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# EXTRACTION AND SEPARATION OF Ti(IV) USING THIOPHOSPHINIC ACIDS AND ITS RECOVERY FROM ILMENITE AND RED MUD

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## ABSTRACT

This paper embodies details on the extraction behavior of Ti(IV) along with Mg(II), Mn(II), Fe(III), Al(III), Ce(IV), and V(V) from hydrochloric acid medium employing mono- and di-sulphur analogues of bis-(2,4,4-trimethylpentyl) phosphinic acid, namely bis-(2,4,4-trimethylpentyl)monothiophosphinic acid (Cyanex 302) and bis-(2,4,4-trimethylpentyl)dithiophosphinic acid (Cyanex 301). The effect of various parameters, such as the concentration of the acid, metal ion, and extractant, and the nature of the diluent on the extraction of Ti(IV) has been investigated. Almost quantitative extraction of Ti(IV) is observed in both the extractants in the $1 \times 10^{-2}$ to 0.5 M acidity range. The extractants can hold the metal ion up to one-tenth of its molar concentration. The extracting species is proposed to be $Ti(OH)_2 R_2$, (HR = Cyanex 301/Cyanex 302). The partition data have been utilized for obtaining some binary separations of Ti(IV) from some commonly associated metal ions. The practical utility of the extractant has been demonstrated by recovering Ti(IV) from ilmenite and red mud.

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*Key Words*: Solvent Extraction; Thiophosphinic acids; Ti(IV); Ilmenite; Red mud.

## INTRODUCTION

Titanium is an important constituent of several alloys and catalysts. There is an ever-increasing demand for this metal in high purity. Because of its widespread use, the primary sources are becoming exhausted and efficient methods have to be developed to recover it from low-grade ores and secondary sources. Viewing the high purity requirements of the metal, solvent extraction offers an attractive alternative. Over the years a variety of compounds, such as molten biphenyl(1), amidines(2), catechol(3), alizarin RS(4), have been employed for the extraction and separation of Ti(IV), but in most of these cases, different ions cause serious interference, and a strict control of pH is required. High molecular weight amines (5–7) have also been used for the extraction and separation of Ti(IV); however, their utility is restricted because of the problem of emulsion formation and interferences from coexisting metal ions. Among the organophosphorus extractants, DEHPA has been employed for the extraction and separation of Ti(IV) from chloride and phosphate media (8–10). Recently Phalke et al. (11) also employed DEHPA for the extraction of Ti(IV) from sulphate media and applied the method for its recovery from ores. Biswas and Begum (12) used kerosene solutions of DEHPA for the extraction of Ti(IV) from chloride medium. Jayachandran and Dhadke (13) employed toluene solution of 2-ethylhexyl phosphoric acid mono 2-ethylhexyl ester for the extraction of Ti(IV) from sulphate medium and proposed the extracting species. TBP and TOPO have been explored by Allal and Huachard (14) for the extraction of Ti(IV) from chloride media, but the requirement of a high extractant concentration discourages the commercial use of the extractants. In the 1980s mono- (Cyanex 302) and disulphur (Cyanex 301) analogues of bis-(2,4,4 trimethylpentyl) phosphinic acid (Cyanex 272) were introduced in the market. These extractants have been successfully employed for the extraction and separation of various metal ions. Rickelton and Robertson (15) have patented a method for the recovery of Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) using monothiophosphinic acid and its salt. Rickelton and Boyle (16) have reported Cyanex 301 and Cyanex 302 as stronger extractants for the extraction of Zn(II) than their oxyacid counterpart Cyanex 272. The utility of these extractants has also been demonstrated for the recovery of Cd(II) from wet-process phosphoric acid solutions (17). Cyanex 301 has been used to recover Mn(II) and Zn(II) from battery waste (18). Besides this, use of Cyanex 301 for the extraction of Fe(III) (19), In(III) (20), Cu(II) (21), Co(II) and Ni(II) (22), Mo(VI) (23), Ni(II) (24), Sb(III) and Bi(III) (25), and Cr(III) (26) is also reported in the literature. However, these extractants have not been used so far for the extraction of Ti(IV). The lower $pK_a$ value of these extractants compared to the parent compound make them an attractive choice to extract metal ions at a higher acidity; this advantage is particularly important for Ti(IV), which requires relatively higher acidity to prevent its hydrolysis.

This paper presents extraction data of Ti(IV) in Cyanex 301 and Cyanex 302 along with some other commonly associated metal ions such as Mg(II), Mn(II), Fe(III), Al(III), Ce(IV), and V(V). The effect of different variables on the extraction has been investigated. The composition of the extracting species has been proposed. Based on the partition data some binary separations from Ti(IV) have been achieved. The practical utility of the extractants has been demonstrated by recovering pure Ti(IV) from ilmenite and red mud. The loading and recycling capacities of the extractants have been determined.

## EXPERIMENTAL

### Materials

Stock solutions of metal ions were prepared by dissolving their chlorides/nitrates/sulphates in double distilled water containing a minimum amount of the corresponding mineral acid. Stock solutions were standardized by the usual complexometric titrations (27). All other chemicals and organic solvents were of Analytical Reagent/Synthetic Reagent grade from Qualigens/E.Merck, Mumbai (India). Kerosene of boiling fraction 160 to 200°C, which contains 65–75% aliphatic and 25–35% aromatic fractions was used.

Radioisotope of $^{141}$Ce(IV) was procured from Board of Radiation and Isotope Technology, Bhabha Atomic Research Centre, Mumbai (India). Cyanex 301 (assay 77%) and Cyanex 302 (assay 84%) were received as gift samples from Cytec Industries, Niagara Falls, Ontario (Canada) and used as such without further purification. Samples of ilmenite ($TiO_2$ = 53%, $Fe_2O_3$ = 22%, FeO = 20%, MgO = 1.5%, MnO = 1%, $Al_2O_3$ = 0.6%, $SiO_2$ = 0.5%, and others) and red mud ($TiO_2$ = 21%, $Fe_2O_3$ = 33%, $Al_2O_3$ = 20%, $SiO_2$ = 6%, others = 10–20%) were obtained from Department of Earth Sciences, University of Roorkee, Roorkee (India) and Hindalco (India), respectively.

### Equipment

Atomic Absorption Spectrometer (Perkin Elmer 3100) Perkin Elmer Corp., Norwalk, USA, and Inductively Coupled Plasma-Atomic Emission Spectrometer (Labtum ICP Spectrometer Plasmalab 8440) Labtum Ltd., Victoria (Australia) were used for the determination of metal ions in the aqueous phase for the distribution studies and in ilmenite and red mud. A well type NaI (Tl) Scintillation Detector (SC 6048), ECIL, Hyderabad (India) was used to measure the gamma activity of $^{141}$Ce radioisotope.

## Procedure

For the distribution studies equal volumes (10.0 mL) of the aqueous phase (metal ion solution in an appropriate mineral acid) and the organic phase (Cyanex 301 or Cyanex 302 in a suitable diluent) were shaken at room temperature (25 ± 2°C) for 5 min to ensure complete equilibration. The two phases were separated and suitable aliquots of each phase were assayed for radioactivity or employed for determination by ICP-AES/AAS. The experimental conditions for the various studies are mentioned with the corresponding data. Based on five observations, the value of percent extraction of Ti(IV) at about 90% extraction exhibits a coefficient of variation of ±3%.

Samples of ilmenite or red mud were dissolved by following the method given elsewhere (28). About 1 g of powdered ilmenite or red mud was treated with a 10 mL mixture of concentrated $H_2SO_4$ and concentrated HF (1:5) containing a few drops of concentrated $HNO_3$. The solution was evaporated to fumes to expel Si as $SiF_4$. The paste was finally dissolved in 100 mL of 1 M HCl. These stock solutions of ilmenite and red mud solutions were diluted 10 times and labeled as Ai and Bi, respectively.

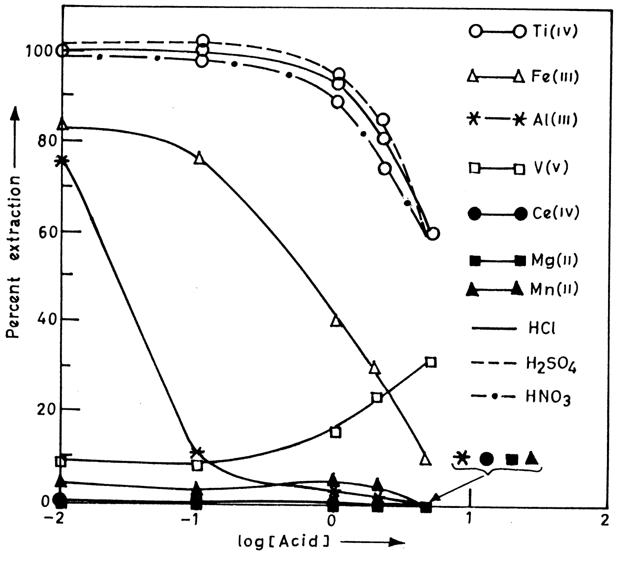

*Figure 1.* Effect of concentration of mineral acid on the extraction of metal ions in toluene solution of Cyanex 301. Conditions: [Metal Ion] = $1.0 \times 10^{-3}$ M
[Cyanex 301] = 0.1 M

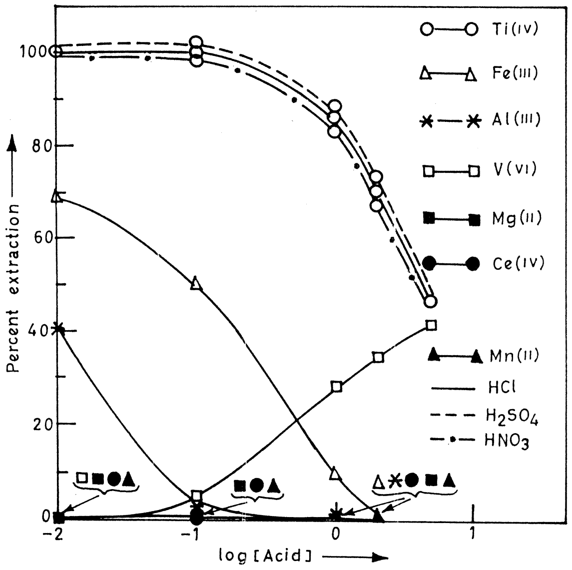

*Figure 2.* Effect of concentration of mineral acid on the extraction of metal ions in toluene solution of Cyanex 302. Conditions: [Metal Ion] = $1.0 \times 10^{-3}$ M
[Cyanex 302] = 0.1 M

## RESULTS AND DISCUSSIONS

### Extraction Behavior

The extraction behavior of Ti(IV) and of other commonly associated metal ions, namely Mg(II), Mn(II), Fe(III), Al(III), Ce(IV), and V(V) ($1 \times 10^{-3}$M) from $1 \times 10^{-2}$ M to 5 M hydrochloric acid in 0.1 M toluene solutions of Cyanex 301 and Cyanex 302 is given in Fig. 1 and 2. In both the extractants Ti(IV) shows almost quantitative ($\gg$98%) extraction up to 0.1 M HCl, beyond which the extraction starts slowly declining to a value of $\gg$70%. Fe(III) and Al(III) show a decreasing trend with increasing acid molarity. The extraction of V(V) is low ($\gg$5%) and almost constant between 0.01 M to 0.1 M HCl and then shows slight increase with increasing acid molarity. Mg(II), Mn(II), and Ce(IV) show negligible extraction ($<$3%) over the entire investigated range of acid molarity. The extraction behavior of all the investigated metal ions from $HNO_3$ and $H_2SO_4$ media is more or less similar to that from HCl. As a representative case, the extraction behavior of Ti(IV) from $HNO_3$ and $H_2SO_4$ is shown in figures. The effect of various parameters were investigated at $1 \times 10^{-3}$M Ti(IV) concentration in 0.1 M HCl using 0.1 M extractant concentration unless otherwise stated.

### Effect of Equilibration Time

The effect of equilibration time on the extraction of Ti(IV) using Cyanex 301 and Cyanex 302 was investigated, and it was observed that 2 min time is sufficient for complete equilibration. In all other studies the two phases were shaken for 5 min.

### Diluent Effect

The extraction of Ti(IV) in both the extractants was studied using different solvents of varying nature. Toluene, *n*-hexane, cyclohexane, nitrobenzene, benzene, and kerosene (boiling fraction 160–200°C) were employed for the said purpose. The results (Table 1) indicate that there is quantitative extraction of Ti(IV) in all the diluents except chloroform where around 92% extraction is observed. It is also apparent that kerosene (160–200°C fraction) can replace toluene for commercial applications. There is no pronounced difference in the behavior of the two extractants.

### Effect of Concentration of the Extractant

The effect of concentration of both the extractants (0.001–0.5M) on the extraction of Ti(IV) is shown in Fig. 3. There is an increase in the extraction with in-

***Table 1.*** Extraction of Ti(IV) in Different Diluents of Varying Dielectric Constant Using Cyanex Extractants[a]

| | | Percent Extraction | |
|---|---|---|---|
| Solvent | Dielectric Constant | Cyanex 301 | Cyanex 302 |
| *n*-hexane | 2.00 | 96.2 | 95.1 |
| Benzene | 2.28 | 97.3 | 98.2 |
| Cyclohexane | 2.22 | 99.5 | 98.9 |
| Toluene | 2.44 | 99.1 | 99.3 |
| Kerosene (160°–200°C) | 2.02 | 99.0 | 99.4 |
| Chloroform | 4.80 | 92.3 | 92.5 |
| Nitrobenzene | 34.8 | 97.7 | 95.0 |

[a] Conditions: [Metal ion] = $1.0 \times 10^{-3}$ M, [HCl] = 0.1 M
[Cyanex 301/302] = 2 (v) × 0.1 M (toluene)

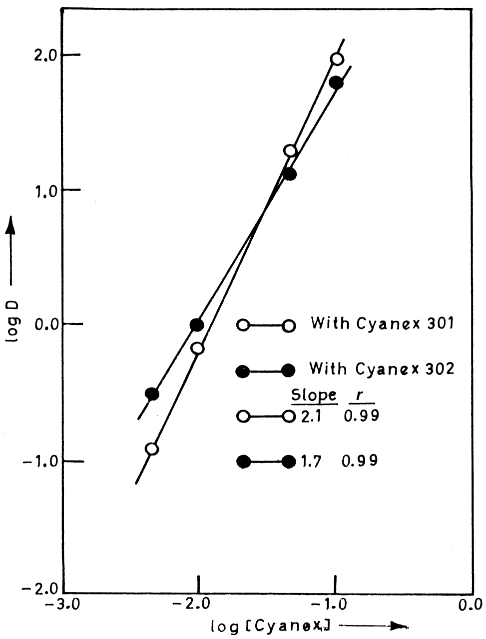

*Figure 3.* Effect of concentration of [Cyanex 301/Cyanex 302]$_{\text{total}}$ on the extraction of Ti(IV) from hydrochloric acid medium. Conditions: [Ti(IV)] = $1.0 \times 10^{-3}$ M
[HCl] = 0.1 M

crease in the concentration of the extractants. The log-log plot between extractant concentration and distribution ratio in both the cases gives a straight line with a slope around two suggesting the involvement of two molecules of extractants in the formation of extracting species. The stoichiometry of the extracting species is 1:2 (metal:extractant). Assuming that Ti(IV) is mainly present as $Ti(OH)_2^{2+}$ in the aqueous solution in the pH range 1.0–1.6 (29), the extracting species can be proposed as $Ti(OH)_2R_2$ where $HR$ = Cyanex 301/Cyanex 302, which acts as a cation exchanger. Similar species has been proposed by Katsuta and Yanagihara (30) for the extraction of Ti(IV) with acetylacetone in the pH range 1.09–1.55. The value of extraction coefficient ($K$) for Ti(IV) with the extractants is calculated using the following relation:

$$\log K = \log D - 2 \log [HR] - 2\,\mathrm{pH}$$

The values of $K$ are found to be $85.1 \pm 3$ and $70.4 \pm 3$ for Cyanex 301 and Cyanex 302, respectively. A lower value of extraction coefficient with Cyanex 302 is on the expected lines as it is a weaker acid (31).

## Effect of Concentration of Metal Ion

The effect of metal ion concentration ($1 \times 10^{-5}$ M–1 M) on the extraction of Ti(IV) has been investigated at 2 M HCl. The results of the loading of the extractant with varying concentration of Ti(IV) are shown in Fig. 4. A straight line in the concentration range $1 \times 10^{-5}$ M–$1 \times 10^{-2}$ M indicates that the distribution ratio does not change with increase in metal ion concentration, suggesting that the extracting species does not change in this concentration range. Beyond $1.0 \times 10^{-2}$ M loading starts setting in, and it can be concluded from the plot that the extractant can hold the metal ion to a maximum of one-tenth of its molar concentration. Similar results have been obtained for the loading of Mo(VI) (23) and Ni(II) in Cyanex 301 (24).

## Stripping of Ti(IV)

Various stripping reagents were tried for the stripping of Ti(IV) from the organic layer. Results are summarized in Table 2. Data indicate that 3% $H_2O_2$ in 0.5 M $H_2SO_4$ and 4 M HCl/$H_2SO_4$ can be used for the quantitative recovery of Ti(IV). 3% $H_2O_2$ in 0.5 M $H_2SO_4$ was preferred for selective stripping of Ti(IV), as

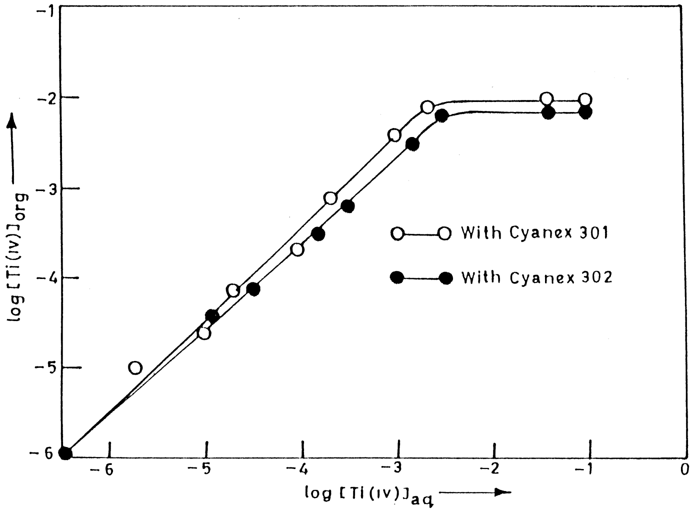

*Figure 4.* Extraction isotherm for the extraction of Ti(IV) from hydrochloric acid medium. Conditions: [Cyanex 301/Cyanex 302] = 0.1 M
[HCl] = 2 M

***Table 2.*** Efficiency of Different Stripping Reagents for the Back Extraction of Ti(IV)[a]

| | Ti(IV) Recovered from Organic Phase (Percent) | |
|---|---|---|
| Stripping Reagent | Cyanex 301 | Cyanex 302 |
| 0.1 M tartaric acid | 25.6 | 37.6 |
| 0.1 M sodium citrate | 15.9 | 25.2 |
| 0.1 M oxalic acid | 33.3 | 43.4 |
| 0.1 M $Na_2S_2O_3$ | 30.2 | 50.8 |
| 2 M HCl | 45.0 | 52.3 |
| 4 M HCl | 98.1 | 98.3 |
| 4 M $H_2SO_4$ | 98.2 | 99.1 |
| 3% $H_2O_2$ in 0.5 M $H_2SO_4$ | 98.2 | 99.1 |

[a] Conditions: [Metal ion] $= 1.0 \times 10^{-3}$ M, [HCl] $= 0.1$ M
[Cyanex 301/302] $= 2$ (v) $\times$ 0.1 M (toluene)

Fe(III) is also stripped by 4 M HCl/$H_2SO_4$. The organic phase after stripping can be easily regenerated by washing it with water till the washings are neutral.

### Hydrolytic Stability and Recycling Capacity of Extractants

The hydrolytic stability of Cyanex 301 and Cyanex 302 towards 0.1 M HCl was determined by keeping a toluene solution of extractant in contact with it. No decrease is observed in the percent extraction of Ti(IV) with the two extractants even after 20 days of contact (Fig. 5). However, the hydrolytic stability of the extractants towards HCl and $H_2SO_4$ for prolonged contact is well documented. (31,32). Experiments were also conducted to determine the recycling capacity of extractants by first loading them with Ti(IV) and then stripping with 3%$H_2O_2$ in 0.5 M $H_2SO_4$. The stripped organic phase was regenerated and used for extraction. The recovery of each step is calculated from the amount of Ti(IV) that gets extracted in the organic phase in that particular cycle. The results reveal practically insignificant change in the efficiency of the extractants up to 10 cycles (Fig. 6).

### Separations

Based on the partition data, some binary separations of Ti(IV) from other associated metal ions can be achieved using Cyanex 301 or Cyanex 302. Both the

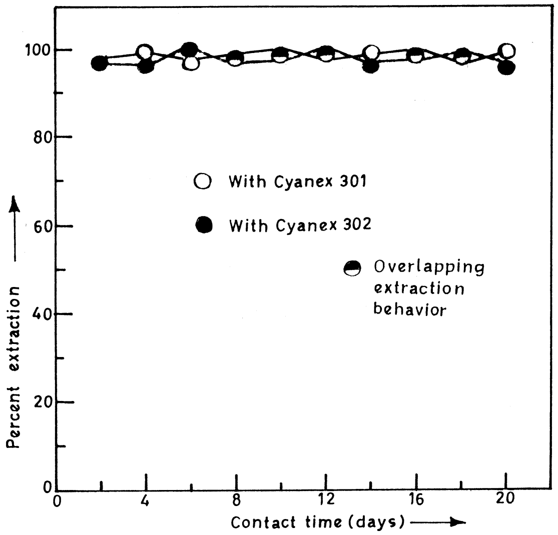

*Figure 5.* Variation in the extraction of Ti(IV). Conditions: [Metal ion] = $1.0 \times 10^{-3}$ M
[Cyanex 301/302] = 0.1 M
[HCl] = 0.1 M

extractants are equally effective for the separation of Ti(IV) from Mn(II) and Ce(IV). In these separations Ti(IV) is extracted in the organic phase leaving the other in aqueous phase. Ti(IV) is then stripped from organic phase using 3% $H_2O_2$ in 0.5 M $H_2SO_4$. However, for the separation of Ti(IV) from Al(III) and V(V) Cyanex 302 is preferred over Cyanex 301 because Al(III) and V(V) show relatively poor extraction in the former. For the separation of Ti(IV) from Fe(III), both the metal ions are first extracted in the organic phase by contacting twice. Ti(IV) is selectively stripped employing 3% $H_2O_2$ in 0.5 M $H_2SO_4$ followed by the stripping of Fe(III) using 0.1 M oxalic acid. Cyanex 301 is a preferred choice for this separation because of higher extraction of Fe(III) in it than Cyanex 302. The separation conditions and recoveries are summarized in Table 3.

## Recovery of Ti(IV) from Ilmenite and Red Mud

In order to test the practical utility of the proposed methods Ti(IV) has been recovered from ilmenite and red mud by the mentioned procedure.

Ten milliliters of ($A_i$) or ($B_i$) solution were equilibrated twice with 10 mL portions of 0.1 M extractant solution. Ti(IV) and Fe(III) along with the traces of Al(III) are extracted in the organic phase leaving behind other metal ions in solution. Ti(IV) is stripped from the organic phase by washing it twice with a 20 mL aliquot of 3% $H_2O_2$ in 0.5 M $H_2SO_4$. ($A_f$) and ($B_f$) refer to the recovered Ti(IV) solution from ilmenite and red mud solution, respectively. Table 4 gives the compositions of $A_i$, $B_i$, and the recovered Ti(IV) solutions $A_f$ and $B_f$. In the case of ilmenite, around 98% recovery of Ti(IV) is achieved, whereas it is around 97% in the case of red mud. The purity of this recovered titanium with respect to major impurities is around 99%. The organic layers can be regenerated by subsequently washing them with 2 × 10 mL 0.1 M oxalic acid to remove Fe(III) and 2 × 10 mL 0.1 M tartaric acid to remove Al(III). It is finally washed with water till the washings are neutral. The extractant is then ready to be reused.

Present investigations reveal that both the extractants are useful for the recovery of reasonably pure Ti(IV) from its sources. Almost quantitative recovery of Ti(IV) can be achieved in a wider acid molarity range using either of the two

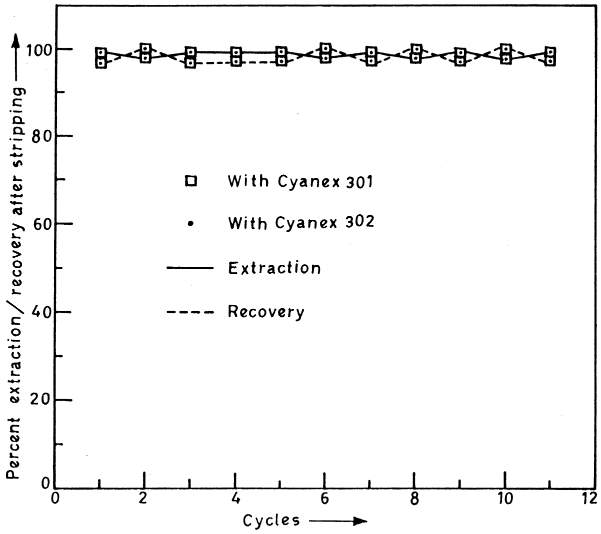

***Figure 6.*** Variation in the extraction/recovery of Ti(IV) in successive cycles.
Conditions: [Metal ion] = $1.0 \times 10^{-3}$ M
[Cyanex 301/302] = 0.1 M
[HCl] = 0.1 M
Stripping Reagent = 3% $H_2O_2$ in 0.5 M $H_2SO_4$

***Table 3.*** Separation of Ti(IV) from Mn(II), Fe(III), Al(III), Ce(IV), or V(V) Using Cyanex Extractants[a]

| No. | Metal Ion Separated | Aqueous Phase HCl (M) | Added Metal Ion (Ratios) | Extractant Cyanex | Mn(II)/Al(III)/Ce(IV)/V(V) Remaining in Aqueous Phase (%) | Ti(IV)[b] Recovered from Organic Phase (%) | Fe(III)[c] Recovered from Organic Phase (%) |
|---|---|---|---|---|---|---|---|
| 1. | Ti(IV)-Mn(II) | 0.1 | 1:1 | 301/302 | 97/96 | 98/97 | |
| | | | 1:10 | | 96/96 | 97/98 | |
| | | | 10:1 | | 97/97 | 98/96 | |
| 2. | Ti(IV)-Fe(III) | 0.1 | 1:1 | 301 | | 98 | 97 |
| | | | 1:10 | | | 98 | 99 |
| | | | 10:1 | | | 99 | 98 |
| 3. | Ti(IV)-Al(III) | 0.1 | 1:1 | 302 | 97 | 96 | |
| | | | 1:10 | | 97 | 97 | |
| | | | 10:1 | | 98 | 99 | |
| 4. | Ti(IV)-Ce(IV) | 0.1 | 1:1 | 301/302 | 96/98 | 97/98 | |
| | | | 1:10 | | 97/99 | 97/99 | |
| | | | 10:1 | | 96/97 | 99/99 | |
| 5. | Ti(IV)-V(V) | 0.1 | 1:1 | 302 | 96 | 98 | |
| | | | 1:10 | | 95 | 99 | |
| | | | 10:1 | | 98 | 99 | |

[a] Conditions: [Metal ion] = $1.0 \times 10^{-4}$ to $1.0 \times 10^{-4}$ (1:1), $1.0 \times 10^{-4}$ to $1.0 \times 10^{-3}$ (1:10), $1.0 \times 10^{-3}$ to $1.0 \times 10^{-2}$ (10:1), [Cyanex 301/302] = 0.10 M (toluene)

[b] Ti(IV) stripped by using 2 (v) × 3% $H_2O_2$ in 0.5 M $H_2SO_4$

[c] Fe(III) stripped by using 2 (v) × 0.1 M oxalic acid

*Table 4.* Composition of Ilmenite Solution (A) and Red Mud Solution (B) before and after Extraction with Cyanex 301/Cyanex302[a]

| No. | | Sample | Metal Ion Separated | Concentration in $A_i$ & $B_i$ (mg/L) | Concentration in $A_f$ and $B_f$ (mg/L) | Percentage for Ti(IV) Recovery | Percentage for Ti(IV) Purity |
|---|---|---|---|---|---|---|---|
| 1 | a | Ilmenite | Ti(IV) | 301.2 | 296.7 | 98 ± 2 | 99 |
| | b | | Fe(III) | 312.7 | 0.1 | | |
| | c | | Mg(II) | 10.1 | <0.1 | | |
| | d | | Mn(II) | 9.4 | <0.1 | | |
| | e | | Al(III) | 4.2 | <0.1 | | |
| | f | | V(V) | 1.0 | <0.1 | | |
| | g | | Ce(IV) | 0.6 | <0.1 | | |
| 2 | a | Red Mud | Ti(IV) | 129.3 | 126.1 | 97 ± 2 | 99 |
| | b | | Fe(III) | 210.2 | 0.8 | | |
| | c | | Al(III) | 99.2 | 0.2 | | |
| | d | | Mg(II) | 25.1 | <0.1 | | |
| | e | | V(V) | 6.9 | <0.1 | | |
| | f | | Mn(II) | 6.8 | <0.1 | | |

[a] [Cyanex 301/302] = 2 (v) × 0.1 M (toluene)
<0.1 = Below detection limit

extractants. Moreover, the recycling power of both the extractants is good, thus suggesting the economic viability of the process.

## CONCLUSIONS

The present findings show that Cyanex 301 and Cyanex 302 can be used to separate Ti(IV) from some commonly associated metal ions found in ilmenite and red mud. The proposed binary separations are conveniently achieved. Also, the separation efficiency is not significantly affected by a change in the molar ratio of the two metal ions involved. Moreover, toluene can be replaced by a cheaper diluent like kerosene (160–200°C boiling fraction). The extraction of Ti(IV) does not require a strict control of pH, and the separation from coextracted Fe(III) can be easily achieved by selective stripping. The extraction system can be used for the recovery of pure Ti(IV) from its ore and an important secondary sector material.

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