on process detail and working behavior in the production step. The sample qualities shown in Table 1 are average

Table 1. Industrial electroplating wastewater characteristics.

Parameter	Chrome rinsed	Copper rinsed	Nickel rinsed	Zinc rinsed
Heavy Metals(mg/l)	2,136	1,020	96.1	8.7
pH	1.66	1.76	5.21	7.60
Conductivity (mS/cm)	30.00	8.12	1.15	1.00
TS (mg/l)	6,130	3,470	530	1,370
SS (mg/l)	12	4	3	4
TDS (mg/l)	6,118	3,466	527	1,366

**Figure 2. SEM micrographs of polysulfone ultrafiltration membrane surface after filtering for 6 hours at 100kPa.**

A = composite synthetic wastewater , B = composite industrial wastewater

values of 3 samples of discharged water from each washing bath.

For the filtration experiment, it was found that the flux from microfiltration and ultrafiltration processes of supernatant from both synthetic and factory rinsed wastewater increased with pressure. The filtration efficiency was high at the beginning and decreased after a certain time. The fouling effect increased filtration resistance and decreased filtration efficiency. Surface washing for several times was necessary for decreasing this fouling.

The decrease of fouling rate depended on pressure. At high pressure, where the flux was high, it seemed to induce quick fouling that caused decreasing of flux rate. This result agreed with the work of Kim *et al* (1994)

The decreasing of flux was high at the be-

ginning for all experiments which agreed with the work of Visanathan and Aim (1989) which was a study of fouling in microfiltration of silver colloids. This can be explained since, at the beginning, colloids accumulated on the membrane surface and formed bridges in the pores which finally became a cake or gel layer which caused decreasing flux.

The comparison between the flux of synthetic and factory rinsed wastewater samples showed that the higher flux from microfiltration and ultrafiltration was obtained from the factory rinsed wastewater. This can be explained as the flocs formed from synthetic wastewater after chemical precipitation were smaller than those of rinsed water. This result was confirmed using the SEM shown in Figure 2.

The smaller particles could diffuse into the

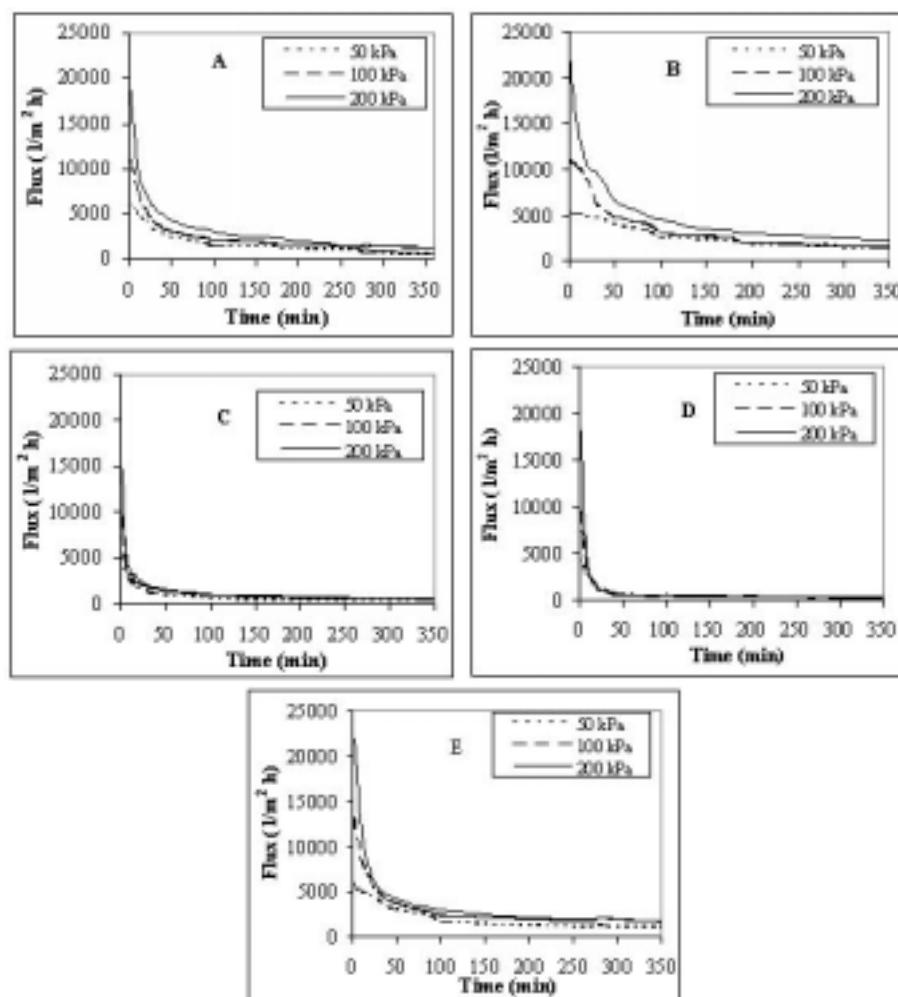


Figure 3. Flux vs time of synthetic wastewater (Cellulose acetate microfiltration membrane pore size = 0.2 μm).

A = Chromium synthetic wastewater , B = Copper synthetic wastewater ,

C = Nickel synthetic wastewater , D = Zinc synthetic wastewater

E = composite synthetic wastewater

pore and attract onto the wall by Van der Waals attraction, electrical double layer and hydrodynamic attraction, thus forming a thin layer of particles in the pore which caused more fouling than the big particles. The flux of filtration of smaller particles was then less than that of the bigger ones. This result also agreed with the work of Visanathan and Aim. (1989) and Kim *et al* (1994).

The graphs of flux vs. time of synthetic and factory rinsed wastewater samples using MF and

UF are shown in Figure 3-6.

Many methods of washing can be used for reducing membrane fouling to improve flux, for example the backwash method which is done by inverting the membrane and passing it with deionized water to push off the accumulated particles on the surface and in the pores, This method has a limit that it should be operated at a pressure less than 200 kPa because of the destruction of the membrane (Kim *et al* 1994). The other meth-

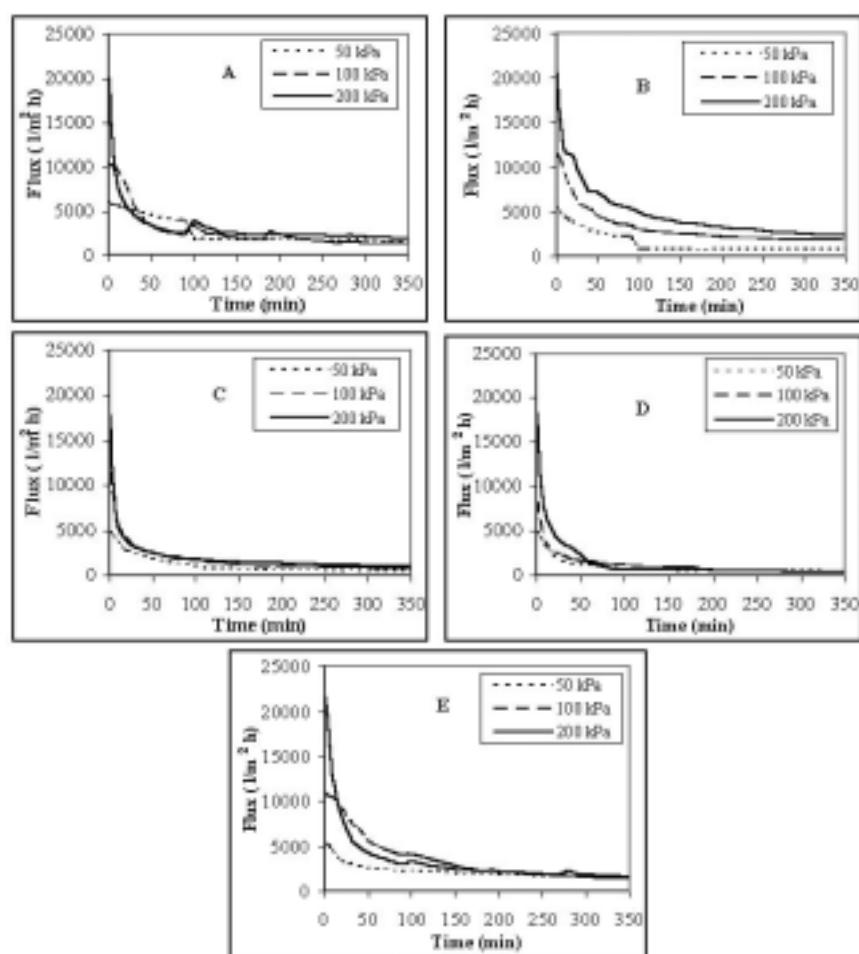


Figure 4. Flux vs time of industrial wastewater (Cellulose acetate microfiltration membrane pore size = 0.2 μm).

A = Chromium wastewater , B = Copper wastewater, C = Nickel wastewater ,
D = Zinc wastewater , E = composite wastewater

Table 2. Solute resistance of synthetic wastewater (Cellulose acetate microfiltration membrane pore size = 0.2 μm).

Time (min)	Solute resistance (m^{-1})														
	Cr			Cu			Ni			Zn			Composite		
	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa
90	0.028	0.041	0.059	0.008	0.023	0.037	0.109	0.109	0.281	0.089	0.256	0.491	0.027	0.036	0.060
180	0.037	0.055	0.073	0.019	0.036	0.064	0.135	0.145	0.291	0.226	0.308	0.816	0.032	0.050	0.081
270	0.038	0.047	0.169	0.026	0.055	0.081	0.160	0.204	0.348	0.281	0.452	0.854	0.035	0.050	0.092
360	0.040	0.056	0.254	0.030	0.085	0.129	0.157	0.270	0.405	0.302	0.464	1.102	0.036	0.050	0.111

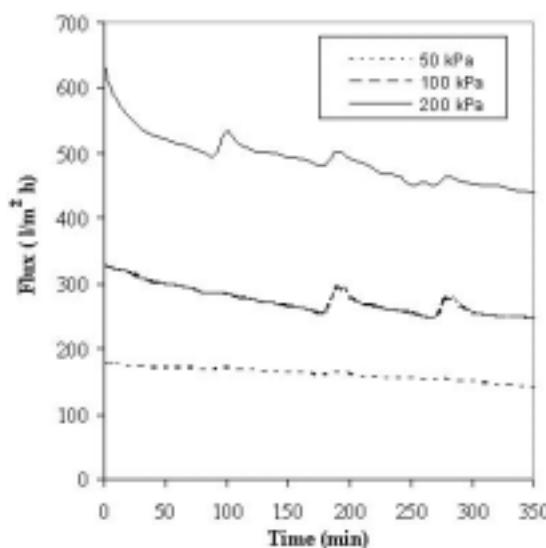


Figure 5. Flux vs. time of synthetic composite wastewater by Polysulfone ultrafiltration membrane (MWCO 30 kDa.)

ods such as washing with acids or bases may be costly methods and have risks of destroying the membrane. The method used in this research was washing using deionized water and a magnetic stirrer agitated at 400 rpm for 30 minutes over the surface to lift the particles in the gel layer out of the surface.

At the end of washing, the washed water was rinsed and the used membrane was ready for the next experiment. This method of washing was found appropriate for reasons of low cost and no risk of destruction of membrane.

Washing was found to improve the flux only for a short period at the beginning of reuse (after 90, 180, and 270 minutes, showed in Figure 3 – Figure 6). It was also shown from the results that washing by agitating above the membrane surface would be effective for the large accumulation of particles on the surface and the cake formed was not too dense, because the distance of the propeller was 2 mm above the membrane surface. The amount and the particle packing types on membrane surface were important parameters to washing efficiency.

At high pressure, as the accumulation of

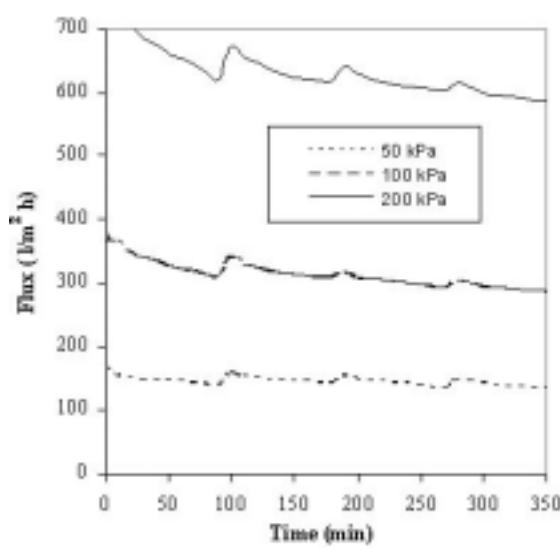


Figure 6. Flux vs. time of industrial composite wastewater by Polysulfone ultrafiltration membrane (MWCO 30 kDa.)

particles on membranes increased, thereby increasing the solute resistance. It is evident that the major membrane fouling during filtration was the cause of increased solute resistance. Even though washing can decrease the degree of fouling, the results of this study in Table 2 and 3 show that solute resistance did not change significantly after washing because the fouling in this research was the internal blocking type.

Conclusions

The concentration of heavy metals in permeate varied with feed pressure. At high pressure, the particles blocked in the pore were pushed away and discharged with the permeate. For longer time of filtration, more particles will be pushed away with the permeate.

From the comparison of the permeate quality with the standard effluent quality of MOI (1996) as shown in Table 6, synthetic wastewater can have better quality than factory wastewater because the flocs formed in the chemical precipitation of synthetic wastewater were smaller than those from factory wastewater. These smaller

Table 3. Solute resistance of industrial wastewater (Cellulose acetate microfiltration membrane pore size = 0.2 μm).

Time (min)	Solute resistance (m^{-1})														
	Cr			Cu			Ni			Zn			Composite		
	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa	50 kPa	100 kPa	200 kPa
90	0.013	0.034	0.020	0.022	0.023	0.032	0.066	0.070	0.120	0.057	0.100	0.090	0.010	0.013	0.050
180	0.016	0.040	0.082	0.051	0.036	0.048	0.076	0.084	0.137	0.074	0.216	0.317	0.023	0.036	0.084
270	0.024	0.078	0.097	0.041	0.047	0.066	0.082	0.125	0.178	0.098	0.390	0.744	0.030	0.056	0.108
360	0.027	0.080	0.097	0.050	0.050	0.078	0.085	0.137	0.193	0.102	0.417	0.755	0.041	0.066	0.126

Table 4. Solute resistance of composite synthetic wastewater (Polysulfone ultrafiltration membrane , MWCO = 30 kDa).

Time (min)	Solute resistance (m^{-1})		
	composite synthetic wastewater		
	Feed pressure 50 kPa	Feed pressure 100 kPa	Feed pressure 200 kPa
90	0.135	0.178	0.169
180	0.275	0.225	0.295
270	0.304	0.209	0.300
360	0.314	0.231	0.304

Table 5. Solute resistance of composite wastewater (Polysulfone ultrafiltration membrane , MWCO = 30 kDa).

Time (min)	Solute resistance (m^{-1})		
	composite factory wastewater		
	Feed pressure 50 kPa	Feed pressure 100 kPa	Feed pressure 200 kPa
90	0.017	0.005	0.036
180	0.133	0.028	0.045
270	0.164	0.122	0.079
360	0.193	0.197	0.115

particles could be pushed to plug into the pores and acted as a secondary filter. The permeate contained less smaller particles than that of the factory wastewater.

The rejection of the heavy metals can be obtained by the fouling at the surface of the membrane, as so called cake or gel layer, and also by the internal blocking in the pores of this mem-

Table 6. Heavy Metals Removal by Membrane

Heavy Metals	MF - Synthetic wastewater						MF - Industrial wastewater					
	Pressure 50 kPa		Pressure 100 kPa		Pressure 200 kPa		Pressure 50 kPa		Pressure 100 kPa		Pressure 200 kPa	
	Rej. (%)	H L	Rej. (%)	H L	Rej. (%)	H L	Rej. (%)	H L	Rej. (%)	H L	Rej. (%)	H L
Chromium	52.83-55.97	✓	47.80-49.69	✓	35.85-48.43	✓	64.60-66.37	✓	19.47-43.36	✓	12.39-16.81	✓
Copper	>98.67	✓	>98.67	✓	>98.67	✓	95.00-96.67	✓	83.33-91.67	✓	79.17-84.17	✓
Nickel	96.00-97.39	✓	64.70-67.83	✓	24.00-40.52	✓	68.26-68.86	✓	62.87-63.47	✓	40.12-43.71	✓
Zinc	74.07-86.67	✓	60.00-62.96	✓	38.52-43.70	✓	33.88-34.71	✓	25.62-28.10	✓	14.88-16.53	✓
Composite												
-Chromium	55.17-63.79	✓	43.10-55.17	✓	37.07-43.10	✓	19.51-28.30	✓	8.52-32.42	✓	9.34-19.51	✓
-Copper	>98.71	✓	98.28-98.71	✓	98.73	✓	40.58-47.83	✓	40.58-44.93	✓	10.14-28.99	✓
-Nickel	>98.05	✓	>98.05	✓	98.05	✓	42.53-45.98	✓	13.79-14.94	✓	8.05-12.64	✓
-Zinc	>98.95	✓	>98.95	✓	98.95	✓	40.00-60.00	✓	30.00-40.00	✓	10.00-25.00	✓
Chromium	-	-	-	-	-	-	-	-	-	-	-	-
Copper	-	-	-	-	-	-	-	-	-	-	-	-
Nickel	-	-	-	-	-	-	-	-	-	-	-	-
Zinc	-	-	-	-	-	-	-	-	-	-	-	-
Composite												
-Chromium	50.51-54.55	✓	47.47-56.57	✓	44.44-51.52	✓	27.25-33.50	✓	25.25-36.25	✓	17.00-20.25	✓
-Copper	>98.69	✓	98.69	✓	= 98.69	✓	15.49-16.90	✓	14.08-18.31	✓	2.82-4.23	✓
-Nickel	97.88	✓	97.30-97.69	✓	96.72-97.11	✓	30.38-32.91	✓	32.91	✓	21.52-25.32	✓
-Zinc	>98.98	✓	>98.98	✓	>98.98	✓	>83.33	✓	50.00->83.33	✓	33.33-66.67	✓

MF = Cellulose acetate microfiltration membrane, pore size 0.2 μm ; UF = Polysulfone ultrafiltration membrane, MWCO 30 kDa

Rej. (%) = Rejection (%) ; H = Concentration of heavy metal after filtration is higher than standard value given by Ministry of Industry (MOI)

L = Concentration of heavy metal after filtration is lower than standard value given by Ministry of Industry (MOI)

brane which will reduce the pore sizes. However, high rejection will come along with low flux and the control of this phenomena was very difficult, as for long filtration period, the particles on the surface and in the pores may be pushed away with the filtrate or may block until the filtration could not be continued.

Smaller pore size membrane may be another alternative for high rejection but require a high pressure unit for operation that lead to high cost and the difficulty in controlling the process at high pressure. If the floc sizes increase by mean of good pretreatment of the wastewater operating at low pressure, lower cost and prolonged membrane life will be well achieved.

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