Scientific African 4 (2019) e00096

Contents lists available at ScienceDirect

Scientific African

journal homepage: www.elsevier.com/locate/sciaf

Recovery of chromium from tannery industry waste water by membrane separation technology: Health and engineering aspects

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ARTICLE INFO

Article history:
Received 5 April 2018
Revised 4 April 2019
Accepted 27 May 2019

Keywords:
Chemical salt
Dissolved
Heavy metal
Toxicity
Wastewater

ABSTRACT

Wastewater containing heavy metals is serious issue for Environmentalist. High percentages of heavy metal in wastewater effect the surrounding Environment as well as harmful for animal, plant and human health. Tanneries come under oldest process industries and consume huge amount of water in several stages. The wastewater generated from tannery industries having large amount of suspended solid, organic, inorganics and heavy metals. To reuse the tremendous amount of wastewater, membrane separation technology is preferred. The main aim of this research work is to study the effects different parameters on recovery of chromium from tannery industry by using membrane separation. The results indicated reverse osmosis was suitable for recovery at optimum pH 6.6; inlet feed concentration 4200 mg/l, flow rate 0.62 m^3/h, and working pressure 40 bar for maximum permeated mass flux (g/min/cm^2).

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Introduction

Tannery industry makes a significant contribution to the country's export earnings as well as in environment pollution [1]. Approximately 31.4 million kilograms of skin are processed annually in Ethiopia [2]. The tanneries discharge 3000 l of waste water, 100 kg of processed hides and the annual discharge of 9420 kl [3]. In chrome tanning, 276 chemicals and 14 heavy metals are used in process, which play major role for water pollution [4]. They discharge large volumes of effluents, because except one or two process in the tannery industry, all the processes are wet processes and generate huge quantities of liquid wastes. The effluents are far from the desired level for acceptance due to heavy load of pollutants like chromium, chlorides, sodium, dissolved solids, BOD, COD, Nitrogen and suspended solids [5].

Chromium has more importance in different metallurgical processing; chromium is traded in world market either as high-grade ore or intermediate like ferrochrome/charge chrome [6]. Problem of chromium disposal can be greatly minimized if recovery the chromium is made to the maximum extent possible. Numerous physical and chemical methods such as adsorption [7], sedimentation [8], electrochemical processes [9,10], biological operations [11], cementation [12], coagula-

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https://doi.org/10.1016/j.sciaf.2019.e00096
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Table 1
Wastewater characteristic of tannery industry.

S. no	Characteristics	Parameters
1	pH	5.3
2	Total chromium	200 mg/l
3	Total Dissolved Solid	3000 mg/l
4	Total Suspended Solid	2100 mg/l
5	BOD	5000 mg/l
6	COD	3800 mg/l

Table 2
Membranes used in the experiments and their specifications.

Membrane type	Average working pressure(bar)	Internal diameter	Length (m)	Effective length(m)
AFC 99	30	12.5	1.20	1.10
AFC 30	20	12.5	1.20	1.10
FB 200	6	12.5	1.20	1.10

tion/flocculation [13], filtration and membrane processes [14,15], chemical precipitation and solvent extraction [16,17] have been employed for the minimization of the pollution level in wastewater only not for recovery. Few methods like membrane separation technology are used for treatment as well as recovery [18].

Membrane separation is a process in which a semi permeable membrane is used to retain species of low molecular weight and a pressure is applied as driving force to revert the solvent natural tendency of passing from a more diluted solution to another more concentrated one [19]. The semi-permeable membrane is a thin film that is constructed by different materials and it is assembled in order to support a high Trans- membrane pressure. There are four types of industrial developed membrane separation processes which are microfiltration, ultrafiltration, reverse osmosis, and electrodialysis [20]. The range of application of the three pressure-driven membrane water separation processes: reverse osmosis, ultrafiltration and microfiltration Ultrafiltration and microfiltration are basically similar in that the mode of separation is molecular sieving through increasingly fine pores [21]. Although reverse osmosis, ultrafiltration and microfiltration are conceptually similar processes, the difference in pore diameter (or apparent pore diameter) produces dramatic differences in the way the membranes are used [22]. With this impression an attempted has been made for eco-friendly recovery and recycling technologies for the treatment of waste water before their disposal.

The main aim of the study is to select best membrane type for the recovery of Chromium (VI). The effect of applied pressure, feed concentration and inlet flow rate on the recovery of chromium using membrane separation

Material and methods

Material

The raw waste water was collected from Kombolcha Tannery Share Company, Ethiopia and stored in 20 °C until used. The waste water quality parameters are mention in Table 1.

Experiment

Membrane modules and machines

The experiment test was carried out with Arm-field lab scale setup Reverse Osmosis (RO) fitted with ultrafilteration unit (UF/RO), shown Fig. 1. The FT18 is fully self-contained in a mobile cabinet having tubular module which can accommodate six 1.2 m PCI membrane types in series was used. The unit has a pack of both reverse osmosis, nanofiltration and ultrafilteration unit membranes. Three different membranes of different average working pressure and similar dimensions are used. The different membrane types, their average working pressure, as specified on the manufacturer catalog and their dimensions are listed in Table 2.

A 12 l volume of the tanning wastewater with low content of fats and denaturalized proteins was treated using different pressure, inlet flow rate and initial concentration of pollutant. The membrane behavior was established from the permeate flux and the rejection of the system majority components. In order to value both aspects, different permeate samples were extracted based on the designed experimental procedure.

Theory

Salt and water permeate reverse osmosis membranes according to the solution diffusion transport mechanism, The water flux, J_i, is linked to the pressure and concentration gradients across the membrane by the equation

$$J_i = A(\Delta p - \Delta \pi) \tag{1}$$

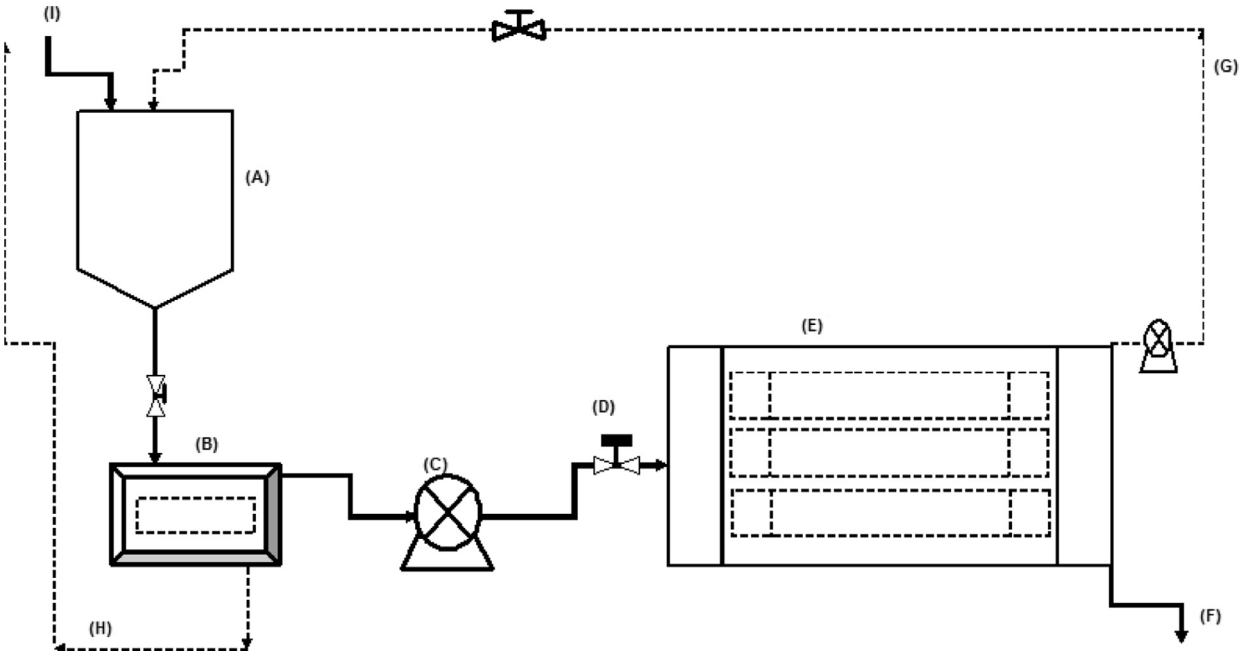

Fig. 1. Experimental setup for chroium recovery (A) storage tank; (B) container; (C) pump; (D) valve; (E) membrane unit; (F) chromium recovered; (G) rejection line; (H) overflow for container and (I) feed.

where Δp is the pressure difference across the membrane, $\Delta\pi$ is the osmotic pressure differential across the membrane, and A is a constant. As this equation shows, at low applied pressure, when $\Delta p < \Delta\pi$, water flows from the dilute to the concentrated salt-solution side of the membrane by normal osmosis. When $\Delta p = \Delta\pi$, no flow occurs, and when the applied pressure is higher than the osmotic pressure, $\Delta p > \Delta\pi$, water flows from the concentrated to the dilute salt-solution side of the membrane.

The salt flux, J_j, across a reverse osmosis membrane is described by the equation

$$J_j = B\left(C_{jo} - C_{jl}\right) \tag{2}$$

where B is the salt permeability constant and C_{jo} and C_{jl}, respectively, are the salt concentrations on the feed and permeate sides of the membrane. The concentration of salt in the permeate solution (c_{jl}) is usually much smaller than the concentration in the feed (c_{jl}), so the equation can be simplified to

$$J_j = B\left(C_{jo}\right) \tag{3}$$

It follows from these two equations that the water flux is proportional to the applied pressure, but the salt flux is independent of pressure. This means that the membrane becomes more selective as the pressure increases. Selectivity can be measured in a number of ways, but conventionally, it is measured as the salt rejection coefficient R, defined as

$$R = 100\left(1 - \frac{C_{jl}}{C_{jo}}\right) \tag{4}$$

The salt concentration on the permeate side of the membrane can be related to the membrane fluxes by the expression

$$C_{jl} = \rho_i \frac{J_j}{J_i} \tag{5}$$

where ρ_i is the density of water (g/cm^3). By combining the above two equations the membrane rejection R can be expressed as

$$R = 100\left(1 - \frac{\rho_i.B}{A(\Delta p - \Delta\pi)}\right) \tag{6}$$

Analysis

Ultra-violet /Visible spectrophotometer was used to detect the color of the wastewater, digital pH meter was used for pH measurement and calibrated with sodium hydroxide (1M NaOH) and hydrochloric acid (1M HCl) solutions, Oxi-Direct

Table 3
Chrome recovery with respect to permeate mass flux and Optimum pH.

S. no.	PH	Feed conc. mg/l	Working pressure, bar	Inlet flow rate m^3/h	Permeate mass flux, g/min	Chrome recovery (Mass%)
1	3.5	2500	30	0.62	16.0141	0.99965
2	5	2500	30	0.62	18.1691	0.99974
3	6.8	2500	30	0.62	23.0901	0.99989
4	7.9	2500	30	0.62	19.7937	0.99983
5	10	2500	30	0.62	15.0988	0.999656
6	12	2500	30	0.62	13.9822	0.99961

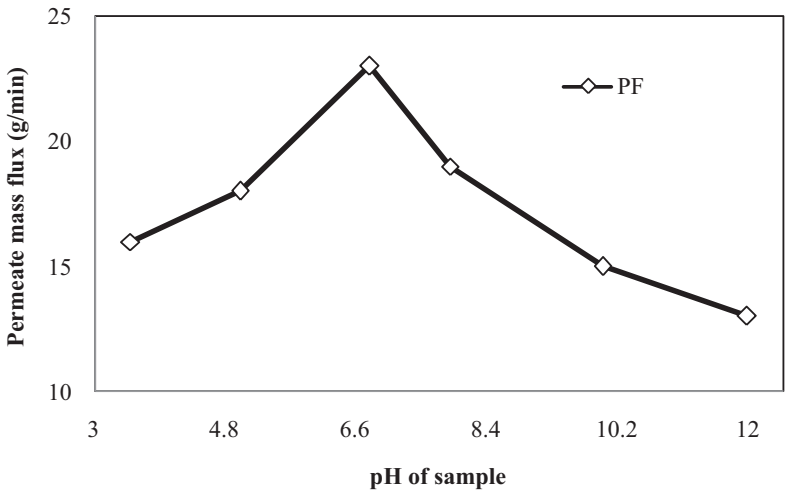

Fig. 2. Effect of pH on permeate flux ate constant feed concentration of 2500 mg/l, inlet flow rate of 0.62 m^3/h and working pressure of 30 bars.

BOD measuring instrument and some reagents were used to measure BOD of the wastewater samples and the permeate of the wastewater [23]. HANNA COD reactor, multi-parameter bench photometer, and reagents were used for COD measurement. Digital weighing balance were used for permeate flux measurement. Turbidity was measured using HACH 2100N turbidimeter.

Results and discussion

Optimum pH determination

To optimized the working pH, an experimental test was carried out at constant feed concentration of 2500 mg/l, inlet flow rate of 0.62 m^3/h and working pressure 30 bars with respect to permeate flux. The chrome recovery with respect to permeate mass flux and working condition is mention in Table. 3**.** It can be observed from Fig. 2 that the optimum pH is around the neutral range hence the excessive alkalinity and acidity faced has to be brought down to the state of neutrality by adding appropriate acid and base respectively. The permeate flux increase starting from the very acidic pH to the neutral point where it reaches maximum and decreases as the alkalinity increases. If pH adjustment is done before running the membrane, it will have an optimum yield. This might be due to speciation of metals differ at different pH ranges, care has been taken in order to find the required metal valence state existing at different pH ranges [24]. Hence at neutral pH Cr (III) is oxidized easily with the help of dissolved oxygen in fresh water or by any organic matter to Cr(VI). As it can be seen in Table 1 the optimum value of pH should be selected by producing a quality permeates. The pH range defined easily what kind of chromium ion can be found at different pH ranges and helps for monitoring of Cr(VI). Further Cr(III) in industrial effluent discharges becomes more important because it can reveal a higher emission of Cr(VI). The permeate quality is also dependent on the pH of the wastewater; the lowest concentration is obtained at pH value of 6.8 which is clearly found at neutral pH.

Effect of feed (inlet) flow rate on permeate flow rate

To determine the effect of feed flow rate the experiment was carried out at different feed concentrations 4500 mg/l, 2500 mg/l and 700 mg/l at different working pressure 25 bar, 30 bar and 40 bar. The graphically representation is shown in Fig. 3. In Fig. 3(a) feed concentration was fixed to 700 mg/l and pressure varies from 25 bar, 30 bar and 40 bar. The maximum permeate flux 18 $g/min/cm^2$, 23 $g/min/cm^2$, and 34 $g/min/cm^2$ was observed when pressure 25, 30, and 40 bar and inlet flow

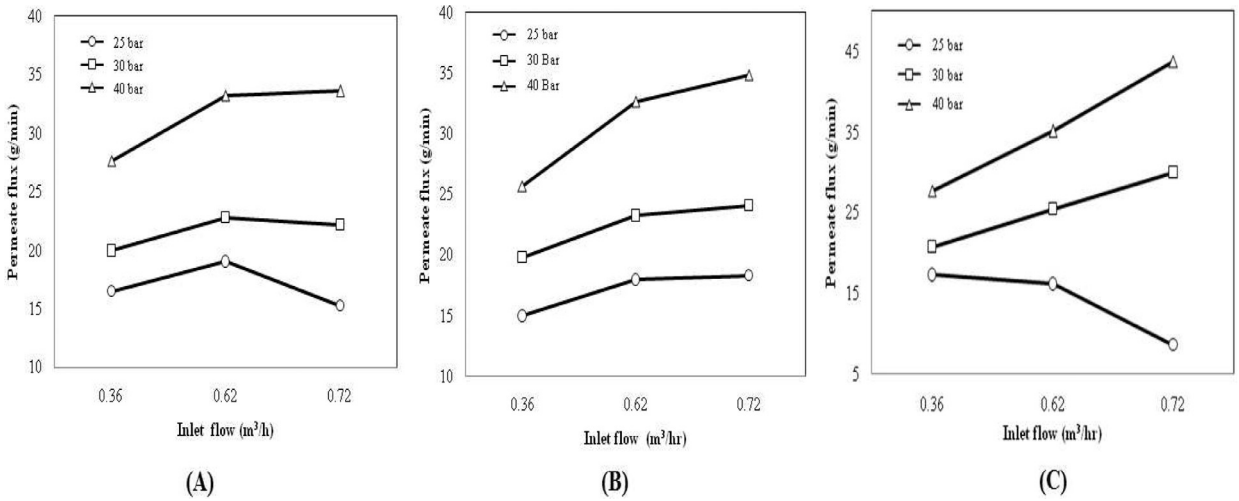

Fig. 3. Effect of different inlet flow rate at different feed concentrations (A) 4500 mg/l, (B) 2500 mg/l and (C) 700 mg/l at different working pressure 25 bar, 30 bar and 40 bar.

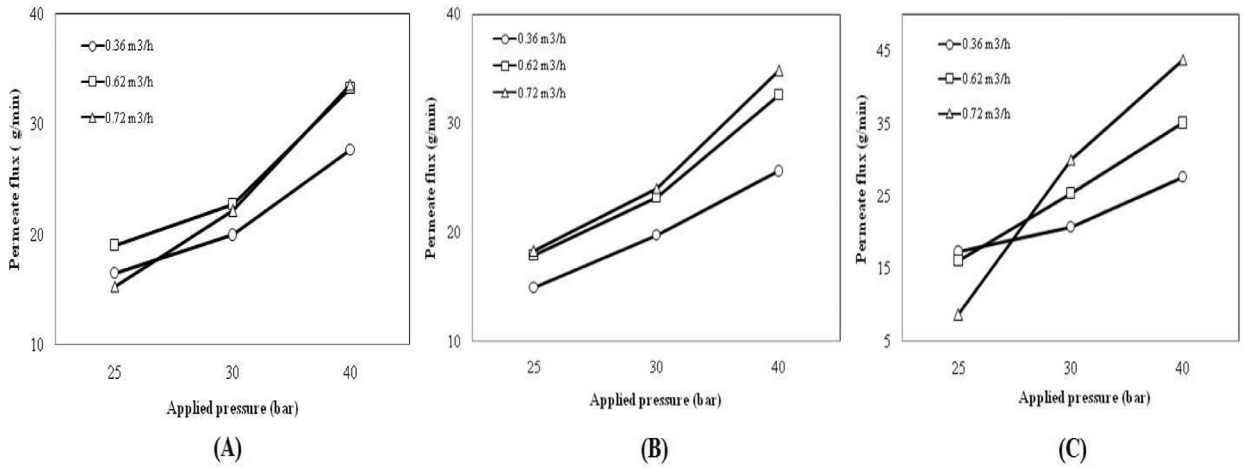

Fig. 4. Effect of applied pressure at different inlet flow rate (A) 0.36, (B) 0.62 and (C) 0.72 m^3/h and feed concentration 700, 2500, 4200 mg/l.

rate was 0.62 m^3/h. It was found that at minimum inlet flow 0.36 m^3/h and maximum 0.72 m^3/h efficiency was poor. For Fig. 3(b) when the flow rate 2500 mg/l the permeate flux was increase with increase in inlet flow rate. The maximum permeate flux 35 g/min/cm^2 was found when inlet flow rate 0.72 m^3/h at 40 bar. Similarly in Fig. 3(c) when the inlet flow rate was (4500 mg/l) 0.72 m^3/h permeates flux was 45 g/min/cm^2 at 40 bars. The permeate flow rate proportionally increases with inlet flow rate for all the concentrations and for all the different working pressures. The reasons for permeate flow rate increase with inlet flow rate is due to increasing of the stress exerted on the wall of the tubular membrane. Which is proportional to the flow velocity and has a positive impact on permeate flow rate, and also the reduction of concentration polarization effect (mass transfer phenomena) by the high velocity flow (usually flow at high velocity has turbulent nature) [25].

Effect of pressure on permeate flow rate

To determine the effect of pressure on permeate flux, experiment was carried out at 25, 30 and 40 bar pressure at different inlet flow rate 0.36, 0.62 and 0.72 m^3/h and feed concentration 700, 2500, 4200 mg/l which shown in Fig. 4. From Fig. 4(a) the maximum permeate flux 33 g/min/cm^2 was observed when inlet flow rate is 0.72 m^3/h and feed concentration 700 mg/l at 40 bar pressure. When feed concentration was 2500 mg/l the maximum permeate flux 38 g/min/cm^2 was observed at 40 bars and 0.72 m^3/h inlet flow rate, which shown in Fig. 4(b). Similarly when the feed concentration was 4200 mg/l the maximum permeate flux 45 g/min/cm^2 was found at 40 bars and inlet flow rate 0.72 m^3/h, which is graphically represent in Fig. 4(c). The applied pressure affects both the permeate flux and solute rejection of RO membranes. In the osmosis process the flow of wastewater across membrane was from the dilute side toward the concentrated solution side.

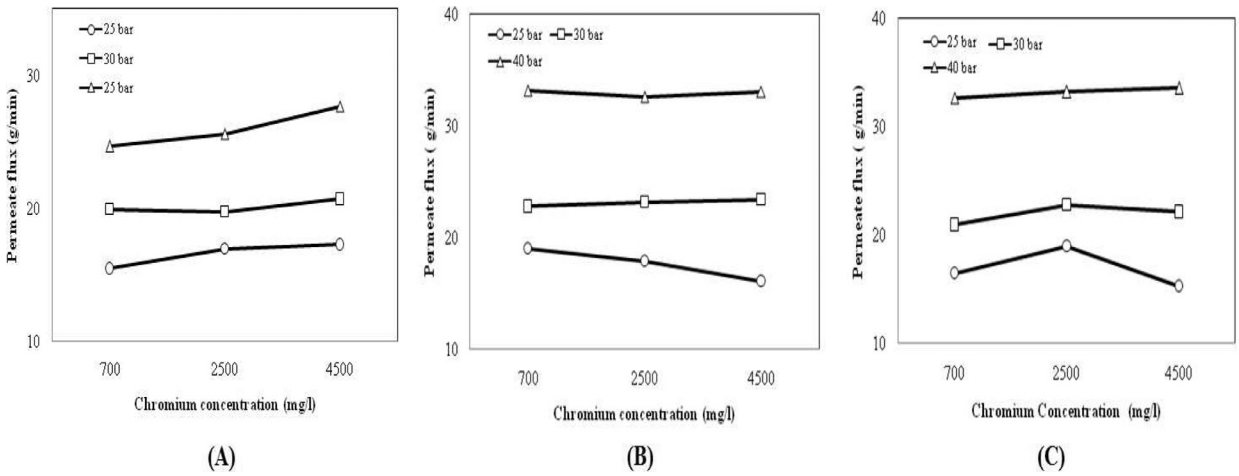

Fig. 5. Effect of different feed concentration at different inlet flow rate (A) 0.36, (B) 0.62, and (C) 0.72 m^3/h a and different applied pressure 25, 30, and 40 bar.

The reverse osmosis technology involves application of pressure to the feed stream to overcome natural osmotic pressure. If excess pressure was applied to the concentrated solution the flow of water is reversed. Besides the slope of the graphs for this case are steeper than the, permeate flow rate verses inlet flow rate, graphs; this shows that working pressure has more effect on the permeate flow rate than inlet flow rate. Increasing the applied /feed pressure also results in increased solute rejection (chromium salt recovery), might be due to reverse osmosis membranes are imperfect barriers to dissolved the salts in the feed. As feed pressure is increases, the solute passage is increasing and the solution is pushed through the membrane at a faster rate than solute can be transported [26,27].

Effect of feed chromium concentration on permeate flow rate

The effect of chromium concentration was carried out at 700, 2500, and 4500 mg/l at different inlet flow rate 0.36, 0.62, 0.72 m^3/h at 25, 30, and 40 bar pressure which is shown in Fig. 5. It was found that feed concentration not affect the efficiency. The slope of the lines on these graphs are a slight deviation from horizontal which indicates that the effect of feed chromium concentration on permeate flow rate is not as significant as the above two parameters. In addition the trend of the curves for all cases is increasing to some extent and then decreasing except some lines which may due to reading or any errors incorporated like experimental leaks, pressure oscillations in factor adjustment. In Fig. 5(a) when the feed concentration 4500 mg/l at inlet flow rate 0.36 m^3/h and 40 bar working pressure, the permeate flux was 28 $g/min/cm^2$ and when feed concentration was 700 mg/l permeate flux was 25 $g/min/cm^2$. In Fig. 5(b) when the inlet flow rate was 0.62 m^3/h and feed concentration was 4500 mg/l, the permeate flux 33 $g/min/cm^2$ was almost similar to 700 mg/l feed concentration and 32 $g/min/cm^2$ permeate flux. Similarly in Fig. 5(c) when the inlet flow rate was 0.72 m^3/h at 40 bar the permeate flux 34 $g/min/cm^2$ at 4500 mg/l and 33.5 $g/min/cm^2$ flux when feed concentration was 700 mg/l. It may be due to the fast clogging of the membrane pores by the chrome particles; because in that case inter particular interaction between the chrome particles is relatively low, which may lead them to occupy any available free space easily and quickly. So the increase in concentration in such cases will have positive impact on permeate flow rate by increasing inter particle interaction, which reduces quickly and easily clogging of membrane pores. But after some critical concentration the dominating factor on the permeate flow rate quantity will not be the clogging or unclogging of the membrane pore rather it will be the concentration polarization effect that becomes dominant. If permeate flux is increased (and feed pressure remains constant), the chrome salts in the residual feed become more concentrated and the natural osmotic pressure will increase until it is as high as the applied feed pressure [28]. This can negate the driving effect of feed pressure; slowing or halting the reverse osmosis process and causing permeate flux and percentage recovery to decrease and even stop [29].

Physical characterization of recovery

The scanning electron micrograph of before used membrane and after used is shown in Fig. 6. It can be seen that from Fig. 6(a) membrane pores are free from chrome particles and more active space is available. Before recovery of chromium, membrane looks like sponge. After recovery of chromium, membrane morphology pores are blocked with chrome particles and damage the membrane surface (Fig. 6(b)). The size of chromium particles are in microns. Chromium can be collecting either back wash or mechanically [30]. Fig. 7 shows (XRD) maximum percentage of chromium in present in recovery sample. Removal of chromium dissolved in tanning wastewaters were obtained with a polyamide semi permeable membrane. The presence of proteins in the tannery industrial effluent affects the permeate flux obtained by reverse osmosis. When the

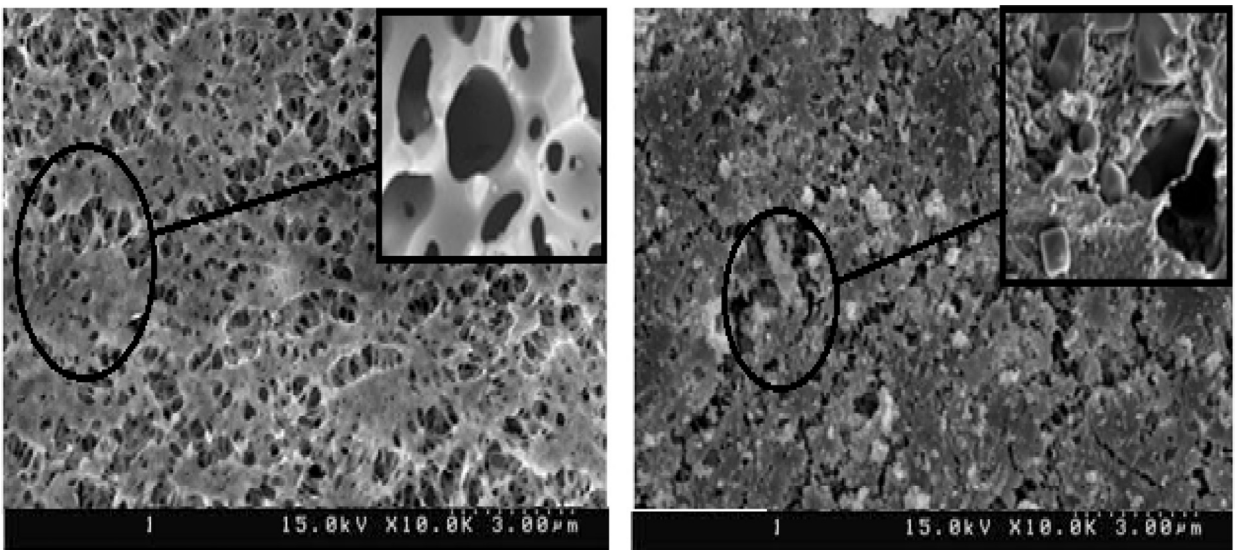

Fig. 6. Scanning electron micrograph of membrane before and after recovery of chromium.

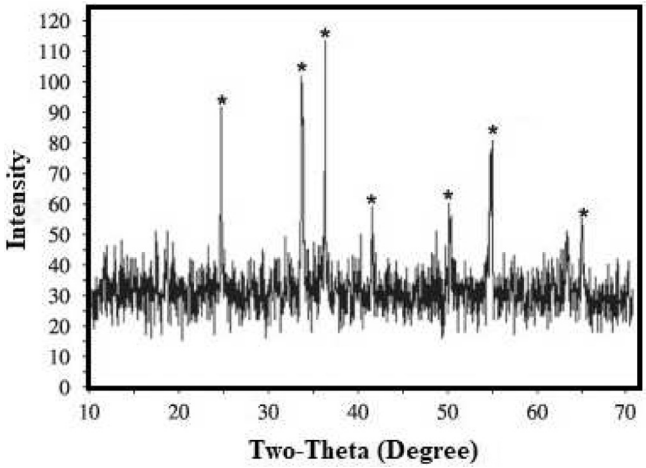

Fig. 7. X-ray diffraction of chromium recovery.

protein concentration in the feed is high, a layer of rejected molecules is formed over the membrane surface (gel layer). Such gel layer adds an additional resistance to the mass transport through the membrane, causing a lower permeates flux [31,32].

Fourier transform infrared spectroscopy

The fourier transform infrared spectroscopy (FTIR) spectra of membrane before chromium loaded; chromium loaded and after chromium loaded is presenting in Fig. 8. The peaks of before and after chromium recovery band indicates that at 1500 cm^{-1} to 1400 cm^{-1} band occurred due to N–H and H–C–H. In addition there was O–H stretching of water, which gives a broad band between 3200 and 3900 cm^{-1}. The broad peak that lies at 1750 cm^{-1} was ascribed to C=O stretch and H–C–H stretch of amide group. In addition it was also due to O–H bending band, all these vibrations are not clearly determined. There are group of peaks below 1400 cm^{-1} that were assigned to H–C–H rocking vibrations. From these two spectra, it was to be illustrating that the membrane stretches are affected by the metal ions from chromium contain water. The FTIR spectra of chromium loaded membrane show the band between 3100 and 4000 cm^{-1} are due to O–H stretch of water and O–H stretch. The broadening of the band demonstrates widespread hydrogen bonding. The H–C–H vibrations give their quality peaks below 3000 cm^{-1}, but they are also overlapped with their main band. The bonding modes of water, amide, carboxylic acid C=O stretching and H–C–H bending modes give a broad intense band at 1800 and 1000 cm^{-1} [33].

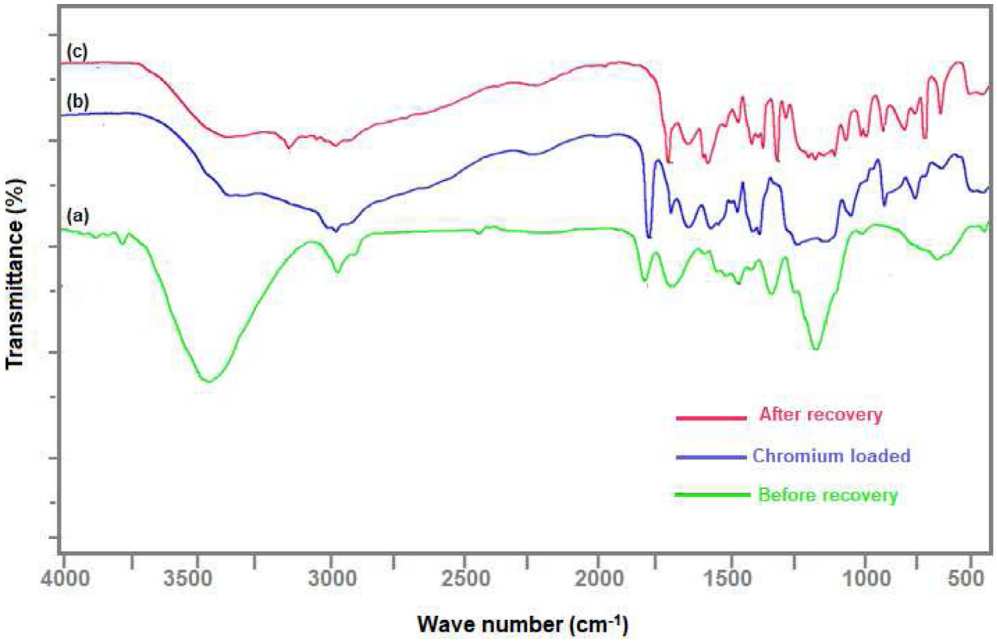

Fig. 8. Fourier Transform Infrared Spectroscopy of (a) before chromium recovery; (b) chromium loaded and (c) after chromium loaded.

Conclusion

Finally using membrane separation methodology for chromium recovery has positive impact. In this research work three types of membrane were employed and AFC 99 was found to suitable for chromium from tannery wastewater. At optimum condition pH 6.6; inlet feed concentration 4200 mg/l, flow rate 0.62 m^3/h, and working pressure 40 bar for maximum permeated mass flux 45 g/min/cm^2 was achieved. Besides selecting an appropriate membrane type, it is also important to distinguish significant operating parameters, which greatly influences the permeate flow and quality. Working pressure, inlet flow rate of the tannery wastewater and initial concentration of wastewater were also found to be influential operating parameters. Concentration of wastewater was shown to have a negative influence on the permeate flow rate. It's recommending to run reverse osmosis process at neutral range pH for chromium recovery. To reduce energy consumption the pressure should be adjusted at medium level, around 30–35 bars. The flow rate should be adjusted also at medium level between 0.6 and 0.66 m^3/h. Samples should be pretreated to avoid clogging and reduce the energy excreted to filter the wastewater.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.sciaf.2019.e00096.

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