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Research Article

LIQUID -LIQUID EXTRACTION AND BLM STUDIES ON Cr (VI) SEPERATION USING (HTMABr) IN AMYL ALCOHOL AS THE EXTRACTANT

Mubeena K and Muthuraman G*

Presidency College (Autonomous) Department of Chemistry, Chennai-600 005 India

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ABSTRACT

Hexadecyl trimethyl ammonium bromide (HTMABr) was used as a carrier for the transport of Chromium (VI) ions through a isoamyl alcohol bulk liquid membrane. HTMABr - isoamyl alcohol (1:1 ratio) was found to be suitable liquid membrane for Cr (VI) transport. The membrane was kept in contact with acidic feed phase and alkaline strip phase. The transport efficiency increased with increasing carrier concentration from 1.0 x 10^{-2} mol/L to 6 x10^{-2} mol/L. The following parameters such as the effect of pH of the so((urce phase, the nature of stripping agents in the receiving phase, ($NaNO_3$ solution), concentration of HTAB in membrane, rate of stirring, effect of transport time, type of solvent, effect of temperature, then Cr (VI) concentration in feed phase were examined. Maximum transport efficiency was observed for Cr (VI) ion when it was present in the concentration of 10 mg/L. At high stirring speed (300rpm) the Cr (VI) transport from the feed phase to the strip phase was completed within 5 h at 27°C.

INTRODUCTION

Cr (VI) is highly toxic and a potential carcinogenic agent due to its high oxidation potential and relatively small size which enables to penetrate through biological cell membranes. The landfills of chromium may thus be considered as a potential hazard to the environment (Sahu *et al*., 2009; Barbara wionczyk *et al*., 2006). It toxicity initially produces immediate cardiovascular shock which later effects kidney, liver, blood-forming organs and cancer etc (Good game *et al*., 1986; Singh *et al*., 1986 ; Srivastava *et al*.,, 2006). Varies methods have been developed for the removal of Cr (VI) from industrial effluents it includes solvent extraction (Lo and Shire 1998; Benjjar *et al*., 2012; Mpinga *et al*., 2014), non- dispersive solvent extraction (Oritiz *et al*; 1996; Alons *et al*., 1998), chemical precipitation, ion exchange, adsorption etc., The conventional and the most commonly method used for Cr (VI) removal is chemical precipitation (Venkateswaran and Palanivelu, 2005). In recent years, much attention has been focused on separation techniques such as liquid-liquid extraction, liquid membrane etc. the advantages of LLE include high through put ,ease of automatic operation and of scale up and high purification (Yang and Pane, 2003).

Liquid membrane (LM) systems are being studied extensively by researchers in such fields as analytical, inorganic and organic chemistry, chemical engineering, biotechnology and biomedical engineering and wastewater treatment etc. (Elumalai and Muthuraman, 2013). It have been successfully used to treat aqueous solutions contaminated with heavy metal ions such as copper, zinc, cadmium, nickel, mercury, lead, silver, palladium, and gold (Fakhari *et al*., 206; parthi *et al*., 2009; Zaghbani *et al*., 2007). BLM consist of three phases: two aqueous phases as a donor or an acceptor and one organic phase in which a carrier is dissolved the transport from an aqueous donor phase through an organic membrane phase to an aqueous acceptor phase occurs. Such experimental rig on a laboratory scale to evaluate new means of improving the efficiency of separation processes is suggested in ref (Izatt *et al*., 1985).

The membrane separation method is of interest as it has several advantages over other separation methods like low capital costs, space requirements, energy consumptions (Jafari *et al*., 2009) and low hydration energy. The main advantage of liquid membrane transport of metal ions over their classical solvent extraction method is that the amounts of organic solvent and metal ion carrier are remarkably reduced (Araki *et al*., 1990). BLM has been studied for pre-concentration and separation of different metal ions with a variety of liquid membrane phase (Yamini *et al*., 2001; Deminl *et al*., 1992; Lamb *et al* 1980; Weidong Zhang *et al*., 2009). In such process the selection of a suitable carrier plays a crucial role in order to selective extraction of a desired substance at the membrane inter face.

*Corresponding author: **Muthuraman G**
Presidency College (Autonomous) Department of Chemistry, Chennai-600 005 India

The transport of other metal ions such as zinc, mercury, silver, palladium and gold in the anionic from of the complexes has also been reported (Izatt *et al*., 1987; Parham *et al*., 1994; Akhond *et al*., 1996; Chiarizia and castagnota, 1983). The Cr(VI) transport has been dealed with different liquid membrane configurations in different kinds of carriers. The choosing of the carrier also important in metal separation. Various carriers used for Cr(VI) transport such as Alamine 336, Tri-n-butyl phosphate, Aliquat 336, cyanex 921 and cyanex 923 as carriers. In our present study the liquid membrane solution contains HTMABr and amyl alcohol which is hydrophobic in nature.

HTMABr was one of the most widely used cationic ion and effective antiseptic agent against bacteria and fungi (Rosen, 1989). Thus widely used in the synthesis of gold nano particles (Rayavarapu *et al*., 2010) and in many house hold products. HTMABr has been showed to be an effective carriers for the separation and purification of a metal due to its low cost, excellent chemical stability, low stability in the aqueous phase etc. (Zhang Weidong *et al*., 2007) it is freely soluble in alcohol so, it was chosen as a carrier and there is no available information in the literature on the study of Cr(VI) transport through BLM using HTMABr as carrier.

In this work transport of Cr (VI) through a BLM contain HTMABr as mobile carrier in isoamyl alcohol has been investigated. The following parameters have been investigated in different experimental conditions such as the effect of HTMABr concentration, effect of stripping agent, effect of equilibrium time, effect of pH etc.

MATERIALS AND METHODS

Materials and reagents

The entire chemical used were of analytical grade reagents. Cr (VI) solution (feed phase) was prepared by dissolving a weighed amount of $K_2Cr_2O_7$ in a known volume of deionized water. The following inorganic salts, acid and organic solvents were used in the experiments without further purification. HCl (35.4%) HTMABr (99.0%) amyl alcohol ≥98%, NaCl (99.5%) H_2SO_4 (98%) sodium nitrate (99%) Di-phenyl carbazide (98%) Acetone (99%) Iso amyl alcohol (≥98%) was obtained from Merck.

Instrumentation

The absorbance of Cr (VI) concentration in the aqueous phase was determined with uv visible spectra photo meter (Elico SL 159) and the absorption wavelength was 540nm using 1, 5 biphenylcarbazide as the indicator. The pH adjustment of source phase was done using Elico. For agitation of solution a shaker and stirrer was used (IKD-KS 50, India). Systronics Electrophoresis 606 was used to find out whether the metal is cationic or anionic.

Preparation of chromium solution

The Cr (VI) solutions (feed phase) were prepared by dissolving a weighed amount of $K_2Cr_2O_7$ in a known volume of deionized water. The pH was adjusted to the desired value by adding HCl. The maximum adsorption wavelength was 540 nm using,1, 5 - diphenylcarbazide as the indicator. The working solutions were prepared by diluting the stock solution with deionized water to give the appropriate concentration of the working solutions

Procedure:-LLE

Liquid- liquid extraction of metal the organic solvent amyl alcohol used for the extraction was added to the prepared aqueous Cr (VI) solution and the extraction experiment was carried out at 27±0.1°C. The pH was adjusted by using 0.5N HCl acid. The feed phase of Cr (VI) (25mg/L) and organic phase (25ml) was transferred into a separating funnel both the phases were immiscible. The separating funnel was shaken well. The wave length of maximum absorption (λ_{max}) for Cr(VI) was 540nm. The percentage of extraction and stripping Cr (VI) was calculated as the following equations.

$E = 1-(metal)_{aq}/ (metal)_{aq}o \times 100$ ----------(1)

$(metal)_{aq}$ = metal concentration in the aqueous phase (mg/l)
$(metal)_{aq}o$= initial metal concentration in the aqueous phase.
E= metal extraction percentage.
In stripping the organic metal V=25 ml and stripping solution sodium nitrate ($NaNO_3$) V=25ml well added to separating funnel and shake well, the aqueous. strip was taken for UV.

$R = (feed)_s/(feed)_{aq}0 \times 100$ ---------(2)

R-Percentage of stripping
$[feed]_s$-concentration of stripping feed phase
$[feed]_{aq0}$ =initial concentration of feed phase.

BLM

The experimental studies were carried out applying the BLM technique using a stirred transfer lewis type cell with bulk liquid membrane layered over the feed and product phase shown in Fig.1.

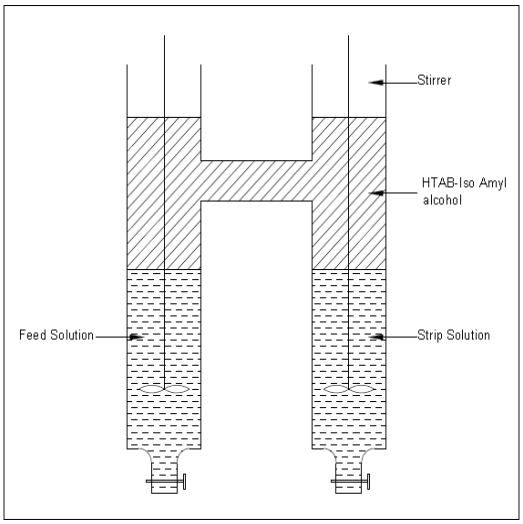
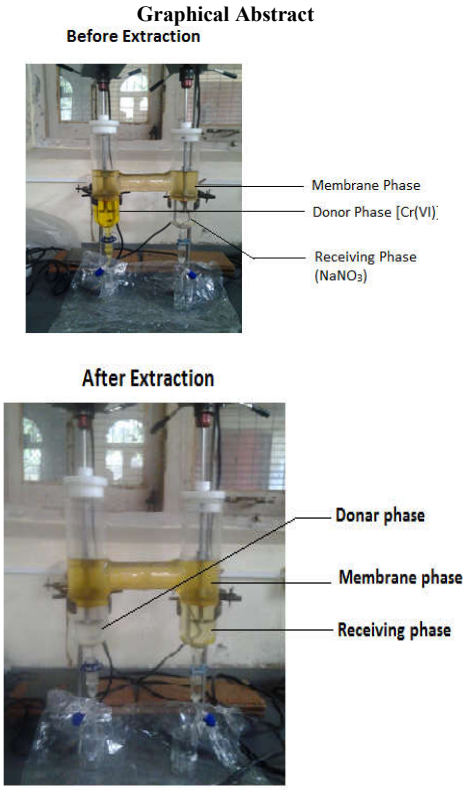


Figure 1 Schematic experimental setup for bulk liquid membrane for transportof Cr (VI) from aqueous phase

The inner dimension of transport cell is 70 mm diameter × 195 mm depth for H-type. The size of the equipment is 120mm × 30mm × 40mm (length × width × height). The aqueous feed phase containing (25mg/L, 260 mol/L) chromium solution and strip phase containing (0.5N, 260 mol/L) Sodium nitrate were taken in the BLM apparatus and the solutions stirred by mechanical stirrers at 300 rpm.



These two layers were separated by the organic solvent such as isoamyl alcohol which acts as liquid membrane phase. Experiment samples were taken out from the feed and strip phases at known time, and the Cr (VI) concentration was measured using spectrophotometer (λ max = 540nm). All experiments performed at 27^0C ± 0.1.

RESULT AND DISCUSSION

Liquid liquid extraction experiments

Effect of pH of feed phase

Extraction of anionic metal Cr (VI)) from the aqueous solutions was studied in 6×10^{-2} mol/L HTMABr at 1:1 aqueous to organic phase ratio with the initial pH ran from 1 to 14. The results are shown in Fig.2 the pH was adjusted using 0.5N HCl.

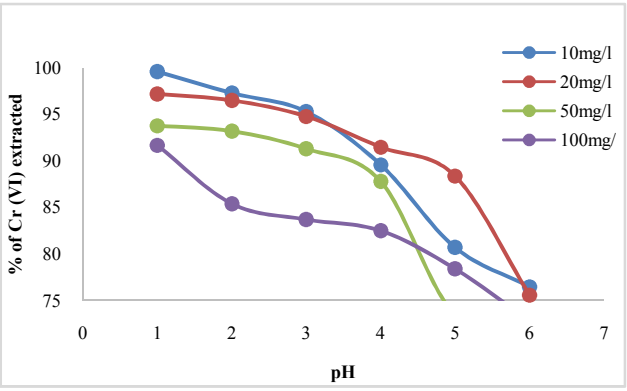


Figure 2 Effect of pH Effect of pH on Cr(VI) transport through the bulk liquid membrane.(Experimental condition : donor phase 25 $x10^{-3}$ g/l $K_2Cr_2O_7$ solution of pH 1.0 ± 0.1. acceptor phase 2.0 x 10^{-2}mol/l $NaNO_3$ solution membrane phase 6.8 x10^{-2}mol/l carrier in Isoamyl alcohol,300 rpm)

The extraction efficiency of the Cr (VI) decreased with increasing pH of feed phase, because at low pH the H concentration was much higher. It was interacted with metal and it enhances the combination of Cr (VI) with iniso amyl alcohol. The maximum extraction efficiency of Cr(VI) obtained were as follows, 99% for 10 mg/L, 98.6% for 20mg/L, 95.7% for 30mg/L,92.8% for 40mg/L, 82.4% for 50mg/L. Further increases pH (8to14) the extraction efficiency was not found, because in alkaline pH, extractant gets negative change and Cr (VI) also negative. So alkaline medium repulsion was takes place between the extractant and the base. The maximum chromate ions transport occurred at pH 1.0 (Muthuraman *et al*., 2009). Thus a pH of 1.0 ± 0.1 was pH of feed selected for further studies.

Effect of HTMABr concentration

Chromate is anionic form so, it requires a cationic carrier. Hexadecyltrimethyl ammonium bromide act as cationic carrier so experiments were run act different carrier concentrations from 1.0×10^{-2} to 6×10^{-2}mol/L Fig.3 shows that chromate ion increases with an increase in the carrier concentration from 0.01M to 0.06 M at pH 1.0 ± 0.1.The highest percentage value obtained in 0.06M. At a lower carrier concentration the interface between the donor solution and the membrane is not saturated by the carrier (Ahmet Saf *et al*., 2006). The Cr (VI) concentration increases with an increase in carrier concentration. No extraction was takes place in the absence of HTMABr carrier. Hence 0.06 M of HTAB was used for further studies.

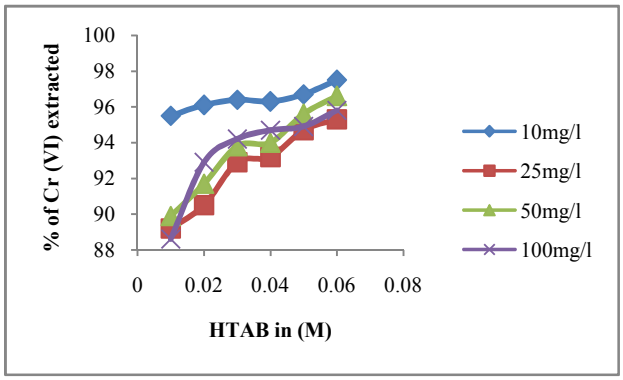


Figure 3 Effect of HTMABr concentration Effect of carrier concentration on the feed phase (Experimental condition : donor phase 25 $x10^{-3}$ g/l $K_2Cr_2O_7$ solution of pH 1.0 ± 0.1. acceptor phase 2.0 x 10^{-2}mol/l $NaNO_3$ solution membrane phase different molar concentration of HTMABr)

Effect of stripping agent

The type of reagents used in the receiving phase is important parameters for influencing the transport efficiencies. Various aromatic and aliphatic stripping reagents such as benzoic acid, oxalic acid, salicyclic acid acetic acid, sodium salicylate, ammonium salicylate and sodium Nitrate have been used.. Among them $NaNO_3$ was found to be suitable stripping agent. So its concentration was varied from 0.2% to 2.0 M. It can be seen from Table 1; maximum stripping efficiency was formed in 0.2M $NaNO_3$ solutions. Further increases in $NaNO_3$ concentration did not improve the efficiency of stripping. Hence 0.2M $NaNO_3$solution was chosen for further studies.

Table 1 Effect of stripping agent

Stripping concentration(mol/L)	Percentage of Cr (VI) extraction
0.2	91.1
0.5	89.3
1.0	83.5
1.5	81.6
2.0	80.4
2.5	80.1

Effect of diluents

The extraction was carried out in different diluents such as benzene, hexane, xylene, toluene, chloroform, carbon tetra chloride, dichloromethane, and Iso amyl alcohol. Among them high extraction percentage of Cr (VI) was obtained only in the the combination of Iso amyl alcohol-HTMABr combination. About 99% of Cr (VI) was extracted in Iso amyl alcohol-HTMABr. So, further studies were carried out using Isoamyl alcohol as diluents. This resultwere presented in Table.2

Table 2 Effect of diluents

Diluents	Percentage of Cr (VI) extraction				
	10mg/L	20mg/L	30mg/L	40mg/L	50mg/L
Benzene	-	-	-	-	-
Hexane	50.1	47.8	44.3	41.7	40.4
Carbon tetra chloride	45.2	43.6	42.9	39.8	39.3
Dichloro methane	32.5	31.3	30.8	29.8	28.7
Iso amyl alcohol	99.0	98.2	97.4	95.6	93.7

Bulk liquid membrane experiments

Effect of pH of the donar phase

In order to investigate the effect of pH parameter the transport experiments of Cr (VI) from acidic medium through Isoamyl alcohol membrane containing HTAMBr and the pH of the feed phase in the range of 1 to 6 were carried out. Fig.4 The maximum amount chromate ions transport occurred at pH 1.0± 0.1 (Venkateswaran and Palanivelu, 2005). In low pH the extraction efficiency of Cr (VI) was very high. The percentage of chromium transported was about 99.6%. Thus, the further experiment to study the other parameters was carried out at pH 1.0 ± 0.1. The maximum Cr (VI) transport occurred at pH 1.

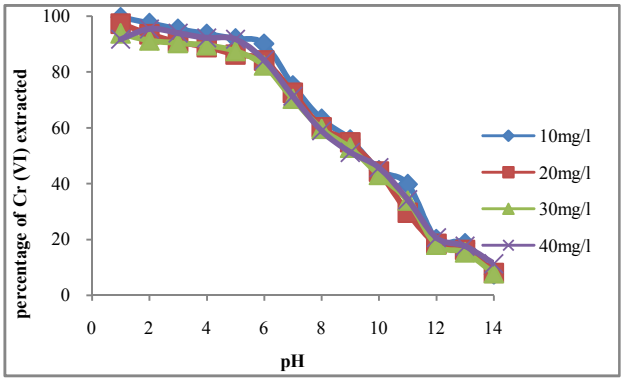


Figure 4 Effect of pH of the donar phase Effect of pH on Cr(VI) transport through the bulk liquid membrane. (Experimental condition : donor phase 25 x10^{-3} g/l $K_2Cr_2O_7$ solution of pH 1.0 ± 0.1. acceptor phase 2.0 x 10^{-2}mol/l $NaNO_3$ solution membrane phase 6.8 x10^{-2}mol/l carrier in Isoamyl alcohol, stirring speed=300 rpm,Rd,Ra and Rm represent the Cr (VI) concentration in donar, membrane and accepter phase)

Effect of stirring

The effect of stirring speed in the source phase and received phase was studied in the range 200-500 rpm in order to obtain optimal stirring speed that allows effective transport of Cr (VI) in the BLM system. Increase in transport was observed when the stirring speed was increased from 200-300 rpm and beyond which no appreciable increase in Cr (VI) transport was observed up to 400 rpm. At a stirring speed of 200-300 rpm the stripping efficiency increased. Further increased the rpm the stripping efficiency decreased. This represented that the aqueous boundary layer thickness diminished continuously with increase stirring speed and the boundary layer was maximum in the range of 200-300 rpm was constant. Further a decrease in transport from above 300 rpm could be due to high turbulence caused by stirring, and displacement of carrier from

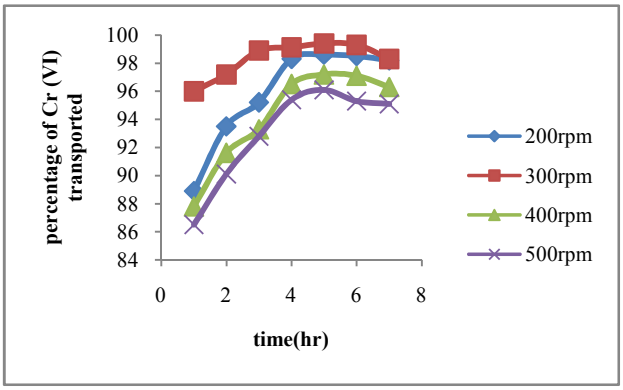


Figure 5 Effect of stirring: Effect of stirring speed of Cr(VI) (Experimental condition : donor phase 25 x10^{-3} g/l $K_2Cr_2O_7$ solution of pH 1.0 ± 0.1. acceptor phase 2.0 x 10^{-2}mol/l $NaNO_3$ solution membrane phase 6.8 x10^{-2}mol/l carrier in Isoamyl alcohol,300 rpm)

membrane pore also noticed (Venkateswaran and Palanivelu., 2005) Thus a 300 rpm stirring speed was maintained through the subsequent investigations these results were presented in Fig.5)

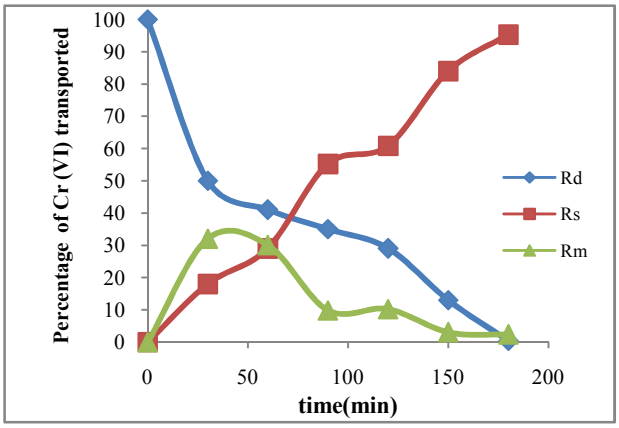


Figure 6 Effect of time Effect of time on Cr(VI) transport (Experimental condition : donor phase 25 x10^{-3} g/l $K_2Cr_2O_7$ solution of pH 1.0 ± 0.1. acceptor phase 2.0 x 10^{-2}mol/l $NaNO_3$ solution membrane phase 750ml for 6.8 x10^{-2}mol/l carrier in Iso amyl alcohol,300 rpm and transport time 180 min.)

Effect of time

Fig.6 shows the time dependence of Cr (VI) transport through the liquid membrane under experimental conditions. It is

obvious that the extraction of chromium ion from the source phase in to the organic membrane occurs almost completely after within 1 hour. However, the plot shows the transport rate decreases gradually with time and a steady state situation in reached in which the amount of Cr (VI) ions. 96.6% of Cr (VI) concentration was transported the time 1 hour. However, 97.13% of Cr (VI) transported in 180 min. Hence the time 180 min recommended for further experiments

Suggested mechanism

Based on the results obtained, the following mechanism was suggested for the transport of Cr (VI) across a BLM was illustrated in Fig.7. As shown fig.$HCrO_4^-$, H^+ react with A^+ Br- at the liquid membrane-feed interface resulting in the formation of the metal complexes. Then, the complexes diffuse through the liquid membrane phase to the liquid membrane-stripping interface and release Cr (VI) in to the stripping phase. The carrier act as a shuttle. The transport mechanism was a coupled-co-transport (symport) mechanism. The transport mechanism of Cr (VI) can be explained as follows.

Suggested mechanism:

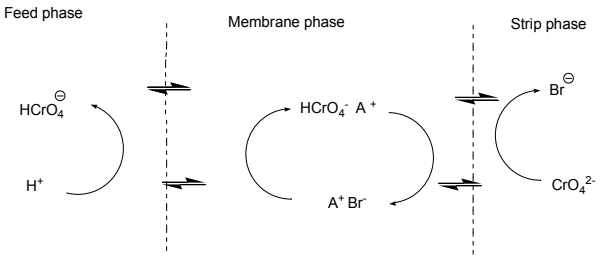


Fig 7

Table 3 Characteristics of chrome plating wastewater

Serial number	Parameters	Chrome Plating wastewater
1	pH	3.5
2	Conductivity(μS/cm)	2900
3	Total dissolved solids(mg/L)	1850
4	Total hardness(mg/L)	450
5	Calcium(mg/L)	110
6	Magnesium(mg/L)	07
7	Chloride(mg/L/)	450
8	Sulphate (mg/L)	600
9	Cr (VI)(mg/L)	11
10	Nickel(mg/L)	0.01
11	Iron (mg/L)	0.9
12	Manganese (mg/L)	0.9
13	Copper (mg/L)	10.0

Application of the developed BLM for textile waste water

The developed BLM was tested for real textile wastewater collected from a local factory. Under optimized condition (feed phase 260ml of 25mg/L Cr (VI), receiving phase =260ml of 0.2M $NaNO_3$ solution, membrane phase=260ml of diluent with HTMABr, equilibrium time=5 hour, stirring speed=300rpm) the industrial waste water was extracted, and the extracted Cr (VI) was successfully stripped into sodium nitrate solution. The extraction efficiency of Cr (VI) was not much affected by inorganic salts the extraction of Cr (VI) was found to be 99 % and extracted Cr (VI) stripping in to 0.02 M of $NaNO_3$ solution

CONCLUSIONS

In the liquid-liquid extraction and bulk liquid membrane method deals for selective extraction of Cr(VI). The efficiency of the method depends on various parameters such as the pH of the donar and acceptor phase, effect of carrier concentration, stirring speed, effect of solvents. The maximum extraction efficiency was obtained at pH 1.0 ± 0.1.The extracted Cr (VI) was successfully stripped into $NaNO_3$ solution from loaded organic phase. In optimized condition after 5 hours the transport of chromium ions was 99.13%. The proposed method was applied to industrial waste water and found to be satisfactory.

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