Performance of sunflower oil as green solvent for Cr(VI) extraction using supported liquid membrane

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ABSTRACT

This work is an experimental study on the extraction of Cr(VI) ions from aqueous acidic solutions using a flat sheet supported liquid membrane. Sunflower oil used as alternative solvent due to the stricter environmental regulations. Aliquat 336 and NaOH solution were employed as carrier and stripping agent. A hydrophobic microporous polytetrafluoroethylene (PTFE) support was impregnated with liquid membrane consist of solvent and carrier. The Cr(VI) pass through the supported liquid membrane from aqueous feed phase to the stripping phase by means of diffusion. Hydrochloric acid (HCl) was used to regulate the feed phase pH. The efficiency of the extraction was optimized as a function of the various parameters: Feed pH, stripping phase (NaOH) concentration, feed concentration, and carrier concentration. The maximum separation of Cr(VI) was about 95.2% in 10 h. Also, the results of experiments were compared with other organic solvents such as kerosene, n-octanol, dodecane, and tetrahydrofurane (THF) to evaluate the efficiency of sunflower oil. Results indicated that Cr(VI) extraction is on average about 0.5%–55% higher with sunflower oil. The optimum conditions were achieved as: pH = 2, NaOH = 0.1 M, feed concentration = 25 ppm, and Aliquat 336 = 35% v/v. The results indicate that the Cr(VI) ions can be efficiently transported through the SLM by using Aliquat 336 dissolved in sunflower oil as a green solvent.

Keywords: Cr(VI); Vegetable oil; Supported liquid membrane; Aliquat 336

1. Introduction

There is an increasing need for energy efficient and economic processes in the removal and recovery of heavy metals for industrial effluents. The heavy metals are among the most common pollutants found in wastewater. These metals pose a toxicity threat to human beings and animals even at low concentration [1]. Separation of heavy metals from aqueous solutions, especially wastewaters have received a considerable amount of attention during recent years due to the concern that heavy metals can be readily absorbed by organisms [2]. One of these heavy metals is chromium. Chromium is a relatively common element and occupies the 21st position of the index of element occurring most commonly in the earth crust and is a common pollutant introduced into natural waters from a variety of industrial wastewaters, including those from the textile, leather tanning, electroplating, steel works, cooling water towers, oxidative dyeing and metal finishing industries, paint, pigments. As a result of unregulated applications and inappropriate waste-disposal practices, chromium contamination of surface, and ground water has become a significant environmental problem [2–4].

Chromium is an element that exists primarily in two different oxidation states, hexavalent and trivalent. The oxidation state of the Cr has a significant effect on the transport and fate of Cr, and on the type and cost of treatment required to reduce Cr concentrations less than regulatory health-based standards. Cr(VI) is far more mobile than Cr(III) and more difficult to remove from water [5]. The properties of Chromium are extremely dependent on the molecular structure of the

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Chromium compound. It is especially depends on the oxidation number of the Chromium. The oxidation number of the Chromium has an important effect on the transport of Chromium. Furthermore, it influences the type and cost of process required to lower Chromium concentrations less than health-based standards [6,7]. Cr(VI) species which known as mutagen and potential carcinogen, having mobile and strongly oxidant characters. On the other hand, Cr(III) is mutagenic, carcinogenic and teratogenic which having a limited hydroxide solubility and low toxicity. Cr(VI) is approximately, 10–1,000 times more toxic than Cr(III) [7–11].

In aqueous environment, Cr(VI) exists in the form of chromic acid (H_2CrO_4) and dichromate salts ($Cr_2O_7^{2-}$). In neutral solutions, Cr(VI) will be present in the form of $HCrO_4^-$ and CrO_4^{2-} at low concentrations. The most probable species of Cr(III) will be in the form of $Cr(OH)^{2+}.4H_2O$ [6,12–14].

Because of the repulsive electrostatic interactions, Cr(VI) anion species are generally poorly adsorbed by the negatively charged soil particles in the environment. Hence, they can move freely in the aqueous solutions. Based on the pH values of the solution, Cr(III) species in aqueous solutions may be in the form of Cr^{3+}, $Cr(OH)^{2+}$, or $Cr(OH)_2^+$. These species are particles due to their positive electric charges. Thus, they are less mobile than the Cr(VI) species in the aqueous environment [6,8,12,13]. In general, the solubility of Cr(VI) salts are higher than those of Cr(III), making Cr(VI) relatively mobile [10]. Consequently, Cr(VI) is far more mobile than Cr(III) in many aquatic system and more difficult to remove from water [6–17].

The Environmental Protection Agency (EPA) has set the maximum concentration of 0.1 mg L^{-1} of hexavalent and trivalent chromium for drinking water and the health effects [18]. While the World Health Organization (WHO) states that the maximum allowable concentration of total chromium is 0.05 mg L^{-1} [19]. Since it is very much required to remove Cr(VI) from the industrial effluents before allowing it to enter any water system or on to the land, several methods have been applied to remove Cr(VI) from aqueous solutions, including adsorption, membrane filtration, ion exchange, and electrochemical treatment methods [20]. Furthermore, there are various investigations on the speciation, pre-concentration, and separation of Cr(III) and Cr(VI) using different methods such as solid-phase extraction, flame atomic absorption spectrometry (FAAS) [21–23], and ion-pair solvent extraction [24]. These Processes have certain disadvantages, which have led us to use other processes such as liquid membranes (LM) [25].

Liquid membranes have a broad potential application for purification of industrials effluents from toxic organic compounds [26]. Liquid membrane processes take advantage of the already established principles of biological treatment and solvent extraction but provide the advantage of reduction in treatment costs [27]. Recently, many researchers have used different types of liquid membrane processes such as bulk (BLM) [28], emulsion (ELM) [29], and supported (SLM) for the separation of Cr(VI). Among these, the Supported liquid membrane (SLM) technique is easy and has high fluxes. The major advantages of SLMs compared with other membrane separation techniques are: (1) High selectivity can be obtained when a proper carrier is chosen. (2) Single step separation method. (3) High concentration gradient of the coupled species is very high. (4) Expensive liquid membrane phases can be used, as the volume ratio liquid membrane phase/aqueous phase is very small. (5) Lower capital and operating cost. (6) Low energy consumption [25].

In SLMs, a microporous support impregnated with the liquid complexing agent separates the feed and product solutions [30]. The SLM may be fabricated in different geometries. Flat sheet SLM is useful for research, but the surface area to volume ratio is too low for industrial applications. Spiral-wound and hollow-fiber SLMs have much higher surface areas of the LM modules [27].

To this day, a number of scientists have studied Cr(VI) transport through SLM by applying various synthetic carriers. Park et al. [31] have studied the facilitated transport of Cr(VI) through a SLM with Aliquat 336 dissolved in toluene as a carrier and measured the overall mass transfer coefficient as 2.019×10^{-6} (m.s^{-1}). Choi et al. [32] have investigated the selective separation of Cr(VI) in aqueous solutions containing Cr(III) by using a SLM, impregnated with Aliquat 336 as a carrier. This result was obtained that the permeability of Cr(VI) was not influenced by concentrations of Cr(VI) and cationic interferences in the feed solution, except for Cr(III). Ashraf et al. [33] have studied the selective separation and pre-concentration of Cr(VI) ions with Alamine 336 SLM, and the flux of Cr(VI) ions were found to be the maximum 3.15×10^{-5} (mol.cm^{-2}.s^{-1}) around pH = 1. Venkateswaran et al. [34] have carried out the transport of chromium(VI) ions from an aqueous feed solution through a flat type SLM containing tri-n-butyl phosphate (TBP) as a carrier. Under the optimum conditions chromium(VI) recovery from real plating wastewater was tested and found to be satisfactory with a flux value of 1.4×10^{-3} (mol.cm^{-2}.s^{-1}). Eyupoglu et al. [35] have dealt with extraction of Cr(VI) by a flat sheet SLM using Alamine 336 as a carrier and calculated the permeation coefficient as 7.88×10^{-6} (m.s^{-1}). Alguacil et al. [36] has studied the transport of chromium(VI) through a Cyanex 923–xylene flat-sheet SLM. The mass transfer coefficient was calculated about 6.7×10^{-5} (m.s^{-1}). Also the transport of chromium(VI) through a Cyanex 921-SLM from HCl solutions has been investigated. The value of the mass transfer coefficient of the aqueous film was obtained 8.7×10^{-5} (m.s^{-1}) [37]. Solangi et al. [38] have reported the transportation of Cr(VI) across SLMs containing p-tert-butylcalix[4]arene diamide. Some investigators have studied the effect of ionic liquids as alternative for conventional volatile organic solvents in order to separation of different metals in different processes [39–41].

In the present work, the results of Cr(VI) transportation through a flat-sheet SLM has been studied. The liquid membrane impregnated with the Aliquat 336 as a carrier dissolved in environmentally friendly solvent (sunflower oil). The NaOH solution was used as stripping solution. The influence of various parameters such as feed pH, strip phase (NaOH) concentration, feed concentration, and carrier concentration has been investigated. Environmentally friendly nature of sunflower oil is the main advantage for this work.

2. Experimental

2.1. Materials

All chemicals used throughout this study were of analytical grade and used without further purification. Sunflower

oil was obtained from local Oila Oil Company (Tehran, Iran). Hydrochloric acid (HCl), Sodium hydroxide (NaOH), and Sodium dichromate dihydrate ($Na_2Cr_2O_7.2H_2O$) were purchased from Merck, Co. (Darmstadt, Germany). Aliquat 336 was purchased from Sigma-Aldrich (Milwaukee, USA) and used as mobile carrier. The support for the membrane was commercially available polytetrafluoroethylene (PTFE) flat microfiltration (MF) with the diameter of 47 mm, pore size of 0.22 µm, thickness of 150 ± 10 µm and porosity of 75%. This support was supplied by from Millipore (Billerica, USA).

2.2. Experimental procedure

Cr(VI) solutions were prepared by dissolving $Na_2Cr_2O_7.2H_2O$ in deionized water. Hydrochloric acid (HCl) was used to regulate the feed phase pH. The stripping solutions were made from NaOH in deionized water in given concentrations. The organic phase used for soaking the filter membrane was preparing by dissolving Aliquat 336 in sunflower oil.

The membranes were prepared by soaking in sunflower oil containing Aliquat 336 for 12 h and then the impregnated membranes were left to drip for a few seconds before being placed in the setup. The batch transport experiments were performed by using a two–circular compartment permeation cell with equal volume (300 ml) of the source and stripping phase. The SLM with effective membrane area of 7.45 cm^2 was placed between the chambers. A new supporting membrane was used for each experiment. The silicon rubber seals were used to prevent any leakage from the chambers. Both compartments were electrically stirred at the equal and constant rate of 300 rpm with the aid of two motors located on the upper part on the cell. All experiments were carried out at 25°C ± 1°C. Several experiments were carried out to achieve the optimum conditions. To studying the influence of various parameters, all the other parameters were kept optimum except the one that was studied.

The permeability coefficient (P) was computed by using following equation [42]:

$$\ln\frac{\left[Cr\right]_t}{\left[Cr\right]_0} = -\frac{A}{V}Pt \qquad (1)$$

where $[Cr]_t$ and $[Cr]_0$ are the chromium concentrations in the feed phase at elapsed time and time zero, respectively; A is the effective membrane area; V is the volume of the feed phase; and t is the elapsed time.

An UV-Spectrophotometer (Vario 2600 Double Beam UV-Vis Scanning Spectrophotometer, England) was used to determine the amount of Cr(VI) that was carried out by monitoring chromium concentration at the source phase of a function of time. The pH of feed phase was measured by using a pH meter (pH900, Precisa Instruments AG, Dietikon, Switzerland).

3. Results and discussion

3.1. Influence of stripping phase concentration

The influence of the stripping phase (NaOH) concentration on Cr(VI) extraction was studied within the range of

0.05–0.2 M. Table 1 displayed the effect of stripping phase concentration on the overall permeation coefficient. Results showed that the permeation coefficient of the Cr(VI) enhanced by increasing the NaOH concentration. At the stripping solution of 0.1 M NaOH, the maximum permeation coefficient was achieved. This is probably due to the saturation of the driving force for diffusion because of an increase of metal–complexes concentration in the membrane–strip interface.

For higher stripping concentration than 0.1 M NaOH, the rate of extraction is almost constant. It is probably because of consumption of $HCrO_4^-$ ions and overpower of OH^- ions in stripping phase completely.

The effect of stripping phase concentration on Cr(VI) extraction is shown in Fig. 1. It can be observed that increasing the stripping concentration from 0.05 up to 0.1 M led to enhancing the Cr(VI) extraction. Further increase in stripping concentration had negligible influence on Cr(VI) extraction.

3.2. Influence of feed pH

To achieve the influence of feed phase pH on the extraction of Cr(VI), the experiments were carried out at different pH values ranging from 2 to 3.5. The overall permeation coefficient was calculated for each experiment, and the results are listed in Table 2. Furthermore, Effect of initial feed phase pH on Cr(VI) extraction is presented in Fig. 2.

It is evident that overall permeation coefficient decreases with increasing pH value of the aqueous feed solution. As we know, the difference between feed phase solution and

Table 1
Influence of the stripping phase concentration on the overall permeation coefficient

Stripping phase concentration (mol/L)	P (cm/s)
0.05	2.21×10^{-3}
0.1	2.82×10^{-3}
0.15	2.87×10^{-3}
0.2	2.89×10^{-3}

Feed phase = 250 ml of Cr(VI) solution; pH = 2; Aliquat 336 = 35% (V/V); Stirrer speed = 300 rpm.
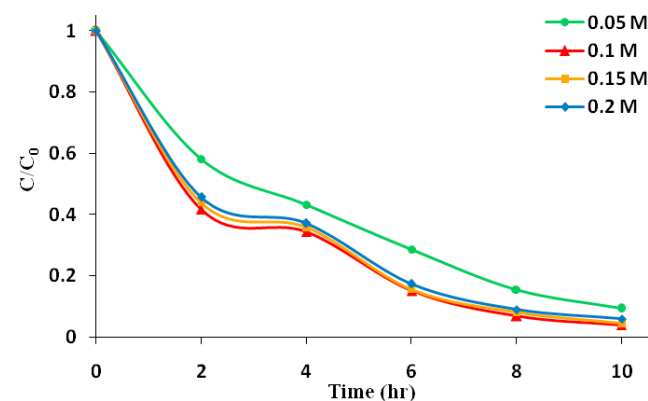

Fig. 1. Effect of stripping phase concentration on Cr(VI) extraction (Aliquat 336: 35% (v/v); Feed phase pH = 2; Feed concentration = 25 mg/l; Stirrer speed = 300 rpm).

Table 2
Influence of the feed phase pH on the overall permeation coefficient

Initial feed phase pH	P (cm/s)
2	2.82×10^{-3}
2.5	2.31×10^{-3}
3	1.64×10^{-3}
3.5	7.22×10^{-4}

Feed phase = 250 ml of Cr(VI) solution; Aliquat 336 = 35% (V/V); Stripping phase = 250 ml of 0.1 M NaOH solution, Stirrer speed = 300 rpm.
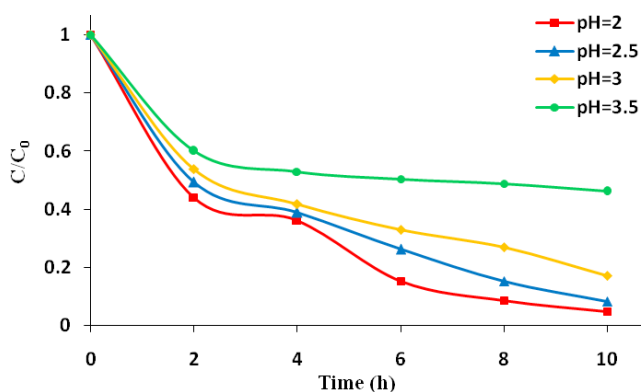

Fig. 2. Effect of initial feed phase pH on Cr(VI) extraction (Aliquat 336: 35% (v/v); Feed phase concentration = 25 mg/l; Stripping phase concentration = 0.1 M NaOH; Stirrer speed = 300 rpm).

stripping phase solution pH is the main driving force for SLM process. So decreasing in feed phase pH led to increase the driving force and increased the transportation rate. This happened because of the decreasing in Hydrogen ions concentration which led to decrease the rate of Cr(VI)–Carrier complexes formation.

3.3. Influence of carrier concentration

The effect of carrier concentration on Cr(VI) extraction was shown in the Fig. 3. For this purpose, A series of experiments was performed with various carrier concentrations (17%–50% v/v). Influence of the carrier concentration on the overall permeation coefficient is presented in Table 3.

It is obvious that the mass flux and permeability increase with increasing in Aliquat 336 concentration as a carrier in the liquid membrane. This manner was seen in the range of 17%–35% v/v. After reaching a maximum value at 35% v/v, the trend was changed and more increase in Aliquat 336 caused to reduction in overall permeation coefficient and Cr(VI) extraction. This is probably due to increasing in the viscosity of the organic phase, result in reduction of Cr(VI) ions transportation.

3.4. Influence of feed phase concentration

The influence of feed concentration on the Cr(VI) transport was investigated. Table 4 presented the permeation
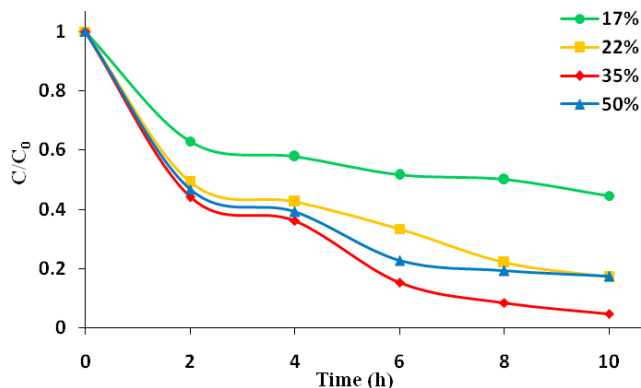

Fig. 3. Effect of carrier concentration on Cr(VI) extraction (Feed phase concentration = 25 g/l; Feed phase pH = 2; Stripping phase concentration = 0.1 M NaOH; Stirrer speed = 300 rpm).

Table 3
Influence of carrier concentration on the overall permeation coefficient

Carrier concentration (% V/V)	P (cm/s)
17	7.51×10^{-4}
22	1.65×10^{-3}
35	2.82×10^{-3}
50	1.63×10^{-3}

Feed phase volume = 250 ml of Cr(VI) solution; pH = 2; Stripping phase = 250 ml of 0.1 M NaOH solution; Stirrer speed = 300 rpm.

Table 4
Influence of the initial Cr(VI) concentration on the overall permeation coefficient

Cr(VI) concentration (mg/l)	P (cm/s)
25	2.82×10^{-3}
50	2.73×10^{-3}
75	6.16×10^{-4}
100	5.85×10^{-4}

Feed phase = 250 ml of Cr(VI) solution; pH = 2; Aliquat 336 = 35% (V/V); Stripping phase = 250 ml of 0.1 M NaOH solution, Stirrer speed = 300 rpm.

coefficient values as the concentration of Cr(VI) ranged from 25 to 100 mg.L^{-1} in the feed phase.

Fig. 4 showed the effect of initial feed phase concentration on the initial mass flux. The flux J is calculated by using following equation [43]:

$$J_0 = P\left[\mathrm{Cr}\left(\mathrm{VI}\right)\right]_0 \qquad (2)$$

where J_0 is the initial flux of mass transfer through the membrane, P is the overall permeation coefficient and $[\mathrm{Cr(VI)}]_0$ is chromium concentration in the initial feed phase.

Results indicated that initial mass flux enhanced with increasing the Cr(VI) concentration until tended to a constant value at higher Cr(VI) concentration. This manner is probably due to membrane saturation and lower effective membrane
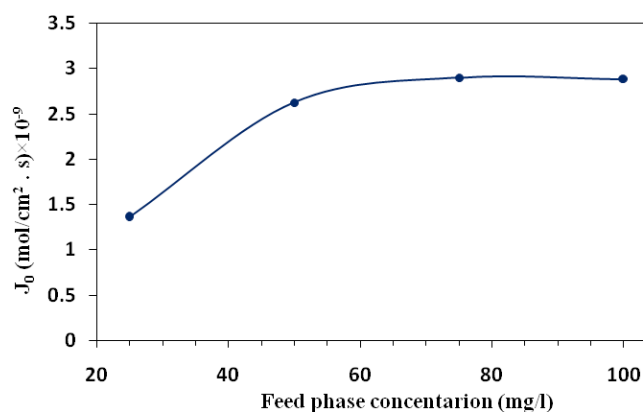
Fig. 4. Effect of initial Cr(VI) concentration on initial mass flux (Aliquat 336: 35% (v/v); Initial pH of feed phase = 2; stripping phase concentration = 0.1 M NaOH; stirrer speed = 300 rpm).
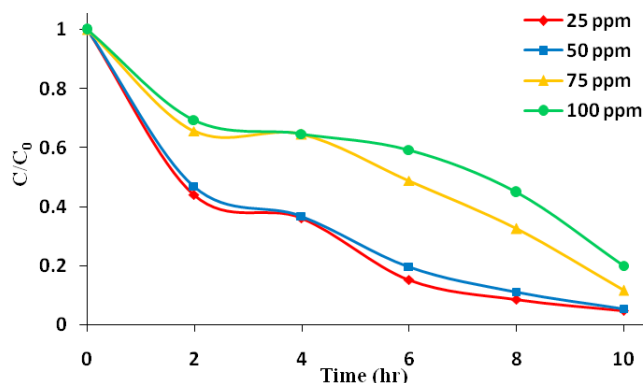

Fig. 5. Effect of initial feed phase concentration on Cr(VI) extraction (Aliquat 336: 35% (v/v); Initial pH of feed phase = 2; stripping phase concentration = 0.1 M NaOH; stirrer speed = 300 rpm).

area in the SLM and also due to maximization of membrane pores with Cr(VI)–Aliquat 336 species.

Fig. 5 exhibited the effect of initial feed phase concentration on Cr(VI) extraction. It was observed that the maximum efficiency of extraction occurred at the concentration of 25 ppm. It can be referred to the highest overall permeation coefficient for the concentration of 25 ppm.

According to mentioned results, the optimum conditions for the extraction of Cr(VI) ions was obtained and summarized in Table 5. At optimum conditions, 95.2% of Cr(VI) was extracted within 10 h through the SLM using sunflower oil as solvent.

3.5. Effect of solvent types and comparison with reported results

Comparative evaluation of different solvents on Cr(VI) extraction efficiency was shown in Table 6. It should be noted that the other investigators results obtained under different conditions. The only common circumstances of these works are the type of extractant (Aliquat 336) and the main process configuration (SLM). According to this table, the result obtained for Cr(VI) extraction in this study (95.2%) appears to be satisfactory. So, the feasibility of using sunflower oil as a green solvent in comparison with other organic solvents is validated.

Table 5
Optimum operating conditions

Parameter	Value
Stripping phase concentration	0. 1 M NaOH
Feed phase pH	2
Aliquat 336	35% (v/v)
Feed concentration	25 mg/l
Stirrer speed	300 rpm
Contact time	10 h

Table 6
The effect of solvent types on the Cr(VI) extraction efficiency

Solvent	Extraction efficiency (%)	References
Kerosene	43	[44]
n-octanol	71.3	[45]
Dodecane/Dodecanol	92.4	[46]
Tetrahydrofurane (THF)	93.2	[32]
Kerosene	94.6	[47]
Sunflower oil	95.2	This work

4. Conclusion

The present study affirmed that the Cr(VI) ions can be efficiently transported through the SLM by using Aliquat 336 dissolved in sunflower oil as a green carrier. Sunflower oil was employed in SLM for the Cr(VI) extraction for the first time. This transport influenced by several parameters, and some of them have been studied for this paper such as: feed phase concentration and pH, stripping phase concentration, and carrier concentration. The best conditions have been observed as follows: feed phase concentration = 25 mg L^{-1}, feed phase pH = 2, stripping phase (NaOH) concentration = 0.1 M, and carrier concentration = 35% v/v. The results indicate that Cr(VI) permeation is independent of carrier concentration in higher concentrations. The flux of transportation is a strong function of chromium concentration in feed phase. Decreasing in feed phase pH led to increase in transportation and the permeation coefficient of the Cr(VI) increase when the NaOH concentration increased and their values reached a maximum at 0.1 M NaOH concentration. At the optimum condition, the extraction efficiency of Cr(VI) was achieved about 95.2% in 10 h.

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