

# Removal of chromium from aqueous solution using cellulose acetate and sulfonated poly(ether ether ketone) blend ultrafiltration membranes

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## Abstract

A process for purifying aqueous solutions containing heavy and toxic metals such as chromium has been investigated. Chromium salts are largely used in various industries including leather-manufacturing industry. Ultrafiltration processes are largely being applied for macromolecular and heavy metal ion separation from aqueous streams. Cellulose acetate and sulfonated poly(ether ether ketone) blend ultrafiltration membranes were prepared by precipitation phase inversion technique in 100/0, 90/10, 80/20 and 70/30% polymer blend compositions and subjected to the rejection of chromium at different concentrations such as 200, 400, 600, 800 and 1000 ppm with a water-soluble macroligand (polyvinylalcohol). Factors affecting the percentage rejection and permeate flux such as pH, concentration of solute, concentration of PVA, transmembrane pressure and composition of blend membranes were investigated. It was found that percentage rejection improved at a pH 6 and a macroligand concentration of 2 wt.%. The transmembrane pressure and concentration of solute also have an effect on the separation and product rate efficiencies of the blend membranes.

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## 1. Introduction

Processing industries produce a wide variety of chemicals and co-products, where the manufacturers require separation, concentration and purification of materials at various stages. These materials include reagents, raw materials, the resulting intermediates, products, by-products and waste streams [1]. On the other hand, many industries employ these chemicals for producing a variety of products. Often, they discharge the unused chemicals in the form of effluent or solid sludge. Tanning industry is one among them, which employs chromium(III) salts for tanning the raw hides and skins and discharges significant quantity of the unused metal in the waste streams [2]. One of the major technical requirements of the leather and other industries employing large quantities of chromium salts in wet processing is the need to minimize the concentration of the metal in the effluents and its removal [2,3]. Reuse and recycling methods [4,5] have therefore

been used, but it has become increasingly popular to employ procedures for obtaining potable water and management of waste water.

During the past decade, there has been a constantly increasing level of interest and research efforts to improve the performance of membranes to supplement or replace techniques such as distillation, adsorption, extraction, crystallization, etc. The term ultrafiltration (UF) is usually applied to a membrane separation process where the solute dimensions are significantly larger than the solvent dimensions. UF has been extensively used for product recovery and pollution control in the chemical, electronic, metal plating, as well as in the food, pharmaceutical, and biotechnological industries [4,5]. Indeed, UF is a membrane technique commonly used to separate and concentrate high molecular weight species present in solution [6]. Traditional techniques such as sorption and chemical precipitation have been used for the removal of metal ions from aqueous effluents [7–9], however, these techniques are either incapable of reducing the concentration to the required levels or prohibitively expensive. The use of membrane separation process in the treatment of waste water and groundwater containing toxic metal ions

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is an attractive and suitable technique, since it offers concentration and separation of metals or valuable chemicals without a change of state and without the use of chemicals or thermal energy [10,11]. Compared to other separation or concentration systems, the energy and space requirements are low for membrane systems and its modular design allows for relatively easy expansion [12].

Conventionally cellulose acetate (CA) membranes are used in reverse osmosis, UF and other membrane processes. The performance of CA membranes can be improved by blending it with appropriate polymers in view of the fact that polymer blends have provided an efficient way to fulfill new requirements for material property [13]. Recently, cellulose acetate-sulfonated poly(ether ether ketone) blended UF membranes have been prepared and applied to the rejection of proteins such as bovine serum albumin, egg albumin, pepsin and trypsin, achieving more than 90% rejection [14]. Heavy metals such as Cu, Ni, Zn, Co, etc., from the waste streams of several chemical, electronic, metal plating and refining industries, have been separated and concentrated through binding of the target metal ions with water-soluble macromolecular compounds and subsequent ultrafiltration of the bound metals from the unbound components [15]. Cellulose acetate has been blended with polyurethane, and the blend membranes have been used for the rejection of  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$  and  $\text{Cd}^{2+}$  ions using polyetherimide as a ligand [16]. Cellulose acetate has also been blended with polysulphone and applied for the separation of chromium using polyvinyl alcohol (PVA) as the macromolecular chelating agent [17]. In the present work, cellulose acetate/sulfonated poly(ether ether ketone) (CA/SPEEK) blend ultrafiltration membranes have been used for separating chromium(III) ions. The main objective of this work is to study the effects of CA/SPEEK blend composition on the rejection and the product rate efficiencies of chromium ions from aqueous streams in the presence of water-soluble macroligand such as polyvinylalcohol at different concentrations.

## 2. Experimental

### 2.1. Materials

Commercial grade cellulose acetate (CA) was procured from Mysore Acetate and Chemical Co. Ltd., Mysore, India. The CA was recrystallized from acetone and then dried in a vacuum oven at 70 °C for 24 h prior to use. SPEEK was kindly supplied by PCI membranes. *N,N'*-dimethylformamide (DMF) and sodium lauryl sulfate (SLS) were obtained from Qualigens Fine Chemicals, Glaxo India Ltd., India, which were of analytical grade. DMF was sieved through molecular sieves (Type-4 Å) to remove moisture and stored in dry condition prior to use. Acetone and formaldehyde of analytical grade were procured from SRL Pvt. Ltd., India. Anhydrous NaOH pellets and PVA were procured from CDH Chemicals, India Ltd., Mumbai, India, which were of analytical grade. Commercial grade basic chromium sulphate was procured from Golden Chemicals, Mumbai, India. Deionised and distilled water was employed for the preparation of chromium solutions and analysis.

Table 1  
Composition of CA/SPEEK blend membranes

Membrane	Polymer (17.5 wt.%)		Solvent (wt.%)
	CA (%)	SPEEK (%)	
M1	100	0	82.5
M2	90	10	82.5
M3	80	20	82.5
M4	70	30	82.5

### 2.2. Preparation of membranes

CA/SPEEK blend ultrafiltration membranes in the composition of 100/0, 90/10, 80/20, 70/30% were prepared and characterized as described earlier [14]. The membrane formulations are shown in Table 1. Membranes were prepared using standard method of phase inversion technique. The polymer solution was first cast on a smooth glass plate with the help of a doctor blade. The thickness of the membrane was maintained at  $0.22 \pm 0.02$  mm with the help of an oil sheet rolled at both the ends of the blade as followed in earlier studies [14]. The casting conditions are reported in our previous study [14]. Prior to casting, a gelation bath of 2 l consisting of 2.5% (v/v) DMF and 0.2 wt.% SLS in distilled water (non-solvent) was prepared and the bath was ice-cooled to  $18 \pm 2$  °C [14]. After 30 s of solvent evaporation in the casting chamber, the glass plate along with the polymer film was immersed in the gelation bath. After an hour of gelation, the membrane was removed from the gelation bath and thoroughly washed with distilled water to remove the residual solvent and surfactant from the membrane. Similar casting and gelation conditions were maintained for all the membranes. Membranes were cut into circular shapes with diameter  $75 \pm 1$  mm. The circular membrane sheets were subsequently stored in distilled water containing 0.1% of formalin solution to prevent microbial growth.

### 2.3. Experimental setup

Rejection studies were carried out in a batch type, dead end cell (UF cell-S76-400-Model, Spectrum, USA) with an internal diameter of 76 mm, 450 ml capacity and holdup volume of 100 ml, fitted with a Teflon coated magnetic paddle. The schematic diagram of ultrafiltration experimental apparatus is shown in Fig. 1. The effective membrane surface area was 38.5 cm<sup>2</sup> and applied pressure was 345 kPa. A constant agitation speed of 500 rpm was used throughout the study, in order to reduce the concentration polarization. The UF experiments were carried out using pure CA membranes and CA/SPEEK blend membranes in the composition of 90/10, 80/20, 70/30% for the separation of chromium ions using a macroligand by varying the concentration as well as pH.

### 2.4. Removal of chromium ions using macroligand

The chromium solutions containing 200, 400, 600, 800, 1000 ppm of chromium were prepared. These solutions were almost similar to the effluents from the chrome tanning indus-

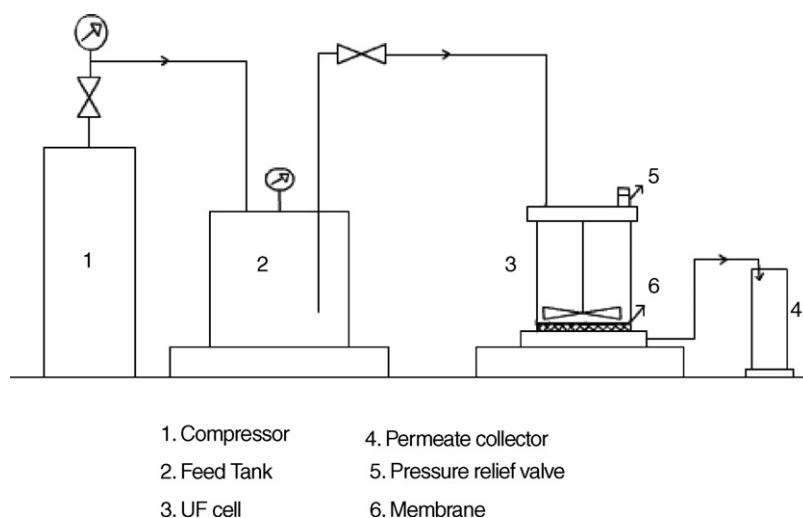


Fig. 1. Schematic diagram of experimental apparatus.

try. The initial pH of the prepared solutions was  $2.5 \pm 0.5$ . The chromium ions present in the solution are complexed using a water-soluble macroligand (PVA), in order to increase the molecular weight [18,19]. The effect of pH was studied by adjusting the pH of the chromium solutions to 3–6, using 0.1 M NaOH for various concentrations of chromium with 2 wt.% PVA. The solution containing PVA and chromium ions were thoroughly mixed and kept for complete binding [20,21]. Pure CA (100%) membrane was chosen for this study. Membrane was mounted in the ultrafiltration kit. The cell was filled with corresponding chromium solution and pressurized under nitrogen atmosphere at 345 kPa, which was maintained constantly throughout the operation. Permeate was collected over measured time intervals in graduated tubes, and the permeate flux was calculated by using Eq. (1)

$$J = \frac{Q}{A \Delta T} \quad (1)$$

where,  $J$  is the permeate flux ( $l m^{-2} h^{-1}$ ),  $Q$  the quantity of permeate collected (l),  $A$  the membrane surface area ( $m^2$ ), and  $\Delta T$  is the sampling time (h). Tube contents were analyzed for chromium concentrations by UV spectrophotometry (Elico Model SL-177, Hyderabad, India) at a maximum wavelength of 420 nm. The percentages of rejections were calculated from the concentration of the feed and permeate using Eq. (2)

$$\%SR = \left[ 1 - \frac{C_p}{C_f} \right] \times 100 \quad (2)$$

where,  $C_p$  and  $C_f$  are concentrations of permeate and feed, respectively.

The effect of composition of CA/SPEEK membranes on the rejection and permeate flux of various concentrations of chromium solution with 2 wt.% PVA at an optimum pH of 6 was studied using the UF kit as described above. The effect of transmembrane pressure was studied by varying the pressure as 69, 138, 207, 276 and 345 kPa in the above procedure using 1000 ppm chromium solution with 2 wt.% PVA at an optimum pH of 6. The concentration of PVA was varied as 0.5, 1.0, 1.5,

2.0 wt.% in 1000 ppm chromium solution at pH 6 and used for chromium rejection through various CA/SPEEK blend membranes as described above. The permeate flux and percentage rejection for all the studies were calculated using Eqs. (1) and (2), respectively.

### 3. Results and discussion

The rejection of chromium ions at various concentrations, with macroligand (PVA) was attempted individually using the blend membranes with 100/0, 90/10, 80/20, 70/30% compositions. In the absence of macroligand, ultrafiltration process cannot be directly applied for ionic level rejections owing to the larger pore sizes of the membranes. To enhance the size and consequently the rejection of chromium ions, a water-soluble chelating polymer (PVA) was used for the complexation of the chromium ions and was subsequently rejected individually from aqueous streams by the CA/SPEEK blend ultrafiltration membranes. These compounds which are bigger than the pores of selected membranes can be retained and flow out in the retentate, whereas, the permeate is relatively free from heavy metal ions depending on the percentage rejection [22].

#### 3.1. Removal of chromium ions using macroligand

The main factors affecting the rejection (%) and permeate flux of the ultrafiltration process are investigated, including composition of the membranes, concentration of the chromium solution, concentration of PVA, pH of the chromium solution and transmembrane pressure.

##### 3.1.1. Effect of pH

The pH of basic chromium sulphate solution of different concentrations with 2 wt.% PVA is varied to study the permeate flux and percentage rejection. The change in the pH increases the intermolecular forces between the chromium ions thereby improves the rejection through membrane. Irrespective of the membrane used, free chromium ions can completely pass

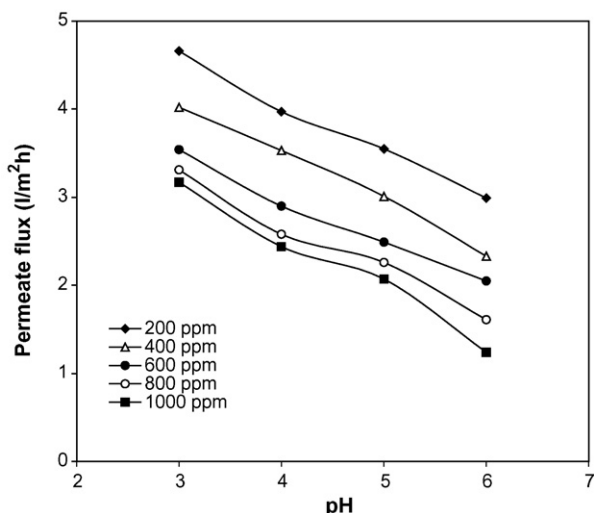


Fig. 2. Effect of pH on permeate flux of various chromium concentrations with PVA (100% CA, 2 wt.% PVA).

through the membrane at a pH below 3 but are almost completely retained at pH higher than 6 due to the precipitation of the chromium hydroxide. In the present study, pure CA membrane was used at constant pressure of 345 kPa, because of its high rejection characteristics [6]. As the pH of the chromium solution increases, the permeate flux decreases and the rejection increases for different concentration of chromium as shown in Figs. 2 and 3, respectively. The rejection is mainly due to the formation of soluble metal hydroxide complexes in the aqueous phase. It is known that the hydrolysis of chromium(III) ion in aqueous solution on treatment with alkali takes place in three steps leading to the formation of chromium hydroxide [23–25] as shown in Eqs. (3)–(5)

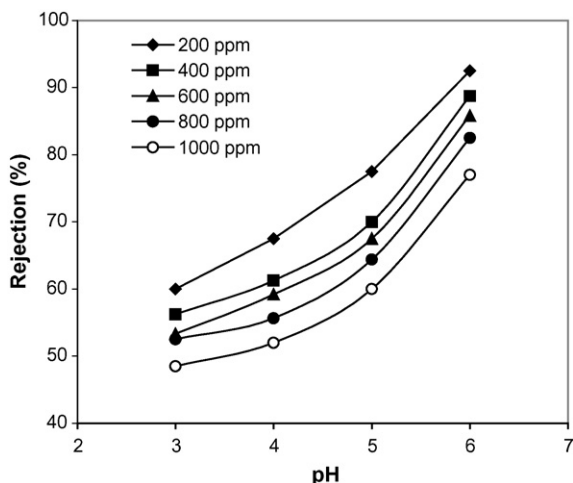
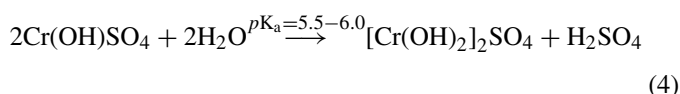
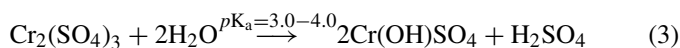


Fig. 3. Effect of pH on rejection of various chromium concentrations with PVA (100% CA, 2 wt.% PVA).

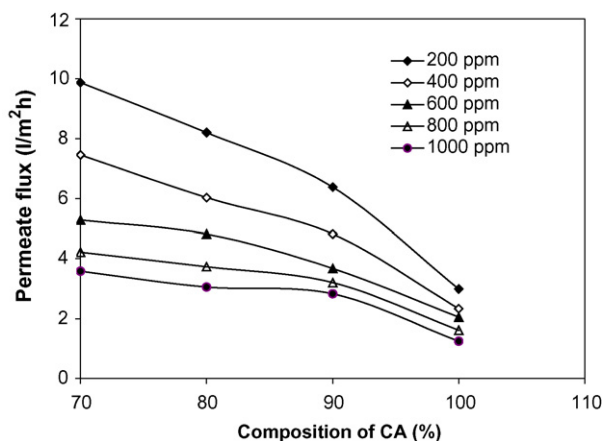
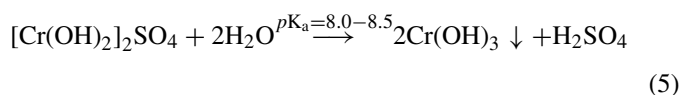


Fig. 4. Effect of composition of CA/SPEEK membranes on permeate flux for various chromium concentrations with PVA (pH 6, 2 wt.% PVA).



It is evident from Eq. (4) that the second hydrolysis leads to chromium complexes in soluble form, which can form polymeric species with macroligand. However, further hydrolysis leads to the precipitation of chromium hydroxide as shown in Eq. (5), which is not present in solution form. Hence, an optimum pH of 6 is chosen for further chromium rejection studies.

### 3.1.2. Effect of chromium concentration

The permeate flux studies of the membranes predict the product rate efficiency and the economics of the membrane process. The permeate flux and rejection of chromium ions in different concentrations such as 200, 400, 600, 800 and 1000 ppm, using CA/SPEEK membranes were carried out individually by complexing with 2 wt.% PVA and the results of the rejection studies are shown in Figs. 4 and 5. Pure CA (100%) membrane offered a

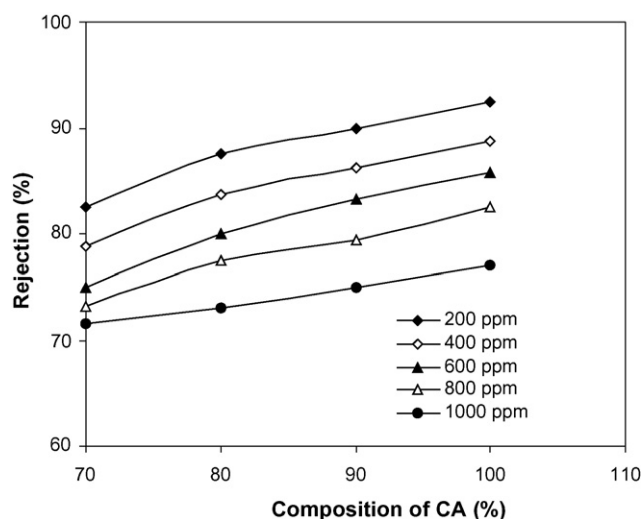


Fig. 5. Effect of composition of CA/SPEEK membranes on rejection for various chromium concentrations with PVA (pH 6, 2 wt.% PVA).



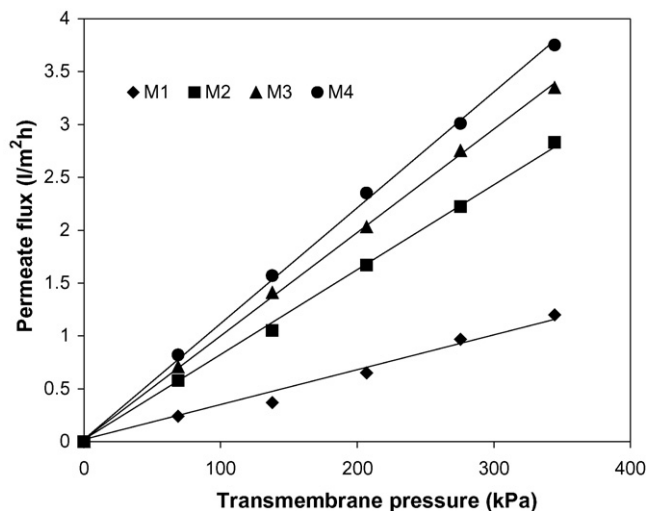


Fig. 6. Effect of transmembrane pressure on permeate flux of chromium solution with PVA for various CA/SPEEK membrane compositions ( $C_f = 1000$  ppm, 2 wt.% PVA, pH 6).

lower permeate flux and higher rejection value of  $2.991 \text{ m}^{-2} \text{ h}^{-1}$  and 93% for 200 ppm chromium(III) ions. This may be due to the smaller macrovoid of the pure CA membranes. When the SPEEK content was increased to 30% in the casting solution, the permeate flux increases to  $9.871 \text{ m}^{-2} \text{ h}^{-1}$  and rejection decreases to 83% for the same concentration of chromium solution. The increase in flux upon increase in SPEEK composition may be due to the higher hydrophilicity of SPEEK in the blend system. The reduction in rejection with increasing SPEEK content may be due to the increase in the pore size in relation to an increasing segmental gap between the polymeric chains during membrane formation. When the SPEEK composition in the blend was increased from 10 to 30%, the rejection decreased for various concentrations of chromium ions. This lower rejection efficiency of 70/30% blend membrane compared to 80/20, 90/10% blend membranes may be due to the presence of higher amount of SPEEK in the blend, which causes changes in the macroscopic structure. Similar trend has also been observed for CA/SPS blend membranes [26].

### 3.1.3. Effect of transmembrane pressure

The effect of transmembrane pressure, viz, 69, 138, 207, 276 and 345 kPa for various compositions of CA/SPEEK blend membranes on permeate flux and rejection (%) of 1000 ppm chromium solution with 2 wt.% PVA are shown in Figs. 6 and 7, respectively. It is evident that an increase in transmembrane pressure results in the increase of permeate flux at a linear rate as the SPEEK content is increased beyond 10 wt.%. Pure CA (100%) membrane offered a lower permeate flux and higher rejection value of  $0.241 \text{ m}^{-2} \text{ h}^{-1}$  and 82% for lower pressure (69 kPa). However, higher permeate flux and lower rejection value of  $1.21 \text{ m}^{-2} \text{ h}^{-1}$  and 77% are obtained at higher pressure (345 kPa) as shown in Figs. 6 and 7. When the SPEEK content in the blend was increased to 30%, the permeate flux increases from 0.82 to  $3.751 \text{ m}^{-2} \text{ h}^{-1}$  and rejection decreases from 77 to 70% as the transmembrane pressure increased from 69 to

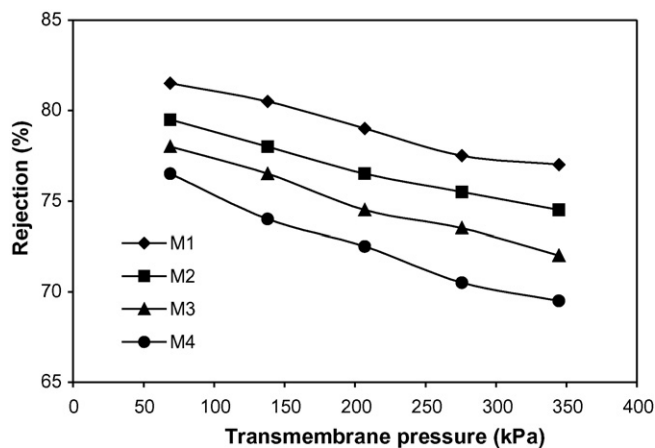


Fig. 7. Effect of transmembrane pressure on rejection of chromium solution with PVA for various CA/SPEEK membrane compositions ( $C_f = 1000$  ppm, 2 wt.% PVA, pH 6).

345 kPa for the same concentration of chromium solution. It was found that the rejection decreases with increasing pressure, and increasing SPEEK content in the membrane. Hence, the rejection of chromium ions depends on the pressure and composition of membranes.

### 3.1.4. Effect of concentration of PVA

The concentration of PVA was varied as 0.5, 1.0, 1.5 and 2.0 wt.% in 1000 ppm chromium solution and permeate flux and rejection (%) through various compositions of CA/SPEEK blend membranes was studied and the results are shown in Figs. 8 and 9, respectively. It is evident that an increase in the PVA concentration results in the decrease of permeate flux but the percentage rejection increases. Pure CA (100%) membrane offers a permeate flux of  $5.02\text{--}1.241 \text{ m}^{-2} \text{ h}^{-1}$  and rejection of 71–77% as the concentration of PVA increased from 0.5 to 2 wt.%. When the SPEEK content was increased to 30% in the casting solution, the permeate flux decreases from 17.51 to  $3.581 \text{ m}^{-2} \text{ h}^{-1}$  and rejection increases from 63 to 72% for lower to higher concentrations of PVA as shown in Figs. 8 and 9,

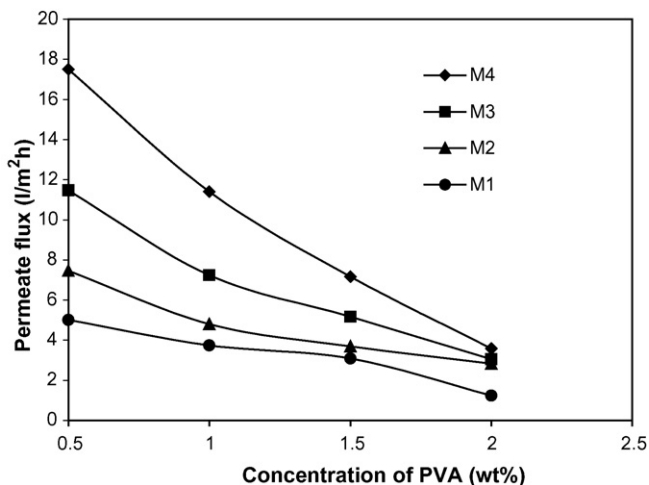


Fig. 8. Effect of PVA concentration on permeate flux of chromium solution for various CA/SPEEK membrane compositions ( $C_f = 1000$  ppm, pH 6).

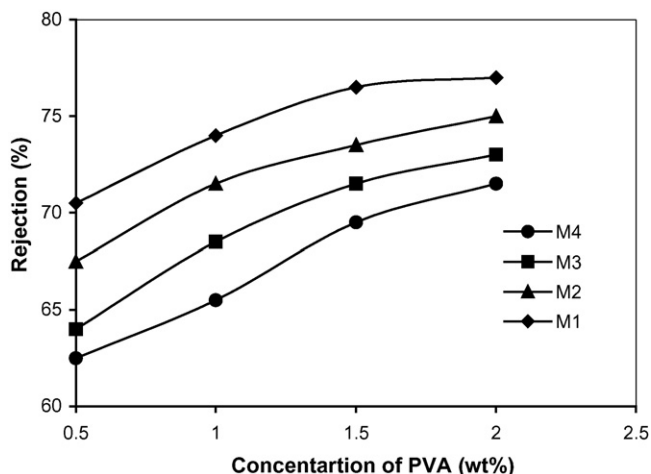


Fig. 9. Effect of PVA concentration on rejection of chromium solution for various CA/SPEEK membrane compositions ( $C_f = 1000$  ppm, pH 6).

respectively. As the PVA concentration increases, the permeate flux decreases and percentage rejection increases for a particular membrane composition, because of the increase in the molecular size of the feed solution.

#### 4. Conclusions

In this work, it has been demonstrated that ultrafiltration assisted by complexation is a promising separation technique applied to purification of effluents containing heavy metals such as chromium. CA/SPEEK blend ultrafiltration membranes with various polymer blend compositions were subjected to the rejection of basic chromium sulphate solutions with a macroligand (PVA). It has been shown that it is possible to separate as much as 93% chromium from a 200 ppm feed solution with 2 wt.% PVA using a membrane of 100% CA at 345 kPa, at an optimum pH of 6. It was also shown that the incorporation of the SPEEK polymer in the blend composition and the concentration of the macroligand play a vital role in determining the separation and product rate efficiencies of the prepared membranes. It was found that the rejection decreases with increasing pressure, concentration of the solute and content of SPEEK in the blend membrane as well as decreasing pH. Although the present study is able to separate the chromium from 200 ppm feed solution up to 93% and yield the permeate in fairly pure form, the separated chromium can not be directly used since it is complexed with PVA. In principle, it would be possible to decomplex the chromium–PVA by acidifying the retentate solution to a pH below 1. Such a study would allow separating the chromium from macroligand for its possible reuse.

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