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Abstract: Polysulfone (PSf) asymmetric hollow fiber membranes with the molecular weight cut-off (MWCO) of 13,000 Dalton were prepared by the dry-wet phase inversion method, with which negatively charged chromate ions, CrO_4^{2-} was removed form aqueous solutions using the polyelectrolyte-enhanced ultrafiltration (PEUF) process. Parameters relating to the chromate removal such as the type of polyelectrolyte, the loading ratio, and the pH value of the aqueous solution were studied. Poly (dially dimethylammonium chloride) (PDADMAC) was found to be the most effective polyelectrolyte. Under the optimal conditions, that is, the polyelectrolyte-metal loading ratio of 20 and the pH value 8.0 respectively, up to 100% of the chromate rejection was achieved. The used polyelectrolyte may be recovered about 70% by the acidified decomplexation combined with ultrafiltration. The concentration polarization was present but imposed limited effect on the PEUF process in the long-term operation.

Keywords: Ultrafiltration, heavy metal ions, hollow fiber membrane, complexation

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INTRODUCTION

Many industrial wastewater streams (e.g. the metal working, semiconductor and copper industries, mine water, etc.) contain heavy metal ions, which could pose serious detrimental effects to both the environment and human health, and thus must be removed prior to water discharge or water recycling (1–3). A number of treatment methods have been applied to remove the heavy metal ions from wastewater including

(i) precipitation as hydroxides, carbonates or sulphides,
(ii) sorption (adsorption, ion exchange)
(iii) membrane processes,
(iv) electrolytic recovery,
(v) (vi) liquid-liquid extraction and
(vi) flotation (3).

Although the precipitation and sorption are still the most popular methods for the removal of heavy metal ions, special attention is given to membrane processes in recent years since they are cost-effective, easy to operate and have high efficiency. However, because heavy metal ions are usually too small to be retained by the ultrafiltration membranes, the membrane filtration is usually combined with other separation processes (so-called hybrid processing) so that it combines advantages of these processes but overcomes the individual limitations (4). For example, the ultrafiltration process combined with polyelectrolyte binding, called polyelectrolyte-enhanced ultrafiltration (PEUF) can obtain a high removal efficiency with a low operating cost (5, 6). It is based on the complexation of heavy metals by water-soluble polymers to form macromolecular complexes that can be retained easily by membranes. Since the complexation of metal ions with polymers takes place in the homogenous phase, there are no difficulties related to heterogeneous reactions, interface transfer, and long contact time problems of multiphase separation processes (7). Furthermore, both the polymer and heavy metals can be recovered by the subsequent decomplexation, hence the cost may be further reduced.

It is known that the hollow fiber membranes can provide a much larger membrane area (500∼9000 m^2/m^3) for water permeation compared with other configurations such as flat sheet and spiral wound (30∼800 m^2/m^3). However, most of the previous PEUF studies were performed in a bath cell with stirring using flat sheet membranes (5–7). Therefore, the performances of the hollow fiber membrane modules for PEUF processes are not very clear (8). Moreover, the study on the removal of negatively charged heavy metal ions such as chromate, which has been widely used in metal finishing and electroplating, is much less than those of heavy metal cations such as Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Hg^{2+}, and Cd^{2+} from aqueous streams by PEUF (9–12). In this work, the removal of hexavalent chromate by PEUF in a hollow fiber membrane module has been studied. Some factors relating to the performances of the hollow fiber membrane module for PEUF such as the effects of the polyelectrolyte type, the polyelectrolyte loading ratio, and the pH value of the aqueous solution on the removal efficiency have been investigated in detail. In addition, the concentration polarization or membrane fouling in a long-term operation and the decomplexation of retentate for the recovery of polyelectrolyte has also been discussed.

EXPERIMENTAL

Materials

Poly (dially dimethylammonium chloride) (PDADMAC) (average MW 400,000–500,000), 80% ethoxylated polyethyleneimine (PEI) of 35–40 wt.% solution in water (average MW 70,000), high MW chitosan (average MW 410,000) were obtained from Aldrich Chemical Company Inc. Polyethylene glycol (PEG) (MW 10000, 12000, 15000, and 20000) were purchased from Merck–Schuchardt Chemical Company. Analytical grade anhydrous sodium chromate, Na_2CrO_4 was supplied by BDH Laboratory Supplies. Reagent grade solutions of 1M NaOH and 1% (v/v) H_2SO_4 were used to adjust the pH of the solution. Polysulfone (PSf) (Udel P-1800) was purchased from Amoco Performance Product Inc (USA). N-methyl-2-pyrrolidone (NMP, 99%, Merck) was supplied by MERCK and used without further purification. Distilled water and ethanol of reagent grad (99.8 v%, Merck) was used as the additive. Tap water was used as both the internal and the external coagulants.

Hollow Fiber Preparation and Characterization

PSf hollow fiber membranes were spun by the dry-wet phase inversion method. The dope solution has a composition of 20 wt.% PSf, which was dried at 100°C for 10 h prior to use, 7.6 wt.% EtOH, 1.95 wt.% H_2O, and 70.45 wt.% NMP. The spinneret used has the orifice diameter of 0.6 mm and the tube inner diameter of 0.15 mm. Water was used as both internal and external coagulants at ambient conditions ($25 \pm 1^{\circ}C$, RH = 60–65%). The air gap was set at 5 cm, and the internal coagulant rate at 1.1 cm^3/min, respectively. The resulting PSf hollow fiber membranes have the dimensions of Φ 0.9/0.52 mm in OD/ID, respectively. The fibers were kept in fresh water for at least 1 week. The details of the spinning equipment and procedures on hollow fiber spinning were described elsewhere (13, 14).

The molecular weight cut-off (MWCO) of the prepared hollow fiber membranes was measured using polyethylene glycol (PEG) with different molecular weights as the solute. Testing modules with both ends open were made. The module contained about four wet fibers with the length of 10–20 cm. Water permeation flux and solute rejection were determined using a membrane testing unit (Nitto Denko Corporation, Japan). The aqueous solution with 1 g/L PEG with a molecular weight from 10,000 to 20,000 was fed into the shell side of the module. The operating pressure (0.2 MPa, gauge) and solution recirculation flow velocity (about 22 cm^3/s) through the module were controlled with the adjusting valve. The total feed volume of the testing solution is about 1 L. The water permeation flux in the permeate side was measured at ambient temperature of 23°C. The concentrations of PEG in the feed, permeate and the retentate streams were measured using a TOC analyzer (TOC 5000A, Shimadzu).

In addition, the pure water permeate flux of the membrane, J_w was measured using distilled water at 0.2 MPa. It was calculated to be around 0.11 $cm^3/cm^2 \cdot min$ after 10 min of operation. Only the hollow fibers with a deviation of J_w within 5% were used for the decolorization experiments.

Apparatus and PEUF Experiments

The PEUF experiments were carried out in a hollow fiber membrane module, which consists of 16 PSf hollow fibers with length of 30 cm housed in a stainless steel tube. The experimental set-up is schematically illustrated in Fig. 1. The weighted sodium chromate and polyelectrolyte were dissolved into a certain volume of MilliQ Plus distilled water in a 5 L tank to form the feed solutions with desired chromate and polyelectrolyte concentrations prior to the experiments. It was stirred with a magnetic stirrer for at least 1 hr to ensure reaching complexation equilibrium. The stirring rate was 300 rpm so that effective mixing was achieved but not forming a serious vortex in the cell, which would destroy the formed complex. Distilled water was circulated in order to wash the membranes before and after each run. The chromate content in the outlet solutions was measured to ensure that only trace amount of chromate remained in the membrane module, when the pure water permeate flow rate was measured. The feed solution was pumped under 2 bar pressure into the shell side of the membrane module at a constant flow rate through a Membrane Master (Nitto Denko NDS-TATAAA), which consists of the pump, pressure gauge, flowrate meter, and flow control valves, while the permeate was obtained from the fiber lumens. Both the retentate and the permeate solutions were returned back to the feed vessel to keep the concentration of the feed solution constant throughout the experiments. At certain time intervals, the permeate flow rate was measured and samples from both permeate and feed solutions were taken to determine their chromate concentrations. During the experiments, the temperature of the feed solution was kept at the ambient temperature of 23°C. Effects of the type of polyelectrolyte, the loading ratio defined as the ratio of polyelectrolyte to heavy metal ion concentrations and the pH value of

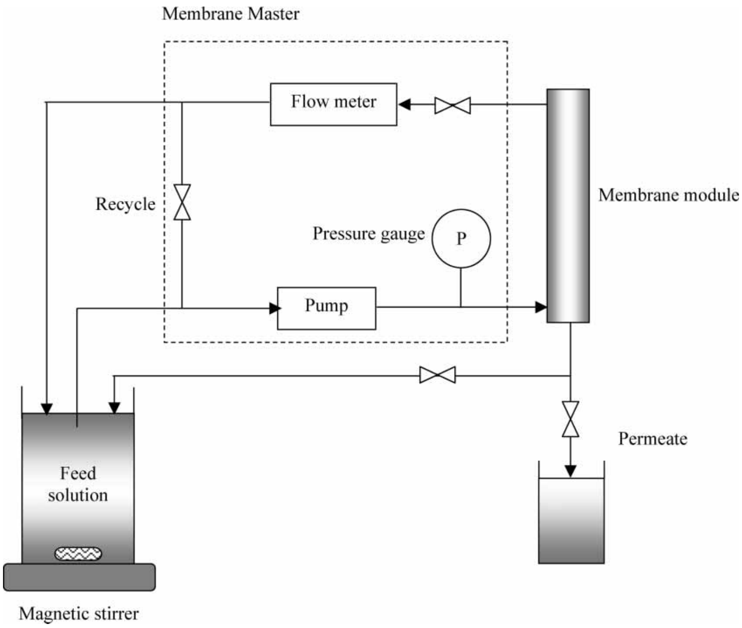

Figure 1. PEUF experimental set-up for chromate removal.

the feed solution were studied. The pH was adjusted with dilute HNO_3 and NaOH solutions and was continuously monitored during each run using a pH meter supplied by Hanna Instruments. The used membrane module was cleaned with 0.01 M NaOH solution for 10 min after a few PEUF experiments.

The rejections of chromate (also of polymer) were calculated by:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \tag{1}$$

where C_p and C_f are the ion (or polymer) concentrations in the permeate stream and in the initial feed, respectively.

Analysis Method

Chromate concentrations were measured using an Atomic Absorption/Flame Emission Spectrophotometer (AAS–Shimadzu, AA–680). Chromate solutions from preliminary studies were also analyzed using the Shimadzu UV–3101 PCUV–VIS-NIR Spectrophotometer. The color development reagent was 1,5-diphenylcarbarzide in acetone. 2 cm^3 of this reagent was added to the acidified chromate solutions in 100 cm^3 volumetric flasks and the absorbances of the prepared solutions were measured at 540 nm as suggested in literature (15).

RESULTS AND DISCUSSION

Characteristics of the PSf Membranes

Determination of the membrane's MWCO, which is defined as the molecular weight at which 90% of the molecules are rejected by the membrane, is essential to applying the appropriate polyelectrolytes for the UF separation. PEG with molecular weight from 10,000 to 20,000 was used as the testing solute, with the results shown in Fig. 2. It can be seen that the MWCO of the prepared PSf membranes is close to 13,000. This suggests that the polyelectrolyte for UF in this PSf hollow fiber membrane should be above 13,000 in molecular weight.

A blank experiment was conducted ahead of the PEUF experiments, that is, the chromate solution without any polyelectrolyte was used as the feed solution. Unexpectedly, 22% of chromate rejection was achieved at the beginning of the experiment but attenuated gradually as the experiment proceeded. This may be due to the absorption of CrO_4^{2-} by the PSf membranes before passing through the membranes. Obviously, such a rejection cannot be continuous because the adsorption capacity of the membranes is limited.

Application of Different Polyelectrolytes

The polymers used in PEUF are highly charged macromolecules or aggregates, formed in aqueous solution by dissociation of charged units. They should have very low acute and chronic toxicity to environmental organisms and be readily biodegradable. Besides the molecular weight required, the polyelectrolyte should also possess enough complexation ability with the heavy metal ions. It is known that the polymers having a high content of non-protonated amino groups (primary, secondary, tertiary

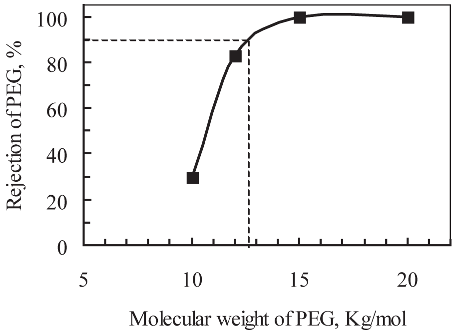

Figure 2. Rejection versus molecular weight of PEG for the PSf membrane (Feed concentration $=1000\ g/m^3$, pressure $= 0.2$ MPa, feed flow rate $= 22\ cm^3/s$, feed volume $= 10^3\ cm^3$).

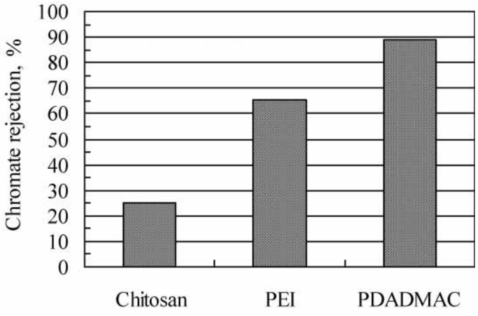

Figure 3. Chromate rejection with respect to different polyelectroltye (Concentration of $CrO_4^{2-} = 20\ g/m^3$, operating pressure = 0.2 MPa, feed flow rate = 1.28 L/min, loading ratio =10, pH = 6).

and quaternary) may enhance the binding ability with the heavy metal ions. Hence, in this work, three kinds of cationic polyelectrolytes with high molecular weight: chitosan, PEI, and PDADMAC were tested for the removal of chromate ions by the PEUF process. The chromate removal in terms of rejection percentage with these polyelectrolytes is shown in Fig. 3, where all other operating conditions are the same. It can be seen that as high as 89.2% of chromate rejection was achieved by using PDADMAC while only 24.9% of chromate rejection obtained with chitosan although they have the comparative molecular weight. It implies that PDADMAC possesses the highest binding capacity while chitosan the least with CrO_4^{2-} for form a complex. This may be explained as that PDADMAC as well as PEI has quarternary amino sites, leading to a higher affinity to anions CrO_4^{2-} at suitable pH via electrostatic attachment. Since PDADMAC may give the highest CrO_4^{2-} rejection, it was thus used as the polyelectrolyte for chromate removal in the following experiments.

Effect of the Polyelectrolyte Concentration

Figure 4 shows the water permeate flow rates with varying PDADMAC feed concentrations (200, 1000 and 5000 g/m^3, respectively) on a logarithm scale at different operating pressures. As is expected, the water permeate flow rate decreases as the polyelectrolyte concentration increases when the operating pressure is kept constant. The linear dependence of the permeate flow rate on the logarithm of PDADMAC concentration implies the concentration polarization behavior. Furthermore, the higher the operating pressure, the faster the decrease in permeate flow rate with increasing the polyelectrolyte concentration. Obviously, the polyelectrolyte concentration applied should not be too high otherwise the detrimental effect to water permeation would be produced. Nevertheless, the experimental data indicate that the gel layer

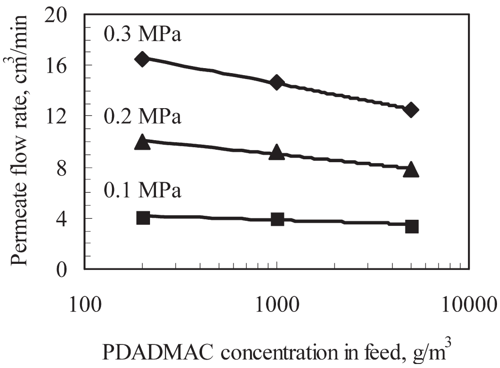

Figure 4. Plot of permeate flow rate against PDADMAC feed concentration at different operating pressures (Chromate feed concentration =10 g/m^3, feed flow rate = 0.5 L/min, Temperature = 23°C).

would not be formed unless the PDADMAC concentration exceeds 10^8 g/m^3 or 0.22 M, which may be obtained by extrapolation of the curves to the abscissa. On the other hand, although the permeate flow rate may be improved by increasing the operating pressure, it is found that the prepared PSf hollow fiber membranes fails to bear the pressures higher than 0.3 MPa. Thus, all the following PEUF experiments would be performed at 0.2 MPa so as to lengthen the module's longevity.

Figure 5 shows the effect of polyelectrolyte (here, PDADMAC) concentration in terms of loading ratio that is defined as the polyelectrolyte/metal in concentration on the chromate rejection, where the operating pressure is 0.2 MPa and the feed flow rate is 1.28 cm^3/min. As is expected, the

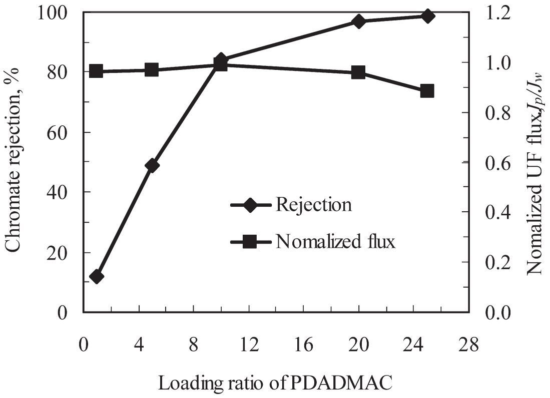

Figure 5. Effect of loading ratio of PDADMAC on the PEUF performance (CrO_4^{2-} concentration =10 g/m^3, operating pressure = 0.2 MPa, feed flow rate = 1.24 L/min, pH = 6).

chromate rejection increases with increasing the loading ratio. When the loading ratio of polyelectrolyte is increased to 20, the chromate rejection has reached 98.5% but little improvement can be achieved with further increasing the loading ratio to 25. In this figure, the normalized UF flux, J_p/J_w where J_w is the pure water permeation flux under the same conditions as UF operation, is also plotted against the loading ratio. It can be seen that the water permeation flux almost remains unchanged as the loading ratio is increased from 1 to 20. However, at the loading ratio of 25, the normalized UF flux would be reduced from around 0.97 to 0.89. Such decrease in permeation flux is resulted from the concentration polarization occurs in a high polyelectrolyte concentration. Therefore, the optimal loading ratio should be about 20 to prevent concentration polarization and membrane fouling.

Effect of pH Value of the Solution

Effect of pH value of the solution on the PEUF performance is shown in Fig. 6, where the chromate concentration is $10\ \mathrm{g/m^3}$ and the PDADMAC loading ratio is 20. As can be seen, the chromate rejection is significantly increased as the pH value increases from 3 to 5. After the pH value of the solution is higher than 6, the chromate rejection may reach above 97% or even up to 100% at the pH value of 8. Therefore, the optimal pH for chromate removal by the PDADMAC-enhanced UF should actually be 8.

An additional experiment was carried out that the pure PDADMAC solution with different pH values was fed into the UF module. The rejection of PDADMAC was hardly changed with pH of the solution, indicating that the cationic behavior of PDADMAC is independent of pH value. On the other hand, it is well known that pH and ionic strength determine

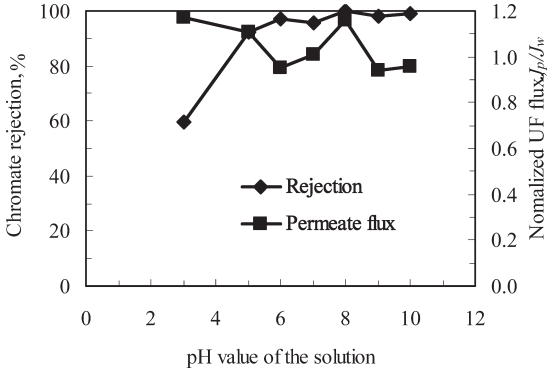

Figure 6. Effect of pH of the solution on the PEUF performance using PDADMAC polymer (CrO_4^{2-} concentration = $10\ \mathrm{g/m^3}$, operating pressure = 0.2 MPa, feed flow rate = 1.24 L/min, Loading ratio = 20).

the major Cr(VI) species in aqueous solutions based on the Cr(VI)–H_2O equilibrium (8).

$$H_2CrO_4(aq) + OH^-(aq) \Longleftrightarrow HCrO_4^-(aq) + H_2O(l)$$

$$HCrO_4^-(aq) + OH^-(aq) \Longleftrightarrow CrO_4^{2-}(aq) + H_2O(l)$$

$$2CrO_4^{2-}(aq) + 2H^+(aq) \Longleftrightarrow Cr_2O_7^{2-}(aq) + H_2O(l)$$

In the acidic solutions, the chromate mainly exists in the form of $HCrO_4^-$ and $Cr_2O_7^{2-}$ anions (coexist) while CrO_4^{2-} anion predominates as the solution pH is increased to higher than 6.5. Consequently, the complexation degree of PDADMAC in the acidic solutions is weaker than that in the basic solutions, resulting in the lower chromate rejection at lower pH values.

The permeate flux as a function of the pH value of the solution is also plotted in Fig. 6. It can be seen that the normalized permeate flux approximates one but still very slightly decreases with increasing pH value. This implies that the concentration polarization is present but is not noticeable.

The chromate rejection against its feed concentration under different pH values is plotted in Fig. 7 where the PDADMAC loading ratio was kept to be 20. It can be seen that the chromate rejection hardly changes with varying the chromate feed concentration at a definite pH. However, at lower PDADMAC loading ratios, it was found that the chromate rejection would slightly increase with increasing the chromate feed concentration even at a constant pH. Nevertheless, the desired chromate removal may be achieved by applying suitable pH value (pH = 8) and PDADMAC loading ratio (about 20).

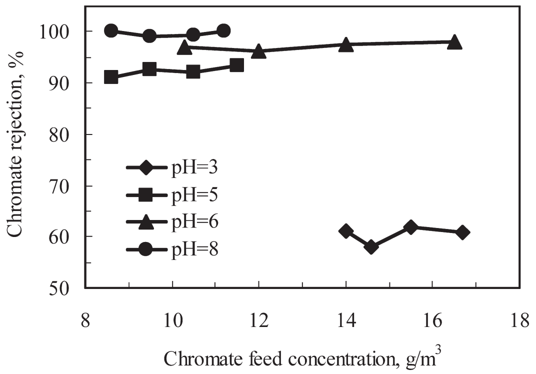

Figure 7. Effect of chromate feed concentration on the rejection (polymer = PDADMAC; operating pressure = 0.2 MPa, feed flow rate = 1.28 L/min, Loading ratio = 20).

Long-term Operation

Membrane fouling is a severe problem for the UF operations. In this work, it is monitored by the water permeate fluxes measured by feeding pure water both before and after each PEUF experiment. All the 12 PEUF experiments were carried out in a period of 6 weeks, during which 3 times of cleaning using 0.01 M NaOH solution were performed. The outcome is shown in Fig. 8. It can be seen that the pure water permeate flux was almost unchanged after a long-term operation. Therefore, membrane fouling is not a serious problem in this PEUF process with PDADMAC as polyelectrolete for the chromate removal. It can be bated by backwashing with pure water after each PEUF operation combined with cleaning using 0.01 M NaOH solution between times.

Decomplexation of the Retentate Solution

Generally, the retentate solution containing the polyelectrolyte-metal complex should be regenerated so that the polyelectrolyte can be reused and the heavy metal also recovered. This can be done through either acidification or electrolysis. In this work, acidification of the retentate was performed, in which the retentate was acidified to pH 1.3 to destroy the complex in the solution and then subject to UF again. The experimental results indicated that the recovery of either PDADMAC or chromate ions was only about 70%. Obviously, this is not an ideal recovery compared with those reported in literatures, which might be up to 99%. Such polyelectrolyte cannot be reused due to the high concentration of CrO_4^{2-} ions in the residual solution. The low electrolyte recovery is mainly resulted from the fact that the decomplexation solution

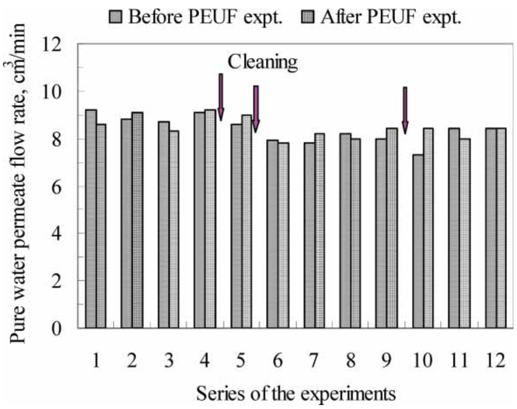

Figure 8. Pure water permeate flux of the hollow fiber membrane module in a long-term operation (polymer = PDADMAC; pressure = 0.2 MPa, feed flow rate = 1.28 L/min, Temperature = 23°C).

cannot be separated well using the presented UF membranes. In order to improve the recovery of polyelectrolyte, the membranes more suitable for the separation of polyelectrolyte and metal ions have to be applied to perform UF of the acidic decomplexation solution.

CONCLUSIONS

Chromate ions can be effectively removed from water by PEUF process using PSf hollow fiber membranes with PDADMAC as the polyelectrolyte. Under the conditions that the pH of the solution is 8 and the loading ratio of polyelectrolyte is 20, the chromate removal may reach up to 100%. The used polyelectrolyte can be recovered about 70% by the acidified decomplexation combined with ultrafiltration. Concentration polarization was present but imposed a limited effect on the PEUF process in the long-term operation.

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