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Extraction and stripping of Cr(VI) from aqueous solution by solvent extraction

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ABSTRACT

The solvent extraction of Cr(VI) from an aqueous solution by hexadecyl trimethyl ammonium bromide (HTAB) in amyl alcohol was investigated at 27˚C. The maximum extraction efficiency was obtained at pH 1.0 ± 0.1. Chromate is an anionic form; therefore, it requires a cationic carrier for the extraction purpose and HTAB acts as cationic carrier in this study. The efficiency of extraction decreased with increasing the chromium concentration. The extracted Cr(VI) in the organic phase can be back extracted into 0.2 mol/L sodium nitrate. The important experimental parameters examined were: the effect of HTAB concentration, the effect of pH, the effect of Cr(VI) concentration, the effect of equilibrium time, and the effect of temperature, aqueous to organic phase ratio, and various stripping agents. Real effluent was also studied under optimized condition [Cr(VI) concentration 10 mg/L at pH 1.0 ± 0.1, carrier concentration = 0.06 mol/L, stripping concentration = 0.2 mol/L, and equilibrium time = 5 min at 27˚C] and satisfactory result has been found.

Keywords: Extraction; HTAB; Stripping; Chromium(VI); Amyl alcohol

1. Introduction

The heavy metal chromium in the natural environment is found in Cr(III) and Cr(VI) forms. Cr(VI) is known to be 500 times more toxic, mutagenic, and carcinogenic than Cr(III) [1]. Chromium is used in many industries such as leather tanning, metallurgy, electroplating, and textile manufacturing [2–5]. Majority of Cr(VI) originates from industrial sources [6] due to its high oxidation potential and relatively small size, which enable it to penetrate through biological cell membranes. Its toxicity can lead to cancer, kidney and liver damages, nausea, vomiting, diarrhea, and hemorrhage [7–9]. Hence, it is considered as a potential hazard to the environment [10]. Therefore, it is necessary to eliminate Cr(VI) from the environment, in order to prevent the deleterious impact on ecosystem and public health. Chromium cannot be eliminated by ordinary treatment process because of the stricter environmental regulations; therefore, a cost-effective alternate technology for the treatment of Cr(VI)-contaminated wastewater is highly desired by the industries [11].

Preliminary studies on the treatment of leather manufacture wastewater by solvent extraction have been performed by Pandey et al. [12]. Several

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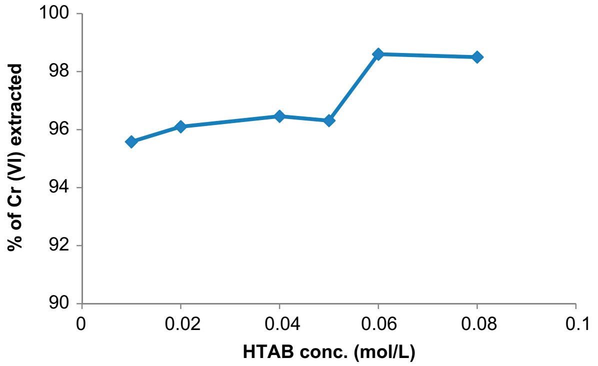


Fig. 1. Effect of temperature (Experimental conditions: Volume of feed phase = 25 mL at pH 1.0 ± 0.1, volume of organic phase = 25 mL, A/O ratio = 1:1, extractant concentration = 6.8×10^{-2} M, chromium concentration = 10 mg/L, and equilibration time = 5 min).

techniques have also been developed to remove Cr(VI) from industrial effluents. Different extraction methods have been studied for the recovery of Cr(VI) from aqueous solution such as solvent extraction [13–16], non-dispersive solvent extraction [17,18], and membrane-based techniques. The recovery and separation of Cr(VI) from aqueous solution by solvent extraction have been reported in the literature [19–23]. In recent years, much attention has been focused on a solvent extraction or liquid–liquid extraction (LLE). Another common method used to remove Cr(VI) is chemical precipitation, where Cr(VI) is first reduced to Cr(III). However, this process consumes a large amount of reducing agents.

The advantage of solvent extraction includes high throughput, ease of automatic operation, and high purification [24,25]. Different parameters affect the extraction of chromium ions in solvent extraction. Cussler et al. [26] observed that the extraction efficiency of Cr(VI) decreased at low carrier concentration, aqueous to organic phase ratio, salt concentration, nature of solvent, and some of the interference mechanisms that affect the extraction efficiency of hexavalent chromium [27–30]. Hexadecyl trimethyl ammonium bromide (HTAB) is one of the extractants to be used industrially and commercially because of its low cost, excellent chemical stability, good extraction kinetics, and low stability in the aqueous phase [31,32]. Hence, it was chosen as a carrier. In the present work, extraction of Cr(VI) through LLE containing HTAB as a carrier was studied. Cr(VI) makes an ion pair with carrier in a sequence of polar solvent amyl alcohol as extractant and also some other influencing parameters, such as the effect of carrier concentration, the effect of stripping agent, the effect of salt and stripping agent concentration were investigated.

2. Experimental

2.1. Reagents

The following inorganic salts, acids, and organic solvents were used in the experiments without further purification: $K_2Cr_2O_7$ (99.9%), NaOH (97.0%), HCl (35.4%), HTAB (99.0%), amyl alcohol (≥98%), NaCl (99.5%), H_2SO_4 (98%), sodium nitrate (99%), and di-phenyl carbazide (98%). Deionized water was used for preparing all the aqueous solutions.

2.2. Instrumentation

pH measurements during aqueous phase were performed using a combined glass electrode Elico Li 120 pH meter. Chromium absorbance measurements were performed with UV–visible spectrophotometer (Elico Sl 159, India). For agitation of solution, a shaker was used (IKD-KS 50, India).

2.3. Extraction of Cr

Organic solvent [amyl alcohol + HTAB (0.06 M)] used for extraction was added to the prepared aqueous Cr(VI) solution (10 mg/L) in a glass-stoppered bottle. The pH of the aqueous solution was adjusted using 0.5 N HCl. The glass-stoppered bottle was shaken at 50 rpm for 5 min in a shaker. The solution mixture was then transferred into a separating funnel. A sample of aqueous solution of the separating funnel was taken for the absorbance measurement of Cr(VI). The wavelength of maximum chromium absorption (λ_{max}) for chromium was 540 nm, using 1,5 diphenylcarbazide as the indicator [31].

The Cr(VI) concentration in the organic phase was calculated on the basis of a mass balance. As K2Cr2O7 was of analytical grade, so it was used without further purification. A stock solution of 1,000 mg/L was prepared by dissolving an appropriate quantity of K2Cr2O7 in a liter of deionized water. The working solution was prepared by diluting the stock solution with deionized water to give the appropriate concentration of the working solution.

3. Results and discussion

3.1. Effect of pH in the aqueous phase

The effect of pH on the extraction of Cr(VI) was investigated at different values of pH in the aqueous phase ranging from 1 to 6 at 27˚C. The result shows that the extraction efficiency decreased with increasing pH. The result shows that the extraction efficiency decreased with increasing pH. The

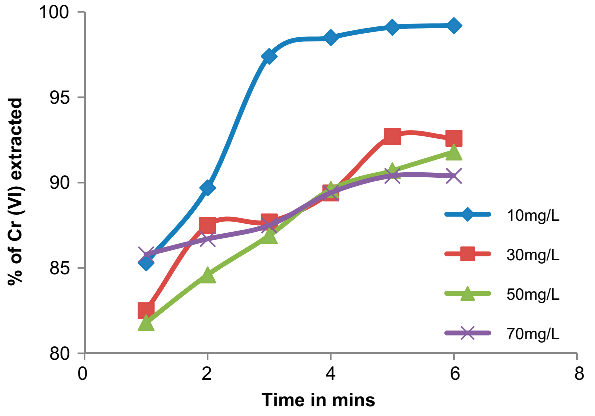
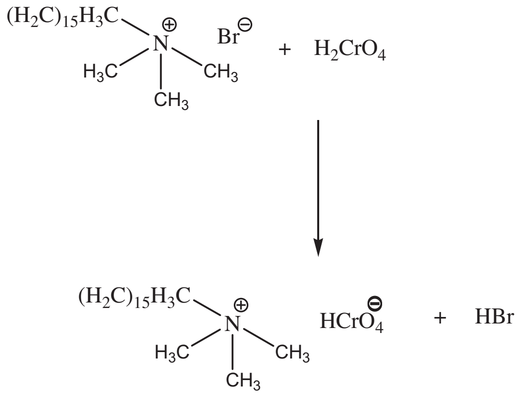


Fig. 2. Effect of equilibrium time (Experimental conditions: Volume of feed phase = 25 mL at pH 1.0 ± 0.1, volume of organic phase = 25 mL, A/O ratio = 1:1, extractant concentration = 6.8×10^{-2} M, and chromium concentration = 10 mg/L).

maximum chromate ions are extracted from aqueous to organic phase at pH 1.0 ± 0.1 [33], it might be at low pH, the H+ ion concentration was much higher. It interacts with metal and enhances the combination of Cr with HTAB in amyl alcohol. Therefore, the H^+ ion concentration decreased with increasing pH which in turn decreases the extraction of Cr from aqueous solutions. Hence, the extraction of Cr(VI) ions was maximum at pH 1.0 ± 0.1 [3]. These results agreed with our findings. Thus a pH of 1.0 ± 0.1 was selected for further experiments. The extraction mechanism is shown in Eq. (1).



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3.2. Effect of HTAB concentration

HTAB is a cationic surfactant and it is used as a carrier (molecular formula C19H42BrN, molecular weight-364.5). It is a solid, so no density occurred. It is freely soluble in water and alcohol but insoluble in ether and benzene. The experiment was carried out with different HTAB carrier concentrations ranging from 0.01 to 0.08 mol/L, using amyl alcohol. Chromate ion transport increased with increase in carrier concentration At a lower concentration, the interface between the organic phase and aqueous phase is not saturated by the carrier. Fig. 1 shows that the efficiency of chromium extraction increased with increasing HTAB concentration. The percentage of Cr(VI) extraction (99%) was obtained using 0.06 mol/L HTAB concentration. Further increase (beyond 0.06 mol/L) in extractant concentration did not show considerable effect on extraction efficiency. A blank experiment was performed in which the solvent contains no carrier and it was observed that extraction took place. This confirms that HTAB was required for the extraction of anionic chromium from source phase. Hence, in the succeeding test the extractant concentration was fixed at 0.06 mol/L. Under the optimized condition (i.e.), in acidic solutions (pH 1.0) with low concentrations of Cr(VI), $HCrO_4^-$ anion dominates in the aqueous phase.

3.3. Effect of diluents

The extraction of Cr(VI) from aqueous phase was carried out in different solvents dissolved in HTAB as carrier viz. benzene, hexane, xylene, toluene, chloroform, carbon tetrachloride, dichloromethane, and amyl alcohol from aqueous solution at pH 1.0 ± 0.1. There was no extraction in benzene and toluene because of low dielectric constant and in xylene the percentage of extraction (5%) is very low. Dichloromethane, chloroform, carbon tetrachloride, and amyl alcohol having high dielectric constant show high extraction of anionic chromium ions. The maximum extraction efficiency was noticed as follows: 99.0% for amyl alcohol, 70% for dichloromethane, 50% for hexane, 45.2% for carbon tetrachloride, and 42.7% for chloroform at 10 mg/L. As the extraction efficiency was very good in amyl alcohol due to its high polarizability ($\varepsilon = 18.23$) it interacted highly with HTAB, and hence the solubility increases. Table 1 shows the extraction efficiency of different diluents.

3.4. Effect of Cr concentration

The extraction efficiency of various initial concentrations of anionic Cr in the aqueous phase (10 mg/L, source phase) was determined at pH 1.0 ± 0.1, contacted with organic layer (25 mL) containing 0.06 mol/L of HTAB in amyl alcohol. At the initial Cr concentration of 10 mg/L, 99% extraction was obtained. When the concentration of Cr was increased

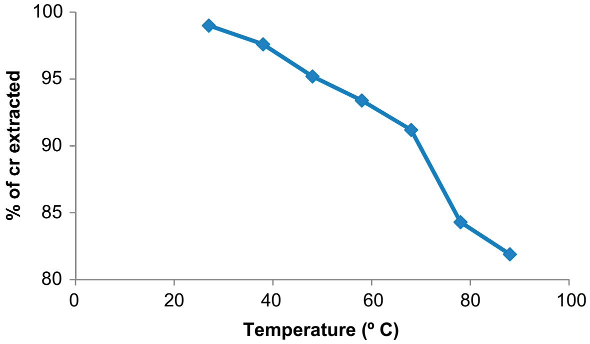


Fig. 3. Effect of stripping reagent (Experimental condition: Volume of source phase = 25 ml, volume of organic phase = 25 ml, and extractant concentration = 6.8×10^{-2} mol/L).

further, the percentage extraction of Cr(VI) ions decreased. The aqueous phase (Raffinate) was analyzed for chromium(VI) concentration and the percentage of Cr(VI) transferred into organic phase was calculated. At higher initial chromium(VI) concentration in the aqueous phase, the extraction efficiency decreased. It can be explained that at higher Cr(VI) concentrations, Cr has high color and higher energy, so HTAB is not able to completely remove chromium (VI) in aqueous phase. However, the absolute amount of Cr extracted increased with increase of initial Cr(VI) concentration [33–35]. The results are shown in Table 2.

3.5. Effect of equilibrium time

The effect of equilibrium time at different time intervals and different Cr(VI) concentrations was studied for 1–6 min. The extraction efficiency of Cr(VI) increased with increasing equilibrium time. The maximum percentage of extraction (99%) at initial Cr(VI) concentration of 10 mg/L was achieved in 5 min. The extraction efficiency did not increase with increased equilibrium time after 5 min. Beyond 5 min, the extraction efficiency remained unchanged. The results are shown in Fig. 2. Hence, an equilibrium time of 5 min was recommended for further studies.

3.6. Effect of salt concentration

In the actual industrial bath effluent, Cr(VI) contains salts such as NaCl and sodium sulfate. To understand the influence of sulfate and chloride concentration on Cr(VI) extraction, Cr(VI) solutions with different concentrations of sodium chloride and sodium sulfate were prepared and tested at pH 1.0 ± 0.1. The results are presented in Table 3, which shows the effect of sodium chloride and sodium sulfate on percentage removal of Cr(VI) from aqueous solution, and it can be seen that the percentage of extraction decreased slightly with increasing Na_2SO_4 and NaCl concentrations. This might be due to competitive extraction between Cr(VI) and Cl^-, SO_4^{2-} with HTAB. Hence, Cr(VI) extraction slightly decreased in NaCl and Na_2SO_4 from 98.8 to 97.4% and 99 to 97% at pH 1.0 ± 0.1, respectively, when Na_2SO_4 and NaCl were present in Cr(VI) solutions.

3.7. Effect of temperature

Fig. 3 shows the effect of temperature on the extraction of Cr(VI) from aqueous phase. The temperature was varied from 27 to 88˚C at pH 1.0 ± 0.1. Maximum extraction of 99% for 27˚C, 97.6% for 38˚C, 95.1% for 48˚C, 93.4% for 58˚C, 91.2% for 68˚C, 84.3% for 78˚C, and 81.95 for 88˚C occurred at 10 mg/L initial chromium(VI) concentration. When temperature increased, the physical bonding between the organic compounds and the extractant weakened. As, the solubility of chromium(VI) increased with increase in temperature, chromium(VI) was more difficult to extract. At higher temperatures, the solvent would evaporate; hence, the effect of temperature on extraction was not very significant at room temperature. Further studies were carried out at 27˚C significant at room temperature for the extraction of Cr(VI) from aqueous solutions. Hence, further studies were carried out at 27˚C.

3.8. Effect of aqueous to organic ratio (A/O)

The phase volume ratio (aqueous to organic phase volume) of 1:1 to 5:1 was used to study the effect of phase ratio on extraction with initial Cr(VI) concentration as 10 mg/L. The results are presented in Fig. 4. About 99% extraction efficiency was achieved in 1:1 (A/O) ratio. When the ratio (A/O) was increased from 2:1 to 5:1, the extraction efficiency dropped from 99 to 90%. The A/O ratio 1:1 yielded a higher percentage of Cr(VI) removal from aqueous solutions. This could be due to higher free concentration of organic phase when the aqueous to organic phase ratio (A/O) is lower [32]. Beyond 2:1 A/O ratio, the extraction efficiency of Cr(VI) decreased, because the extractant was not able to extract Cr(VI) in higher amounts from the aqueous phase. For further studies it was decided to maintain 1:1 ratio.

3.9. Effect of stripping reagent concentration

It is imperative to back extract the Cr from a loaded organic phase. Various acids and bases are used in this study, such as nitric acid, hydrochloric acid, sulfuric acid, oxalic acid, sodium hydroxide, sodium carbonate,

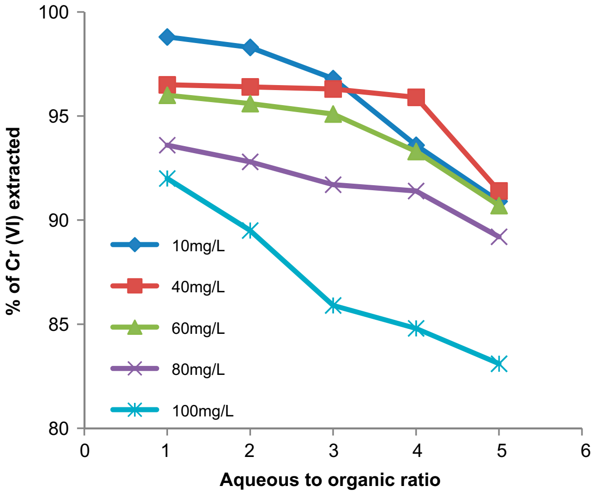


Fig. 4. Effect of aqueous to organic phase ratio (Experimental conditions: Volume of feed phase = 25 mL at pH 1.0 ± 0.1, extractant concentration = 6.8×10^{-2} M.

ammonium carbonate, sodium nitrate, and potassium hydroxide; sodium thio sulfate salts such as sodium salicylate, ammonium salicylate, and sodium nitrate, have been tried as stripping agents. Among them, $NaNO_3$ was best to strip Cr from a loaded organic phase. Fig. 5 shows that the stripping efficiency decreased with increasing concentration of $NaNO_3$ from 0.2 to 2 mol/L. Maximum stripping efficiency (91.1%) was found in 0.2 mol/L $NaNO_3$ solution. The maximum amount of Cr(VI) was stripped within 5 min. Further increase in time did not improve the stripping efficiency. Hence, 0.2 mol/L of $NaNO_3$ solution was chosen for further studies. The stripping reaction mechanism has been given in Eq. (2).

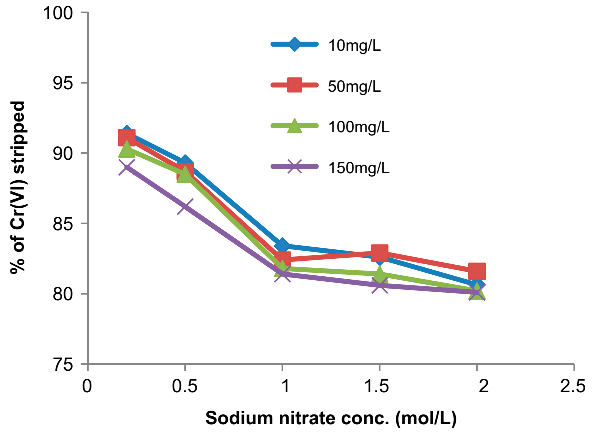
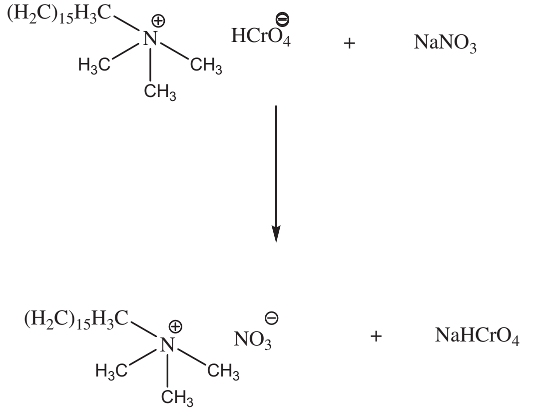


Fig. 5. Effect of carrier concentration (Experimental conditions: Volume of source phase = 25 ml at pH 1.0 ± 0.1, extractant concentration = 6.8×10^{-2} mol/L, volume of organic phase = 25 ml, A/O ratio = 1:1, Cr concentration = 10 mg/L, and equilibrium time = 5 min).



(2)

3.10. Effect of stripping phase ratio

The stripping phase ratio (organic to aqueous phase, O/A) is an important parameter in the stripping process. From Fig. 6 the percentage of chromium(VI) stripping decreased with increasing O/A ratio. The maximum stripping efficiency found for the O/A ratio was between 1:1 and 2:1. Beyond 3:1 O/A ratio, the efficiency of stripping decreased, because the quantity of stripping reagent was not enough to neutralize the acid in the organic phase. Thus, a phase ratio O/A of 1:1 was maintained.

3.11. Effect of stripping contact time

The stripping contact time was determined for the system containing 10 mL of loaded organic phase and

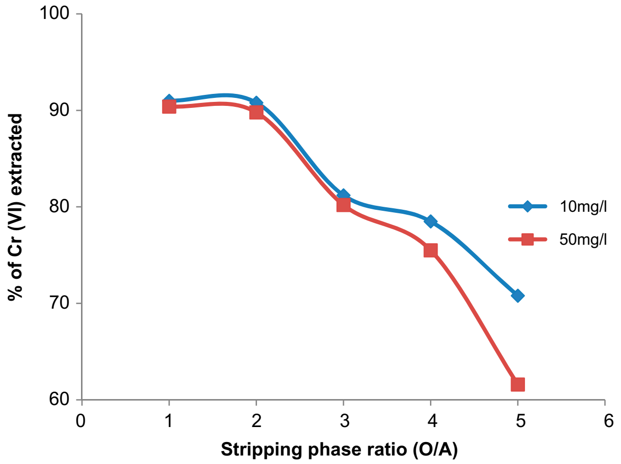


Fig. 6. Effect of stripping phase ratio (Experimental conditions: Volume of feed phase = 25 mL at pH 1.0 ± 0.1, extractant concentration = 6.8×10^{-2} M, Cr(VI) concentration = 10 mg/L, and equilibrium time = 5 min).

Table 1
Effect of diluents on Cr(VI) extraction efficiency

Diluents	Dielectric constant (ε)	Percentage of extraction
Amyl alcohol	18.23	99.0
Dichloromethane	9.1	70.3
Chloroform	4.90	42.7
Carbon tetrachloride	2.23	45.2
Toluene	2.38	–
Xylene	2.28	5.0
Benzene	2.3	–
Hexane	2.02	50.0

Table 2
Effect of Cr concentration

S.No.	Cr concentration (mg/L)	Percentage of extraction
1	10	99.0
2	30	97.5
3	50	85.8
4	70	79.4
5	90	68.7

Table 3
Effect of salt concentration

	Percentage of Cr extraction			
Concentration of anions (mg/L)	10 mg/L	50 mg/L	100 mg/L	150 mg/L
Chloride				
1,000	98.8	97.0	97.2	95.0
2,000	98.7	96.9	97.0	95.1
3,000	97.8	97.0	96.0	94.0
4,000	97.7	97.0	96.2	94.1
5,000	97.4	97.1	95.8	94.1
Sulfate				
1,000	99.2	97.0	95.0	94.0
2,000	99.2	98.0	95.0	94.0
3,000	98.0	97.1	94.9	94.1
4,000	98.0	96.9	95.0	95.0
5,000	97.0	97.0	95.0	95.2

10 ml of stripping agent (0.2 mol/L Sodium nitrate), equilibrated with increase in contact time in the range of 1–3 min. Maximum stripping efficiency was found to be 91.1% in 3 min. Further increase in contact time did not improve the stripping efficiency. Hence, 3 min was selected as the stripping contact time.

Table 4
Characteristics of chrome plating wastewater

Sample number	Parameters	Chrome plating wastewater
1	pH	3.0
2	Conductivity (μS/cm)	3,000
3	Total dissolved solids (mg/L)	1,800
4	Total hardness (mg/L)	350
5	Calcium (mg/L)	130
6	Magnesium (mg/L)	6
7	Chloride (mg/L/)	600
8	Sulfate (mg/L)	500
9	Cr(VI) (mg/L)	10
10	Nickel (mg/L)	0.02
11	Zinc (mg/L)	20.8
12	Iron (mg/L)	1.0
13	Manganese (mg/L)	1.0
14	Copper (mg/L)	9.0

3.12. Application of the developed solvent extraction for industrial wastewater

The developed solvent extraction (or) LLE system was tested for applicability to real industrial electroplating wastewater from local industry. The wastewater was in acidic nature and the chromium concentration was 10 mg/L. The characteristics of the industrial wastewater are given in Table 4 under optimized condition (chromium(VI) concentration 25 ml of plating wastewater solution at pH 1.0 ± 0.1, carrier concentration = 0.06 mol/L, stripping concentration 25 ml of 0.2 mol/L $NaNO_3$ solution, equilibrium time = 5 min at 27°C, and A/O ratio 1:1). The extracted chromium was found to be 99% and it was stripped into 0.02 mol/L of sodium nitrate solution. The extraction and stripping experiments were not affected by various inorganic ions present in the electroplating wastewater.

4. Conclusions

Cr(VI) ion can be effectively extracted by solvent extraction containing HTAB in amyl alcohol. The efficiency of the method depends on various parameters such as the pH of the feed and strip phase, carrier concentration, type of diluents, and temperature. HTAB is able to extract 99% of anionic chromium(VI) from aqueous solution in a equilibrium time of 5 min. The extraction efficiency of chromium(VI) decreases with increased concentration of chromium(VI). The extracted chromium(VI) was successfully stripped into sodium nitrate solution from a loaded organic phase. The maximum extraction efficiency was obtained at pH 1.0 ± 0.1. The A/O ratio 1:1 is maintained in this study. Under optimized conditions, industrial wastewater was also tested and the result was found to be satisfactory.

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