



Preparation and characterization of ceramic microfiltration membranes for removal of Cr (VI) and Pb from electroplating effluent

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Present work deals the removal of Cr (VI) and Pb from electroplating effluent (EPE) using ceramic membrane MD-1 (ceramic membrane without coating) and MD-2 (ceramic membrane with chitosan coating). Uniaxial compaction followed by sintering process has been applied to prepare the membrane from locally available clay. Results indicate that maximum removal of 65% Cr (VI) and 68% Pb has been achieved by use of MD-1 membrane, while, MD-2 removed 81% Cr (VI) and 93% Pb, from the initial feed concentration of 55.3 mg/dm³ Cr(VI) and 3.5 mg/dm³ Pb at optimum operating condition pH 3.5 and applied pressure 300 kPa.

Keywords: Chitosan, Electroplating effluent, Ceramic membrane, Microfiltration, Cr (VI), Pb

Electroplating industries (EPI) are major source of metals discharge in water stream. Most of the water sources are polluted by metal ions such as chromium, cadmium, copper, lead, nickel and zinc, which leads several diseases like skin irritation, stomach problem and cancer etc. As contaminated wastewater reach in both surface water and ground water, therefore, it can easily come into contact of human bodies, animals and plants.

Electroplating (EP) is a process to deposit a metallic layer on actual metal surface by use of electrochemical devices. At the time of EP process, small amount of metal become uncoated gets discharge along the processing water. Among the various metals, the chromium and lead is widely used coating material. Discharge of untreated EPE to aquatic system is harmful to plants, animals and human. Central Pollution Control Board (CPCB) a pollution regulatory agency of India has fixed the standard discharge norms 2 mg/dm³ for Cr and 0.1 mg/dm³ for Pb in inland surface water¹. Therefore, proper treatment of EPE is required before discharge in any natural water system. The conventional chemical precipitation method² is not suitable to treat EPE because it produces undesired sludge containing chemicals, more over it cannot be used for the recycling of either Cr (VI) and Pb. Various other techniques available for removal of Cr (VI) and Pb from waste water are electrocoagulation³,

coagulation⁴, ion exchange⁵, adsorption⁶ and membrane separation processes⁷. High removal efficiency, lesser energy and no need to add any chemicals are advantages of membrane separation process⁸. In the last few decades numerous articles have been reported about constantly growing of ceramic membranes on the treatment of waste water⁹⁻¹¹. It has been reported that the inorganic ceramic membranes have good thermal and chemical stability and good mechanical strength then polymeric membranes¹²⁻²¹. The literature review related to treatment of wastewater using ceramic membrane is depicted in Table 1. To the best of our knowledge, only few works have been reported for separation of Cr (VI) and Pb from EPE. In the present work the preparation of low cost disc shaped ceramic membrane from clay and its application to separate Cr (VI) and Pb has been presented. Two type of disc MD-1 (without coating) and MD-2 (with chitosan coating) were used for microfiltration of EPE. As a cross linking agent glutaraldehyde was used and the cross linked chitosan was deposited on the top surface of the ceramic to reduce its pore size. The hydraulic permeability of water, Thermo Gravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), XRD analysis and Fourier Transform Infrared Spectroscopy (FTIR) were carried out to characterize the membrane. The ceramic membrane that has smallest pore size was used to separate Cr (VI) and Pb from

Table 1 — Literatures for use of ceramic membrane

Investigator	Membrane	Effluent	Initial concentration (PPM)	% Metal removal
Choudhury <i>et al.</i> (2018) [12]	Cellulose composite ceramic membrane	Contaminated water	Cr (VI)=5, Pb=5	Cr (VI)=91.44 Pb=97.14
Kumar <i>et al.</i> (2017) [13]	Tubular ceramic membrane	Simulated waste water	Cr (VI)=250	Cr (VI)= 78
Basumatary <i>et al.</i> (2016) [14]	Zeolite-ceramic composite membranes	Aqueous solution	Cr (VI)=1000	Cr (VI)= 82
Hubadillah <i>et al.</i> (2016) [15]	Ceramic hollow fibre membrane	Aqueous solution	Zn=50, Pb=50, Ni= 50	Zn=99, Pb=99, Ni= 99
Moradihamedani <i>et al.</i> (2016) [16]	Mixed matrix membrane	Aqueous solution	Pb=50, Ni= 70	Pb=99.4, Ni= 96.2
Yin <i>et al.</i> (2016) [17]	Ceramic membrane	Aqueous solution	Pb=30	Pb=100
Stancl <i>et al.</i> (2015) [18]	Ceramic membrane	Aqueous solution	Cr (VI)=5	Cr (VI)=90
Doke and Yadav (2014) [19]	Titania membrane	Aqueous solution	Cr (VI)=100	Cr (VI)=99
Piedra <i>et al.</i> (2014) [20]	Spiral wound membranes	Filtered tap water	Cr (VI)=84	Cr (VI)=99
Vasanth <i>et al.</i> (2012) [21]	Ceramic membrane	Aqueous solution	Cr (VI)=100	Cr (VI)=94

EPE. The performance of membrane was evaluated in terms of permeate flux and rejection of both the metals.

Experimental Section

Clay used to prepare the membrane was collected from NIT, Raipur campus. It was grinded and passed through 200 mesh (sieve size 120 μm). Loba made sodium metasilicate, sodium carbonate, boric acid and CDH made kaolin were used for the preparation of membrane. All materials have their own importance during membrane preparation. Kaolin plays a major role to provide low plasticity and high refractory properties to the ceramic membrane, sodium metasilicate increases mechanical strength by creating silicate bonds; sodium carbonate improves dispersion properties of membrane and boric acid increases mechanical strength by creating metaborates. Hi Media made chitosan was used to produce the ultrafiltration top layer over the ceramic. The Merck made acetic acid and glutaraldehyde was also used in preparation of top layer.

Preparation of the ceramic support and the membranes

The ceramic membrane was prepared in disc shape, which has 50 mm diameter and 5 mm thickness. The composition of material used to prepare the membrane is presented in Table 2. The soil and chemicals were mixed thoroughly and made to dished shape by pressing in uniaxial machine, which was followed by sintering at 800°C. The ceramic membrane was further modified by coating of chitosan at the top using spin coating technique. To prepare the solution of chitosan (1–2 wt %), the chitosan flakes were dissolved in a 3 wt % aqueous acetic acid solution. Further, in this 0.12% (v/v) glutaraldehyde solution was added in 4:3 ratios and

Table 2 — Composition of membranes on dry basis

Composition (%)	Name of the membrane	
	MD-1 (Without Coating)	MD-2 (Chitosan Coated Membrane)
Clay	75	75
Kaolin	20	20
Sodium meta silicate	1.2	1.2
Boric acid	1.2	1.2
Sodium carbonate	2.6	2.6

stirred for 3 min; it promotes crosslink reaction²². Glutaraldehyde releases aldehyde groups, which includes amino groups of chitosan consequently form covalent amine bonds. To stabilize the pore penetration capacity of chitosan, the ceramic membranes were directly dipped in water for 3 h earlier to spin coating. By this, water displayed the air present in the porous structure of membrane. The operating speed of spin coating machine (Apex India Spin NXG-P1) was 3500 rpm for 1 min, 2 min and 3 min coating, where, 2 min coating was used for experiment due to optimization point of view as 1 min coating gave higher pore size and 3 min coating gave very less pore size. At the end of coating process, the membrane was taken out from the solution and dried at 105°C for 5 h in hot air oven, by this water present in membrane was removed. The nomenclatures of membranes were kept uncoated (MD-1) and chitosan coated (MD-2).

Experimental filtration studies

Pure water flux and microfiltration of EPE was carried out in a dead end filtration setup as shown in Fig. 1. The setup was made of stainless steel, which was consisted of two parts, as bottom section and top section. The bottom section had circular base plate in which mould was provided to keep the membrane.

The top part had cylindrical compartment of 300 mL capacity, which was attached together with help of circular flanges. Upper part contained 2 inlets, one for liquid feed and other for compressed gas to maintain the pressure and one outlet for retentate stream. Bottom part had one outlet for permeate stream. The top inlet was connected to compressor to provide air, which maintained necessary pressure in reactor. Permeate flux, J ($\text{m}^3/\text{m}^2.\text{s}$) was calculated using the Eq.1 at different applied pressure 75 to 300 kPa at room temperature by collecting volume of permeate at specified time period for every applied pressure

$$J = \frac{Q}{A \times t} \quad \dots (1)$$

where, Q is the volume of permeate collected (m^3) in time t (s) and A is the effective membrane area for permeation (m^2). The Cr (VI) and Pb were analyzed using AAS. Percentage metal removal was determined by Eq. 2 as below

$$\%R = \frac{C_i - C}{C_i} \quad \dots (2)$$

where, C_i (mg/dm^3) is the initial metal concentration in the feed and C (mg/dm^3) is the final metal concentration in permeate.

Results and Discussion

Membrane characterization

Physical and chemical properties of membrane widely affect the separation, therefore it was

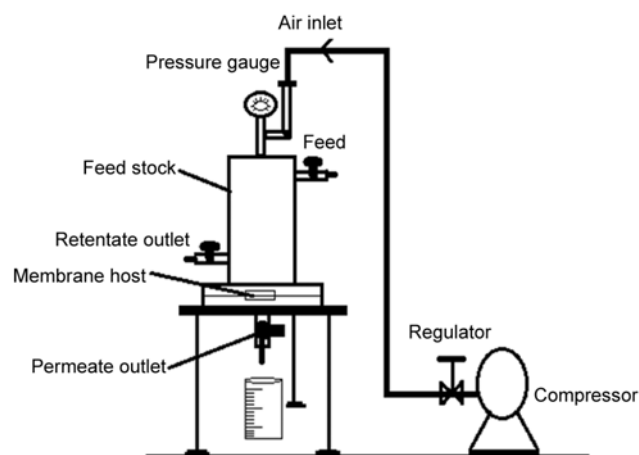


Fig. 1 — Schematic diagram of dead-end filtration unit used in the experiments

characterized for various parameters, which is discussed below.

X-Ray diffraction analysis

Red clay was used for membrane preparation in which silica is present as main component. The clay was characterized by the X-ray diffraction using XRD machine (Model- D8 advance, Hy Pix-400MF, Bruker made, Germany) with 2θ values ranging from 10° to 90° , patterns of XRD is shown in Fig. 2. The sharp peak at 27° indicates material is crystalline in nature. Other peaks were also found at different angles such as from 15° to 20° and 30° to 50° , which indicates the quality of material is good. XRD of chitosan coated membrane MD-2 and uncoated membrane MD-1 (sintered at 800°C) is also presented in Fig. 2b and 2c. The top successive peak of MD-2 obtained at 28° and that of MD-1 at 25° which indicated purity of material. Quartz reflections did not change significantly. In all, the three peaks at $25\text{-}28^\circ$

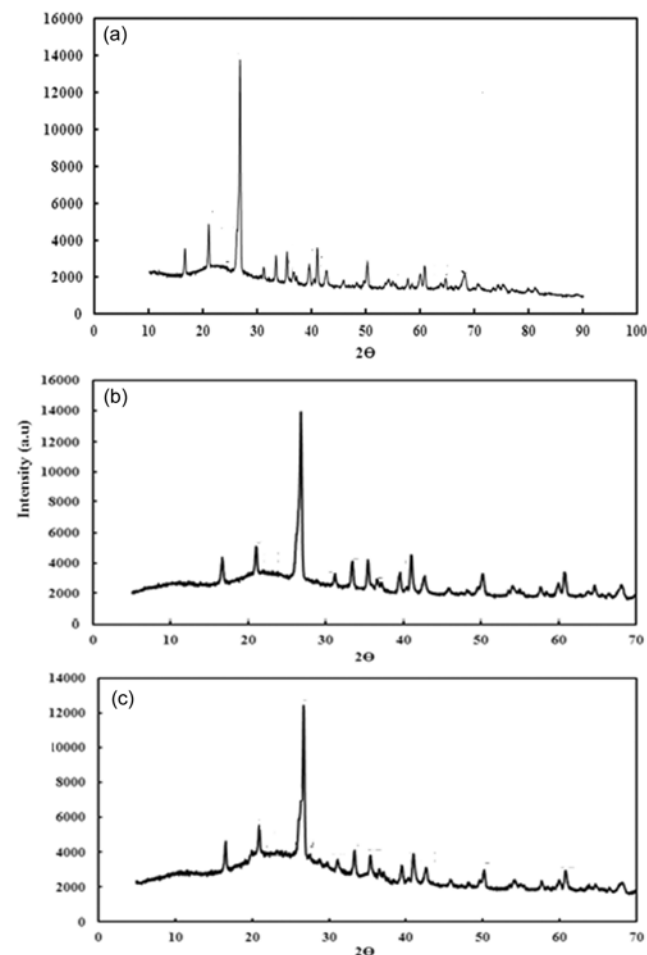


Fig. 2 — XRD image of (a) Clay (b) Chitosan coated membrane (MD-2) and (c) Uncoated membrane (MD-1)

show presence of same phase. Silica and quartz are major constituents present in sand, MD-1 and MD-2. Kaolinite and quartz peak appears in between 25-30°²³.

Morphological assessment

The micrographs images of the membrane MD-1 and membrane MD-2 was determined to visualize shape, size and distribution of pore. For this, scanning electron microscope (SEM) (JEOL JSM, 5600, USA) was used. The results are presented in Fig. 3a and 3b. From the figure it can be seen that the surface of MD-1 is looking white with uniform structure and that of MD-2 is covered with black layer, which is due to chitosen coating on it. SEM images also shows that there are no cracks and discontinuous coating over the membrane surfaces. In the case of membrane MD-1

the pore size is large while chitosan reduces the opening size of membrane, therefore, it gave effective removal of Cr (VI) and Pb from the EPE.

FTIR analysis

The FTIR spectra of chitosan and membrane MD-2 was evaluated using FTIR Model Vertex 70, Bruker made, Germany. The spectra are shown in Figure. 4a and 4b, and listed in Table 3. A characteristic band around 3500 cm^{-1} is representing to $-\text{NH}_2$ and $-\text{OH}$ stretching for both chitosan and MD-2. This confirmed the occurrence of crosslinking reaction of glutaraldehyde with NH_2 groups. Another weak band at around 3000 cm^{-1} is observed in chitosan which indicates $=\text{C}-\text{H}$ stretch. Band at around 1600, $\text{C}=\text{O}$ amide stretch can be seen for chitosan, which is not in MD-2. Similar result is also reported as FTIR trend

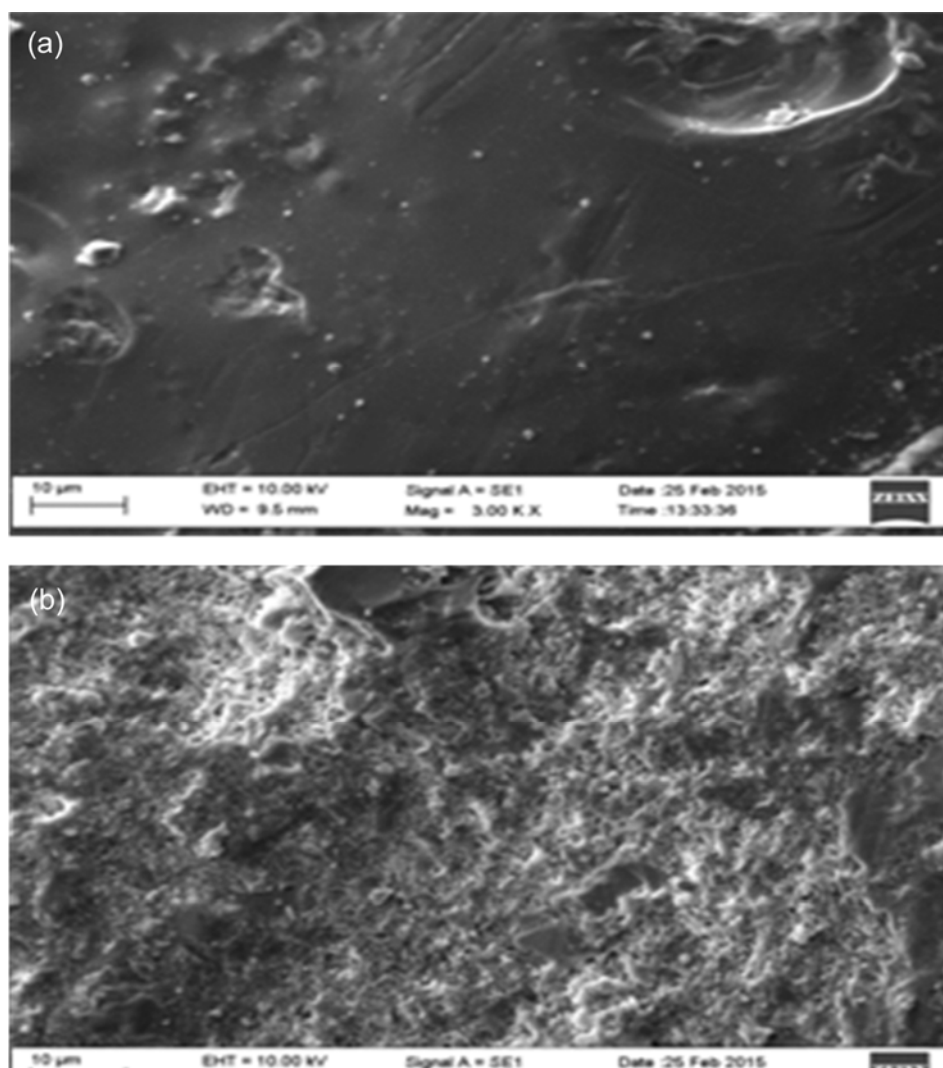


Fig. 3 — SEM images of (a) Chitosan coated membrane (MD-2) (b) Uncoated membrane (MD-1)

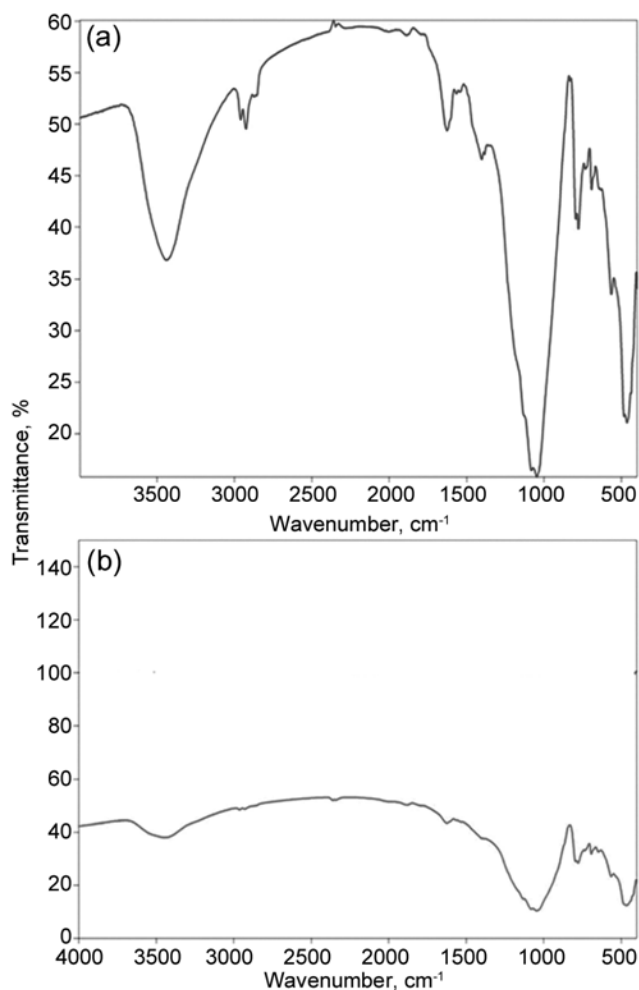


Fig. 4 — FTIR analysis of (a) Chitosan (b) Membrane MD-2

Table 3 — FTIR band analysis of chitosan and glutaraldehyde crosslinked chitosan

Band frequency (cm ⁻¹)	Chitosan	Crosslinked chitosan MD-2	Band assessments
3429	Present	Present	NH ₂
2923	Present	Not present	C-H stress
1641	Present	Not present	Bending vibration of 1° amine
1637	Present	Not present	NH ₂ bend

for the crosslinked reaction of chitosan with tannic acid²⁴. Intensities of bonds are found to decrease in MD-2 to that contain in chitosan.

Thermo gravimetric analysis

Thermo Gravimetric Analysis (TGA) was performed to know weight loss of membrane during heating. For this, thermo gravity analyzer model 4000, Perkin-Elmer was used. The results of thermal analysis are presented in Fig. 5. During heating up to 100°C, the weight loss was 0.11% for MD-1 and

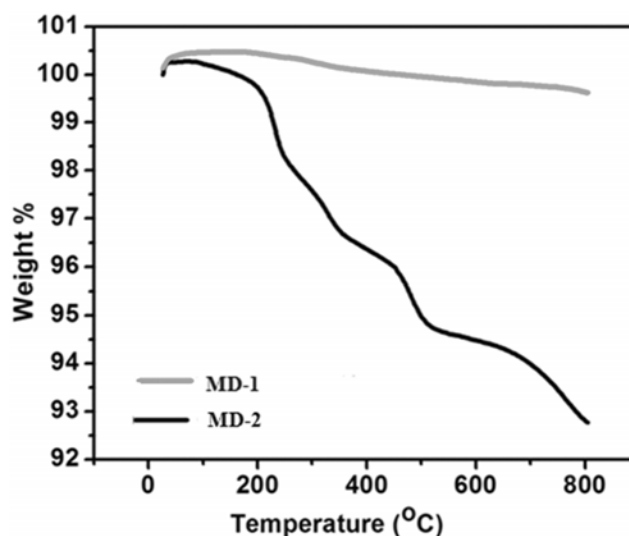


Fig. 5 — Thermo-gravimetric analysis of membrane MD-1 and MD-2.

2.1% for MD-2. Upto 200°C the weight loss of MD-1 was 0.23%, whereas, MD-2 was 3.1%. The higher mass loss of MD-2 is due to the presence of polysaccharide. At temperatures up to 800°C, MD-1 and MD-2 showed overall mass loss of 1.07% and 7.29%, respectively. At temperature above 500°C, there is allotropic transformation of silica quartz from alpha phase to beta phase, which causes weight loss²³ overall very less weight loss was observed in membrane MD-1, while high weight loss in the case of MD-2. This is due to vaporization and decomposition of part of coated material.

Physical properties of membrane

Some physical properties of ceramic membranes, which widely affect to filtration was determined. The surface pore density and pore size was determined from SEM image as per earlier reports²⁵.

Surface pore density

Average number of pores present per unit area of the membrane surface is known as surface pore density, which is presented in Table 4. The pore density was calculated from SEM images (Fig. 3). For membrane MD-1, pore density 1.34×10^{10} and for membrane MD-2 pore density 1.64×10^{10} were observed. In a study by Jana et al., pore density in range of $2.09-13.3 \times 10^{10} \text{ m}^{-2}$ for ceramic membrane prepared from muddy clay have been found²⁵.

Pore size distribution and average pore diameter

For the calculation of pore diameter, SEM images of the membrane were considered. As around 600

Table 4 — Physical properties of membrane

Membrane	Sintering temperature	Average pore diameter		Porosity	Pore density (m ⁻²)	Water permeability (m ³ /(m ² .kPa. s))	Flexural strength (MPa)
		From SEM images (μm)	From water permeability (μm)				
MD-1	800 °C	2.56	1.46	0.423	1.34 x 10 ¹⁰	5.286 x 10 ⁻⁴	2.25
MD-2	800 °C	1.41	1.10	0.264	1.64 x 10 ¹⁰	6.312 x 10 ⁻⁵	2.25

pore diameters were determined by the Image J software (Developed at National Institute of Health and the Laboratory for Optical Computational Instrumentation). Average pore diameter (d_s) was calculated with the help of following equation

$$d_s = \left(\frac{\sum_{i=1}^n n_i d_i^2}{\sum_{i=1}^n n_i} \right)^{0.5} \quad \dots (3)$$

where, n is the number of pores, and d_i is the pore diameter (μm) of the i -th pore. It can be seen in Table 4, the average pore size of membrane MD-2 (1.41 μm) is less than the average pore size of membrane MD-1 (2.56 μm). This happened because chitosan covered the pore of ceramic membrane. Pore size of 2.16-4.73 μm was evaluated by Jana *et al.* [25] for clay based ceramic membrane. The pore size of membranes were also determined for permeability experiments (Table 4), which is smaller than pore size obtained from the SEM images. This may be due to the presence of dead-end-pores not incorporated in water permeability, while in the case of SEM image analysis was integrated.

Porosity

Total porosity of membrane was determined using Archimedes principle by measuring the weight of membrane at different conditions. Following formula was used for this.

$$\mathcal{E} = \frac{M_w - M_d}{M_w - M_a} \times 100 \quad \dots (4)$$

where, M_w (gm) is weight of membranes in wet saturation condition, M_d (gm) is weight of membranes in dry condition and M_a (gm) is the suspended weight of membrane in water. It can be seen in the Table 4, the porosity of membrane MD-1 = 0.423 greater than the porosity of membrane MD-2 = 0.264. Chitosan reduced the porosity of membrane. During sintering, the gaseous products are formed, that make the surface porous and void spaces generated are filled up by other materials through structural densification. Further, the densification increase with increase in

sintering temperature followed by transformation of phase from amorphous to crystalline of clay material²⁶. The low porosity favors the retention of impurities over the membrane. In our case porosity is less than other clay based membranes which were evaluated in the range of 0.43 to 0.85 by Ghosh *et al.*²⁶.

Mechanical strength

Membrane is fixed in module for filtration and pressure is applied on liquid by air or nitrogen which is transferred to membrane, thus, proper strength of membrane is essential. The flexural strengths of MD-1 and MD-2 were calculated by a three point bending strength method. For membrane MD-1 and MD-2, flexural strength was found to 2.25 MPa which is sufficiently high. The values are presented in Table 4.

Water permeation experiment and removal of Cr (VI) and Pb

The prepared membrane was used for water permeation test in pressure range of 75-300 kPa. For good membrane permeation flux should be high, and metal retention should be also high. Effect of pressure on water flux and metals removal is discussed below.

Effect of pressure on water flux

The effect of pressure during microfiltration for pure water flux collection is shown in Figure. 6a and 6b. It can be seen from Figure. 6a and 6b, pure water flux collection was increased with increase in pressure for both the membranes, which is due to enhancement of driving force. Further, the permeate flux varied almost linearly with increasing applied pressure, which is due to fact that there is no significant contribution of additional transport resistance from concentration polarization and adsorption²⁵. The permeate flux was found to decrease with time, which is because of deposition of metal on membrane surface. The value of permeate flux was lies in between $1 - 7 \times 10^5$ m³/m²s¹ for MD-2 and $7-58 \times 10^5$ m³/m².s for MD-1. The permeate flux of the MD-1 and MD-2 was noted to slightly lower for EPE than the pure water. This may due to the osmotic pressure generated by the retained ions, which resulted in reducing of effective pressure across the membrane.

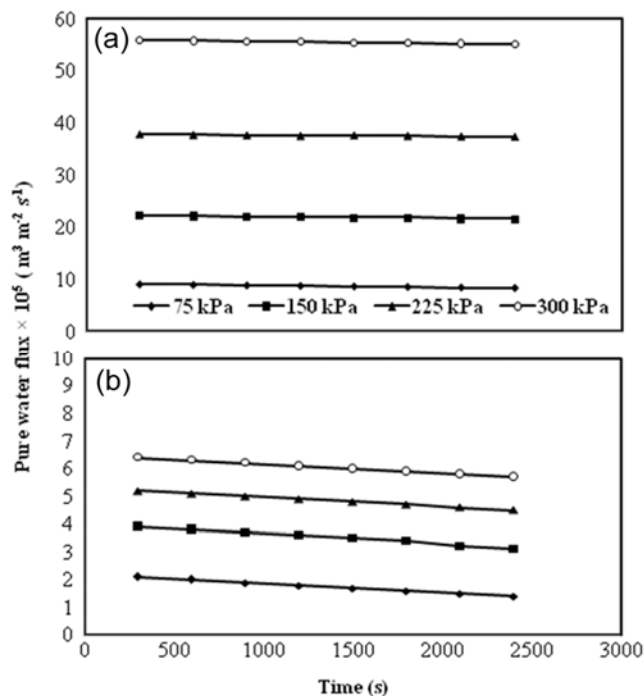


Fig. 6 — Pure water flux as a function of applied pressure (75-300 kPa) for (a) MD-1 (b) MD-2

In addition, the flux collection rate in MD-1 is higher than MD-2, this may be due to pore size of MD-1 is larger as compared to MD-2. In a study permeate flux in the range of $5.4\text{-}313.4 \times 10^5 \text{ m}^3/(\text{m}^2.\text{s})$ has been reported by Ghosh *et al.*²⁶ low permeable flux for MD-2 is due to low pore size.

Effect of pH on Cr (VI) and Pb removal

The pH of the effluent plays an important role for rejection of metals during the filtration process²¹. pH study was performed at pressure 300 kPa. The results are presented in Fig. 7. It has been found that the percentage rejection of Cr (VI) and Pb was decreased with increase in the pH of the effluent for both the membrane (MD-1 and MD-2). At the pH 3.5 maximum 63% Cr (VI) and 67% (Pb) removal was obtained over membrane MD-1, while, membrane MD-2 gave maximum 81% Cr (VI) and 93% (Pb) removal. The coating material (chitosan) reduces the pore of membrane, due to this, metal removal are more. For pH study in between 2 to 9, the pH 3.5 was found to be best. The metal rejection was almost same at pH 2 and pH 3.5 for MD-2, while for MD-1, metal rejection is about 2 % less at pH 2 as compared to pH 3.5. Cr (VI) existed in solution in different ionic forms (HCrO_4^- , CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$), which depends on solution pH and concentration of Cr (VI)²¹. At low pH

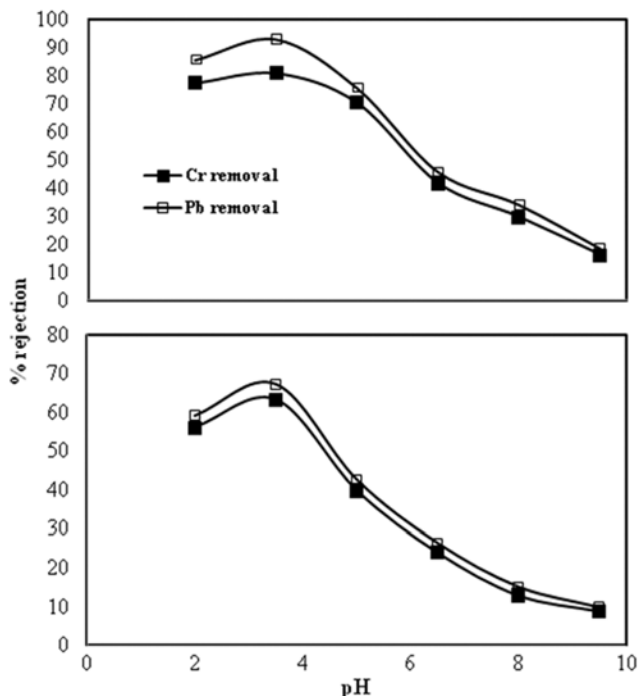


Fig. 7 — Effect of pH on the rejection of Cr (VI) and Pb using (a) MD-2 (b) MD-1. Applied Pr = 300 kPa

HCrO_4^- is the dominant species. The HCrO_4^- has the property to exchange easily with OH^- ions at active surface under acidic condition. Further, at low pH the hydronium ions present, which increases the Cr (VI) interaction causes retention of Cr over the membrane²¹. At near to neutral pH, the Pb contain in four oxidation stage, which changes to two oxidation stage at acidic pH. The formation of lead oxides is expected by reaction of lead with dissolved oxygen at high acidic pH. The lead sulfate (PbSO_4) could be also form at low pH in presence of sulphate anions, which is quite insoluble²⁷. The size of ions and compounds forms are also different at different pH, thus its rejection is varied due to its size. As pH increases, the overall surface charge of the cell becomes negative and hence binding capacity decreases²¹. Due to all these removal of Cr (VI) and Pb was affected.

Effect of applied pressure on Cr (VI) and Pb removal

Applied pressure on effluent highly affect the permeate water flux and removal efficiency of membrane. The pressure applied on water transfer to membrane, therefore, its effect on metal removal was studied. Figure 8a and 8b offered Cr (VI) and Pb removal rate at different pressure for membrane MD-1. At the optimum pH 3.5 and 300 KPa applied

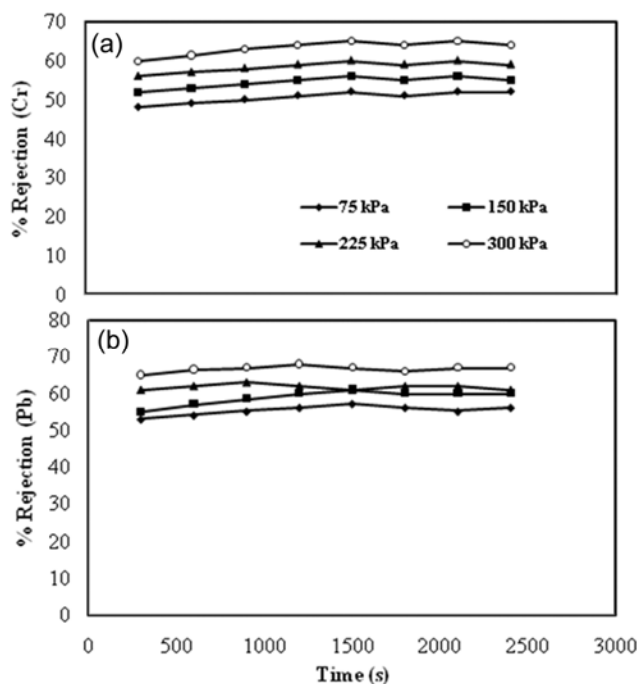


Fig. 8 — Removal of (a) Cr (VI) and (b) Pb using MD-1 as a function of applied pressure (75-300 kPa)

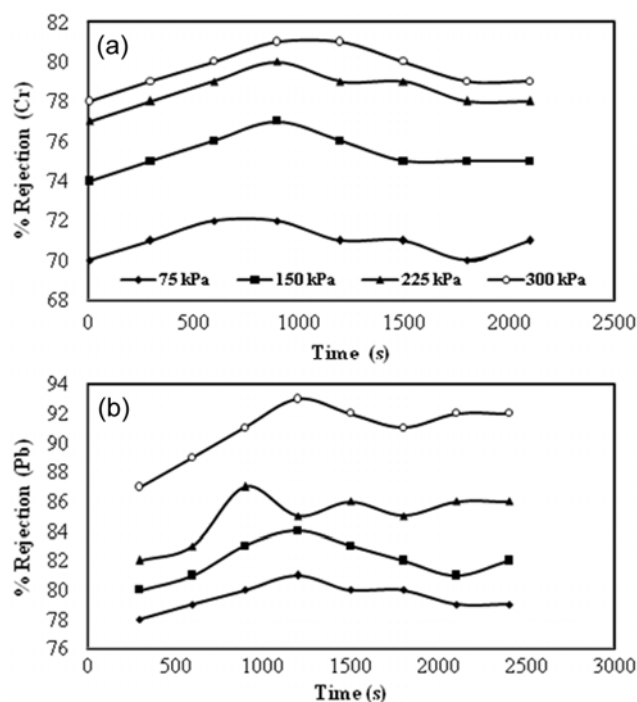


Fig. 9 — Removal of (a) Cr (VI) and (b) Pb using MD-2 as a function of applied pressure (75-300 kPa)

pressure, 65 % Cr (VI) and 68 % Pb removal was observed. Similarly Figure 9a and 9b present the metal removal rate at different applied pressure for membrane MD-2. At the optimum condition (pH 3.5

Table 5 — Cost analysis of the MD-1 membranes from the unit cost of raw materials.

	Raw materials	Material required (kg/m ²)	Unit price (\$/kg or \$/L)	Total cost (\$/m ²)
Support	Clay	9	-	-
	Kaolin	2.1	8.1	17.0
	Sodium carbonate	0.65	8.31	5.401
	Sodium meta silicate	0.395	17.48	4.78
	Boric acid	0.395	10.7	4.22
Coating material	Water	11.1	-	-
	Acetic acid	0.21	8.9	1.86
	Glutaraldehyde solution (25%)	5.34	93.89	418.37
	Chitosan	0.21	660.39	138.7
	Total			590.68

5 cm diameter and 5 mm thick membrane was prepared from 25 g material.

and pressure 300 kPa) 81% Cr (VI) and 93% Pb removal was observed. It can be seen from the figures, the metal removal rate was increased with increase in applied pressure. The removal rate was slightly increased with time (upto 1000 s), after that it became almost constant. In our process the Pb and Cr removal is comparable to that given in Table 1. The membrane used in present studies has low cost, while the membranes used in Table 1 have high cost.

Membrane cost

Verities of membrane are available in market for industrial purpose, which rate is varies at the range of 600–2100 \$/m²²⁵. In present work the thickness of the support (membrane) was 5 mm and total 8.5 dm³/m² solution was needed for the successful spin coating. The details of chemicals used and its price are presented in Table 5. The total price is calculated to be 590.68 \$/m². A part from this additional cost including manufacturing and shipment required, thus, the cost may reach up to 690 \$/m², which is competitive with the cost of the commercial ceramic membranes. The calculated cost is competitive to the cost of other commercial ceramic membrane.

Conclusion

Two types of ceramic membrane have been used. First is ceramic membrane without coating (MD-1) and second is chitosan coated membrane (MD-2). Chitosan was coated using spin coating technique. The membrane has been characterized using XRD, SEM and TGA, indicate that prepared membrane had good quality. Studies demonstrate that membrane

separation process can be applicable for treatment of EPE. The highest 65 % Cr (VI) and 68 % Pb removal with MD-1 membrane and 81 % Cr (VI) and 93 % Pb removal with MD-2 membrane are obtained from the initial feed concentration of 55.3 mg/dm³ (Cr(VI)) and 3.5 mg/dm³ (Pb). The prepared membrane is cost effective as compared to traditional membrane available in market. In terms of price of material required, the cost of membrane is evaluated to be 590.68 \$/m².

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