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## Surfactant Enhanced Chromium Removal Using a Silica Gel Column

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### ABSTRACT

A simple and rapid method is proposed for the removal of chromium based on its adsorption as an ion pair with Cetyl Trimethyl Ammonium Bromide (CTABr) on a silica gel column. The surfactant (CTABr) is bonded to chromate by electrostatic force of attraction and the resulting ion-association complex is held on a silica gel column. The resulting solution from the column is analyzed for chromium by the standard diphenyl carbazide spectrophotometric procedure. The method has been applied successfully to study the recovery of chromium in various synthetic mixtures.

**Keywords:** Chromate, CTABr, Ion-association, Silica Gel column.

### RESUMEN

Está investigado simple y rápido método de separación del cromo en la base absorción como pareja iónica con CTABr en la columna con sílica gel. El agente de superficie CTABr tiene los enlaces de fuerzas electrostáticas con cromato y el compuesto complejo está colocado en columna con sílica gel. El cromo en la solución saliente de la columna está analizado con la metodología espectrofotométrica con difenil carbazide. Este método está utilizado con éxito para analizar el cromo recuperado de diferentes mezclas sintéticas.

### INTRODUCTION

The study of pollution by toxic metal compounds assumes considerable importance in chemical process industries. In view of their high toxicity for human health, heavy metal concentrations in wastewater are restricted by strict standards. Chromium is one such metal that requires considerable attention. Industrial wastewater from textile, leather tanning, electroplating, pigmentation and dyes contain chromium in high concentration. Chromium exists in oxidation states from +2 to +6, but only two states, +3 and +6, are of environmental significance. Chro-

mium is found in nature primarily in the trivalent species, as  $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ . Chromium (III) is only slightly soluble in ground water and is very strongly adsorbed by soils. In trace amounts, trivalent chromium is an essential human nutrient [1].

The hexavalent chromate ( $\text{CrO}_4^{2-}$ ) and dichromate ( $\text{Cr}_2\text{O}_7^{2-}$ ) oxyanions are soluble in aqueous systems and are readily transported in ground water. A number of hexavalent chromium compounds are known to be carcinogenic. As a result, the tolerance limit for chromium in drinking water has been established at 100 mg/L total chromium.

The treatment of chromium bearing effluents has been reported through several methods, such as, reduction, precipitation, ion exchange, electrochemical reduction, evaporation, reverse osmosis and direct precipitation. The chemical reduction method involves the conversion of Cr(VI) to Cr(III) with the help of a suitable reducing agent. The reduced form of chromium is then co-precipitated along with Ferric hydroxide or Aluminium hydroxide. Highly selective extractions of chromate from slightly acidic solutions (0.1 to 0.2 mol dm<sup>-3</sup> sulphuric acid) using long chain quaternary ammonium salts such as Aliquat - 336 (Methyl trioctyl ammonium chloride) in chloroform has been studied in detail [2]. In this method vanadium and uranium were co-extracted. Precipitation of voluminous chromium hydroxides involves sludge disposal problem. Ion exchange and membrane separations are more expensive and sophisticated. Studies on the treatment of effluents bearing heavy metals have revealed adsorption to be highly effective, cheap and an easy method among the physiochemical treatment processes. Conventional adsorbents like activated carbon have also been widely explored for the removal of chromium.

Separation of cadmium and chromate ions from waste water has been reported utilizing the process based on enhanced ultra filtration [4]. In this process inorganic membranes (zirconium oxide coated on carbon) were used in the separation cell, and ionic surfactants such as Sodium dodecyl sulphate (NaDS) or Cetyl Trimethyl Ammonium Bromide (CTABr) were added in the effluent. The surfactants, which entrap metal ions present in the feed are retained by the membrane barrier and thus the permeate is clear of metal compounds. In this paper we propose a simple and rapid method for the removal of chromium based on its adsorption as an ion pair with cetyl trimethyl ammonium bromide (CTABr) on a silica gel column. Cetyl trimethyl ammonium bromide is a long chain quaternary ammonium cationic surfactant. This surfactant is bonded to chromate by electrostatic force of attraction and the resulting ion association complex is held on a silica gel column. The resulting solution is then analyzed

for chromium content by the standard diphenyl carbazide spectrophotometric method.

## EXPERIMENTAL

### Reagents

All reagents used were of analytical grade.

Chromium (VI) 1000 mgml<sup>-1</sup>

0.373 g of Potassium chromate (Glaxo laboratories, India) was dissolved in minimum amount of triple distilled water and diluted to 100 ml.

Cetyl trimethyl ammonium bromide (CTABr) (Reidel, India) 0.1 mol dm<sup>-3</sup> was prepared by dissolving 3.645 g of CTABr in minimum amount of triple distilled water and diluted to 100ml.

Silica gel (Sarabhai Chemicals, India). Silica gel column was prepared by making slurry of silica gel and packing it on to a column, which was a buret. The column was packed up to a height of 15 cm.

Diphenyl carbazide (Central Drug House, Mumbai, India). A 0.25% solution was prepared by dissolving 0.25 g of diphenyl carbazide in 100ml of acetone.

## INSTRUMENTATION

A Spectronic -20D (Milton Roy and Company, United Kingdom) spectrophotometer was used for absorbance measurements; pH meter supplied by Elico India Ltd. was used for pH measurements. Glass absorption cells were used to measure the absorbencies.

### General Procedure

A 1 ml of 100 mg ml<sup>-1</sup> Cr (VI) solution was mixed with 5 ml of 0.1 mol dm<sup>-3</sup> CTABr. The aqueous volume was maintained at 10 ml and the solution was transferred to the silica gel column. The flow rate of the liquid was maintained at 1ml min<sup>-1</sup> and the resulting solution was diluted to 50 ml. A 5 ml volume was taken and analyzed for the chromium

content by the spectrophotometric method described below

**Analysis of Chromium**

A series of standard (2-10 mg ) Cr(VI) was prepared and 0.25% of diphenyl carbazide in acetone was added and diluted to 25 ml maintaining an overall acidity of 0.1 mol dm<sup>-3</sup> H<sub>2</sub>SO<sub>4</sub>. The chromium–diphenyl carbazide complex was analyzed spectrophotometrically at a wavelength of 540nm. The calibration graph was found to be linear in the range 2-10 mg / 25ml aqueous volume.

**RESULTS AND DISCUSSION**

**Effect of pH**

The pH of the feed was varied from 3-8 and the adsorption studies were carried out, as before. The adsorption was found to be quantitative over the entire range of pH studied.

**Table I: Effect of pH**

pH	%Recovery
3.0	100
3.5-5.5	100
6.0	99.98
7.0	99.9
8.0	99.7
8.0-11.0	98.6

**Effect of CTABr concentration**

The concentration of the surfactant was also varied from 2ml to 5ml of 0.1 mol dm<sup>-3</sup> in 25 ml of the feed. The ion pair was adsorbed and the chromium in the resulting solution was analyzed as before. No significant reduction in the recovery was observed as a result of the change in the concentration of the surfactant.

**Effect of flow rate and height of the column**

The flow rate and the height of the column were varied from 1ml to 5 ml per minute and 10-15 cm. The adsorption and the recovery were independent of these parameters.

**Influence of other ions**

The effect of the various foreign ions was investigated at 1mg level. A synthetic mixture containing these ions was prepared and the recovery of chromium content was studied by the proposed method. Satisfactory recovery was obtained in the presence of these ions. The results are presented in Table II

**Effect of sample volume**

The sample volume was varied from 10—50 ml and the studies were carried out as before. The

**Table II: Recovery Studies in Synthetic Mixtures**

Synthetic Mixture	Recovery (mg ml <sup>-1</sup> )
Cr(VI) (0.4 mg ml <sup>-1</sup> ) + Fe <sup>2+</sup> (1 mg) + Cu <sup>2+</sup> (1mg) + Cd <sup>2+</sup> (1 mg)	0.398
Cr(VI) (0.4 mg ml <sup>-1</sup> ) + Co <sup>2+</sup> (1 mg) + Cu <sup>2+</sup> (1mg) + Mn <sup>2+</sup> (1 mg)	0.398
Cr(VI) (0.4 mg ml <sup>-1</sup> ) + Ni <sup>2+</sup> (1 mg) + Zn <sup>2+</sup> (1 mg)	0.398
Cr(VI) (0.4 mg ml <sup>-1</sup> ) + Cl <sup>-</sup> (5 mg) + I <sup>-</sup> (5 mg) + SO <sub>4</sub> <sup>2-</sup> (5 mg)	0.396
Cr(VI) (0.4 mg ml <sup>-1</sup> ) + C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> (5 mg) + SO <sub>4</sub> <sup>2-</sup> (5 mg)	0.368

adsorption of chromium was quantitative in the range studied (Table III).

**Table III: Effect of Sample Volume**

Sample Volume	% Recovery
10 ml	100
20 ml	200
30 ml	100
40 ml	99.96
50 ml	99.96

## CONCLUSIONS

Adsorption enhanced by diluted surfactants is an efficient process for the separation of chromium from aqueous solutions. The proposed method is simple and rapid. In comparison with the conventional adsorbents such as activated charcoal, the proposed method provides an alternative novel approach for the removal of chromium. The proposed method is also less tedious and less time consuming when compared to the removal of chromium as chromium hydroxide. The application of the

proposed method to various synthetic mixtures (containing ions such as Copper, Iron, Zinc, Chloride etc resulted in quantitative recovery of Chromium. The advantage of the process is that none of the experimental parameters are highly critical. Efforts are on to apply this procedure to a leather tannery effluent.

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