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Preparation of Hematite Powders with Plate-Like Morphology from Titania Wastes by Solvent Extraction Followed by Precipitation Stripping and Hydrothermal Synthesis

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ABSTRACT

This paper embodies the results of the investigations carried out on the solvent extraction of iron(III), magnesium(II), aluminum(III), titanium(IV), vanadium(V), chromium(III), and manganese(II) from hydrochloric acid solutions using tributylphosphate (TBP) in kerosene as an extractant. The results demonstrate that iron(III) is extracted into kerosene as $HFeCl_4 \cdot 2TBP$. On the other hand, magnesium(II), aluminum(III), titanium(IV), vanadium(V), chromium(III) and manganese(II) were not

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extracted with TBP from 2.0 mol/dm^3 hydrochloric acid solutions. The potential of the TBP system for the recovery of high purity ferric chloride from the simulated waste chloride liquors of the titanium minerals processing industry was assessed. Studies were also carried out to prepare hematite powders from titania wastes by solvent extraction combined with precipitation stripping and hydrothermal synthesis. The iron oxide powders were characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM) and particle size analysis. The recycling capacity of the TBP system was tested by both extraction and infrared (IR) spectral data.

Key Words: Hematite powders; Solvent extraction separation; Precipitation stripping; Hydrothermal synthesis; Titania wastes.

INTRODUCTION

The manufacture of versatile white pigment, TiO_2, through chloride process generates large amounts of acidic waste chloride liquors containing multivalent metal ions such as magnesium(II), aluminum(III), titanium(IV), vanadium(V), chromium(III), manganese(II), iron(II), and iron(III). The typical composition of the waste chloride liquor from the titanium minerals processing industry is given in Table 1. The management of the waste liquors from the titanium minerals processing industry represents a major environmental issue, affecting every industrialized country. Pyrohydrolysis of the spent acid for the recovery of hydrochloric acid generates impure iron oxide containing several other metal ions with no marketable outlets and, hence, results as a solid waste. Further, many valuable metal species were not

Table 1. Typical composition of waste chloride liquor from titanium minerals processing industry (Kerala Minerals and Metals Ltd., India).

Constituent	g/dm^3
$FeCl_2$	170–210
$FeCl_3$	40–60
$MgCl_2$	15–16
$MnCl_2$	7–8
$AlCl_3$	3–6
$TiOCl_2$	2–3
$CrCl_3$	3–7
$VOCl_3$	3–4
HCl	30–40

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recovered for potential use. The more our economy recovers and recycles useful metals from its wastes, the less mining will be needed and less environmental damage will result from waste disposal. Nowadays, solvent extraction is being used for the recovery of metal values from industrial wastes streams.[1,2] Recently, a solvent extraction process was reported from our laboratory for the recovery of high purity ferric chloride from the titanium minerals processing industry using a mixed-solvent system consisting of tributylphosphate (TBP) and methyl isobutyl ketone (MIBK).[3] However, the above process has some drawbacks, as it employs MIBK. As is well known, MIBK suffers from drawbacks such as low flash point, high vapor pressure, and solubility in the aqueous phase. In order to overcome the problems associated with the use of MIBK, in the present study, TBP in kerosene was explored as an extractant for the selective extraction of iron(III) from the waste chloride liquors of the titanium minerals processing industry.

Ultrafine iron oxide powders have a wide range of applications, including use as catalysts, pigment, recording media, and ferrofluids.[4–6] Various methods of preparing iron oxide ultrafine powders, such as spray pyrolysis,[7] forced hydrolysis,[8] freeze-drying,[9] mechanochemical processing,[10] microwave irradiation of ferrous hydroxide,[11] microemulsion technique,[12] and hydrothermal synthesis from a solution of $FeCl_3$ and iron powder[13,14] have attracted much attention. A technique for controlling particle size, morphology, and size distribution is critical in synthesizing and processing of iron oxide. Hydrothermal preparation is one of the promising soft chemical routes to obtain crystalline and phase pure products at low temperatures. As a part of our ongoing research efforts for the recovery of metal values from titania wastes, in the present study, an attempt was made to recover high purity hematite powders by solvent extraction technique followed by precipitation stripping and hydrothermal synthesis.

EXPERIMENTAL

Reagents and Apparatus

TBP supplied by Aldrich Chemical Company, USA, was used in the present study. Decanol supplied by Merck, Germany, was used as a modifier. Distilled kerosene (boiling range 160–200°C, composed of aliphatic hydrocarbons) was used as a diluent in this work. All other chemicals used were of analytical reagent grade.

Iron(III) stock solution was prepared by dissolving 324.42 g of $FeCl_3$ in concentrated hydrochloric acid and diluting to 1.0 dm^3 with distilled water. Stock solutions of magnesium(II), aluminum(III), chromium(III), and

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manganese(II) were prepared by dissolving 12.32 g of $MgSO_4 \cdot 7H_2O$, 18.75 g of $Al(NO_3)_3 \cdot 9H_2O$, 25 g of $CrK(SO_4)_2 \cdot 12H_2O$, and 8.45 g of $MnSO_4 \cdot H_2O$ in 1.0 dm^3 of distilled water, respectively. Titanium(IV) solutions were prepared from $TiCl_4$ (Spectrochem, India; purity = 99.0%) by diluting to the required concentration with hydrochloric acid. Vanadium(V) stock solution was prepared by dissolving 5.85 g of ammonium monovanadate in hydrochloric acid and diluting it to 1.0 dm^3 with distilled water. Freshly prepared solutions of vanadium(V) were used in all the experimental studies to prevent partial reduction to tetravalent vanadium in hydrochloric acid solutions with respect to time.[15] Suitably diluted stock solutions of the above mentioned metal ions were used in the extraction and analytical studies.

A Hitachi 220 double beam microprocessor-controlled spectrophotometer (Tokyo, Japan) was used for measuring absorbances. An Orion 720A ion analyzer (Beverly, USA) was used for the pH measurements. A Perkin Elmer Analyst 100 atomic absorption spectrophotometer (Norwalk, USA) was used for the analysis of metal ions in multicomponent mixtures. A Parr 4842 reactor (USA) was used for hydrothermal synthesis. The reactor was A Seifert equipment; CuK_α radiation (Holland) was used for x-ray diffraction patterns. A Nicolet impact 400D IR spectrometer using KBr (Madison WI) was used for infrared (IR) spectra. A Jeol JSM 5600LV scanning electron microscope (Tokyo, Japan) was used for SEM studies. A Sedigraph 5100 x-ray based particle size analyzer (Micromeritics, USA) was used for the particle size analysis.

Extraction and Analytical Procedures

Solvent extraction and stripping experiments were carried out by shaking required volumes of aqueous and organic phases in a glass-stoppered vial using a mechanical shaker at 303 ± 1 K. Preliminary experiments showed that the extraction equilibrium was attained within 2 min. After phase separation, the concentration of the specific metal ion remaining in the aqueous phase was determined by standard procedures. Thus, iron(III) was analyzed by the standard $SnCl_2$ reduction–$K_2Cr_2O_7$ titration method or spectrophotometrically by 1,10-phenanthroline method (whenever the concentrations were low).[16] Magnesium(II), aluminum(III), and chromium(III) were analyzed spectrophotometrically using Eriochrome black-T,[16] Eriochrome Cyanine R,[16] and 1,5-diphenyl carbazide,[17] respectively. Vanadium(V) and titanium(IV) were analyzed spectrophotometrically using hydrogen peroxide.[16] Manganese(II) was analyzed titrimetrically using EDTA.[18] The concentration of the metal ion in the organic phase was obtained bymass balance. The distribution ratio, *D*, is defined as the ratio of the concentration

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of metal ion in the organic phase to that present in the aqueous phase. The batch type counter-current extraction studies were performed at a laboratory scale using separatory funnels of suitable volume. The concentrations of the above metal ions in simulated waste chloride liquor were determined using an atomic absorption spectrophotometer.

Hydrothermal Synthesis

A pressure reactor with monel alloy as material of construction and lined with teflon (capacity of 600 cm^3) was used to synthesize the iron oxide powders. Temperatures were varied from 120°C to 180°C (under autogeneous pressure corresponding to the temperature) with constant stirring (200 rpm). The heating time in the hydrothermal reactor was varied between 30 and 120 min. The precursor, ferric hydroxide used in the hydrothermal transformation was prepared by the following procedure: the iron hydroxide was prepared from the ferric chloride loaded organic phase obtained from the solvent extraction process by precipitation stripping using sodium hydroxide in the range 1.5–3.0 mol/dm^3. The alkaline suspension containing iron hydroxide was then separated from the organic phase and transferred into a hydrothermal reactor. After hydrothermal reaction, the powders were filtered, washed with deionized water and acetone, dried at 50°C, and stored in the desiccator. The precipitates were characterized by XRD analysis and scanning electron microscopy (SEM). The particle size was analyzed by x-ray based particle size analyzer.

RESULTS AND DISCUSSION

Effect of Hydrochloric Acid Concentration

The extraction behavior of iron(III) (1.0 mol/dm^3), magnesium(II) (0.04 mol/dm^3), aluminum(III) (0.03 mol/dm^3), titanium(IV) (0.03 mol/dm^3), vanadium(V) (0.02 mol/dm^3), chromium(III) (0.01 mol/dm^3), and manganese(II) (0.03 mol/dm^3) present in the waste chloride liquors of the titanium minerals processing industry was investigated as a function of hydrochloric acid concentration (0.2–6.0 mol/dm^3) using 2.5 mol/dm^3 TBP in kerosene as an extractant and the results are shown in Fig. 1. The extraction of iron(III) was found to increase with increasing hydrochloric acid concentration in the aqueous phase. On the other hand, magnesium(II), aluminum(III), titanium(IV), vanadium(V), chromium(III), and manganese(II) were found

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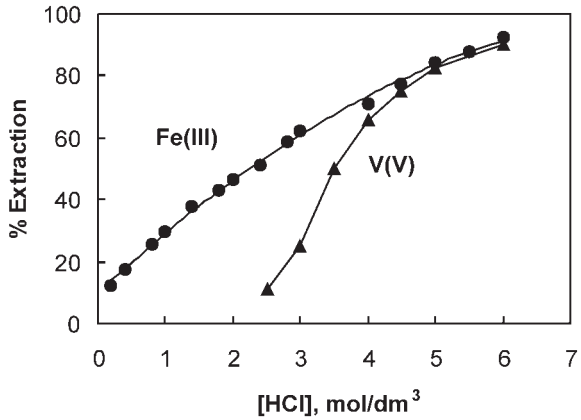

Figure 1. Effect of hydrochloric acid concentration on the extraction of iron(III) ($1.0\,mol/dm^3$) and vanadium(V) ($0.02\,mol/dm^3$) using $2.5\,mol/dm^3$ TBP in kerosene.

not to be extracted into the organic phase between 0.2 and $2.0\,mol/dm^3$ hydrochloric acid concentrations. In view of the better extraction efficiency and selectivity observed between iron(III) and other associated metal ions, in the present study $2.0\,mol/dm^3$ hydrochloric acid was chosen as the feed acidity in the subsequent experiments.

The effect of hydrogen ($2.0–4.0\,mol/dm^3$) and chloride ion ($3.5–6.0\,mol/dm^3$) concentrations on the extraction of iron(III) chloride ($1.0\,mol/dm^3$), respectively, was studied from HCl + NaCl mixtures using $2.5\,mol/dm^3$ TBP in kerosene as an extractant and the results are depicted in Figs. 2 and 3. From the slopes of the log–log plots, it is clear that iron(III) is extracted as $HFeCl_4$ in to the organic phase.

Effect of TBP Concentration

The effect of TBP concentration ($1.5–3.3\,mol/dm^3$) on the extraction of iron(III) chloride was investigated at constant metal ion ($1.0\,mol/dm^3$) and hydrochloric acid ($2.0\,mol/dm^3$) concentrations and the results are shown in Fig. 4. It is clear from the results that the extraction efficiency of iron(III) increases with increasing TBP concentration in the organic phase. From the slope of the plot, log D vs. log[TBP], it is inferred that two molecules of TBP are involved in the extracted complex of iron(III).

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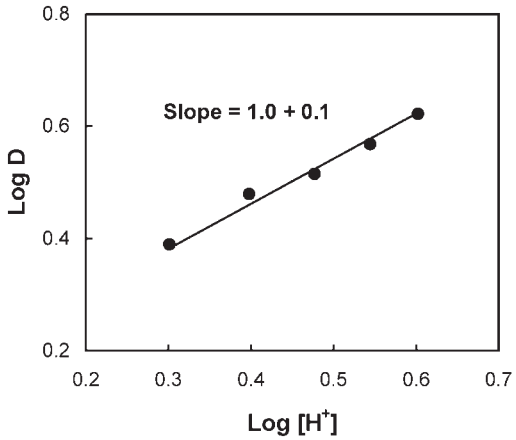

Figure 2. Effect of hydrogen ion concentration on the extraction of iron(III) at constant Cl^- concentration. $FeCl_3 = 1.0\,mol/dm^3$; $[Cl^-] = 4.0\,mol/dm^3$; $TBP = 2.5\,mol/dm^3$.

Extraction Equilibrium

Based on the preceding studies the extraction equilibrium of iron(III) using TBP as an extractant can be represented as:

$$Fe^{3+}_{aq} + H^{+}_{aq} + 4Cl^{-}_{aq} + 2TBP_{org} \rightleftharpoons HFeCl_4 \cdot 2TBP_{org}$$

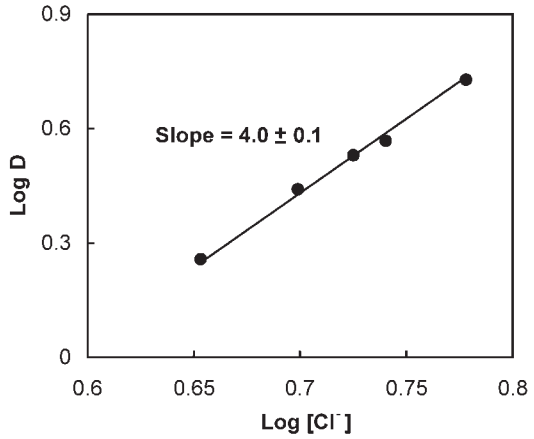

Figure 3. Effect of chloride ion concentration on the extraction of iron(III) at constant H^+ concentration. $FeCl_3 = 1.0\,mol/dm^3$; $[H^+] = 2.0\,mol/dm^3$; $TBP = 2.5\,mol/dm^3$.

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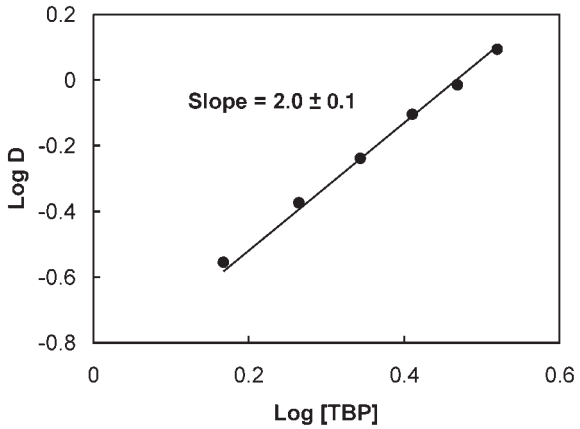

Figure 4. Effect of TBP concentration on the extraction of iron(III). $FeCl_3 = 1.0\,mol/dm^3$; $HCl = 2.0\,mol/dm^3$.

The above extraction equilibrium is in good agreement with the earlier report by Sahu and Das[19] for the extraction of iron(III) from hydrochloric acid solutions with TBP as an extractant. From our laboratory, similar extracted complexes have also been observed for the extraction of iron(III) from $2.0\,mol/dm^3$ HCl solutions with a mixed-solvent system consisting of TBP (70 vol.%) and MIBK (30 vol.%).[3] On the other hand, Majumdar and De[20] reported the extracted species as $FeCl_3 \cdot 3TBP$ from $2.0\,mol/dm^3$ HCl solutions with 100% TBP for the extraction of iron(III). Saji et al.[21] have also reported $HFeCl_4 \cdot 2TRPO$ for the extraction of iron(III) from HCl solutions using trialkylphosphine oxide (TRPO) as an extractant.

Effect of Diluent

The extraction efficiency of iron(III) ($1.0\,mol/dm^3$) from $2.0\,mol/dm^3$ hydrochloric acid solutions was investigated using $2.5\,mol/dm^3$ TBP in various diluents and the results are shown in Table 2. The results clearly demonstrate that diluents such as benzene, xylene, toluene, and kerosene, which have low dielectric constants, show higher extraction efficiency for iron(III). On the other hand, diluents having higher dielectric constants, such as chloroform, gave poor extraction. This may be due to the strong interaction between TBP and chloroform through hydrogen bonding.[22] However, in the present work, MIBK, which has high dielectric constant, shows high extraction for

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Table 2. Effect of nature of diluent on the extraction of 1.0 mol/dm^3 iron(III) chloride from 2.0 mol/dm^3 HCl by 2.5 mol/dm^3 TBP in kerosene.

Diluent	Dielectric constant	% Extraction
Kerosene	2.00	46.3
Cyclohexane	2.02	47.3
Xylene	2.26	48.4
Toulene	2.24	47.3
Benzene	2.28	47.3
Chloroform	4.90	38.0
MIBK	13.11	54.7

iron(III). This can be attributed to the synergistic effect of the mixed-solvent system as described in our previous publication.[23] In view of the commercial availability and high extraction efficiency, kerosene was used as the diluent in the present study.

Loading Capacity

Aliquots of 10 cm^3 of 2.5 mol/dm^3 TBP in kerosene were repeatedly extracted with equal volumes of the aqueous phase containing 1.0 mol/dm^3 of iron(III) chloride in 2.0 mol/dm^3 hydrochloric acid at 303 $\pm$ 1 K for 10 min. The aqueous phases were analyzed for iron(III) after each stage of extraction and the amount of iron(III) content transferred into the extractant phase was calculated. The cumulative concentration of iron(III) in the organic phase after each stage of contact was determined and plotted against the number of stages of contacts (Fig. 5). The loading capacity of 2.5 mol/dm^3 TBP in kerosene for the extraction of iron(III) from 2.0 mol/dm^3 hydrochloric acid solutions was calculated and found to be 40.5 g of iron(III)/dm^3 of the solvent.

IR Spectra of Iron(III)–TBP Complex

The IR spectra of the extracted complex, $HFeCl_4 \cdot 2TBP$, show that, the stretching frequency of the P═O in TBP has shifted from 1276 to 1243 cm^{-1} (Fig. 6). These spectral changes confirm the nature of the extracted complex formed via the coordination of a lone pair of electrons of oxygen in the P═O group of TBP to the metal ion.

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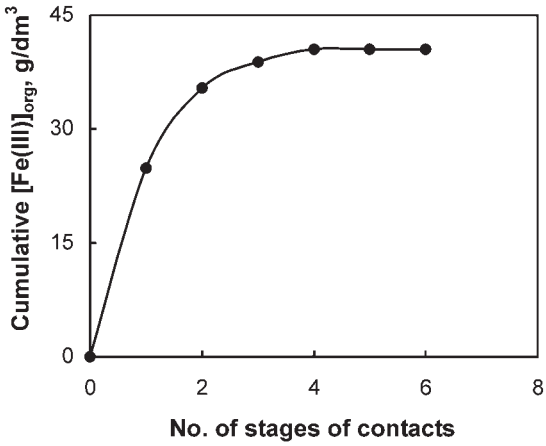

Figure 5. Loading capacity of the solvent system 2.5 mol/dm^3 TBP in kerosene for the extraction of iron(III) chloride. HCl = 2.0 mol/dm^3.

Extraction Isotherm

The extraction isotherm was obtained using a typical feed solution containing 1.0 mol/dm^3 of iron(III) chloride and 2.0 mol/dm^3 of hydrochloric acid using 2.5 mol/dm^3 of TBP in kerosene as an extractant and the results are depicted in Fig. 7. The McCabe-Thiele plot for a feed solution containing 1.0 mol/dm^3 of iron(III) chloride showed that almost quantitative extraction of iron(III) was possible in two counter-current extraction stages at an aq : org phase ratio (A : O) of 1 : 2.

Stripping Behavior of Iron(III) Chloride with Hydrochloric Acid

The effect of hydrochloric acid concentration on iron(III) stripping from a loaded organic solvent system of 2.5 mol/dm^3 of TBP in kerosene [0.5 mol/dm^3 Fe(III)] was investigated and the results are shown in Fig. 8. It is clear from the results that the percentage stripping of iron(III) decreases with increasing hydrochloric acid concentration. In view of its better stripping efficiency, 0.1 mol/dm^3 hydrochloric acid was chosen as a stripping agent for the recovery of iron(III) chloride from the loaded organic phase.

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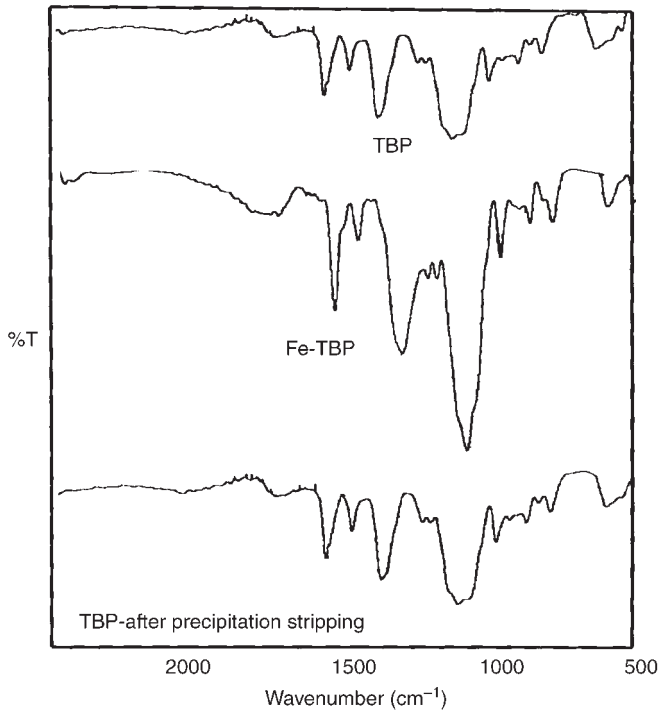

Figure 6. IR spectra of pure TBP, Fe–TBP complex, and TBP after precipitation-stripping.

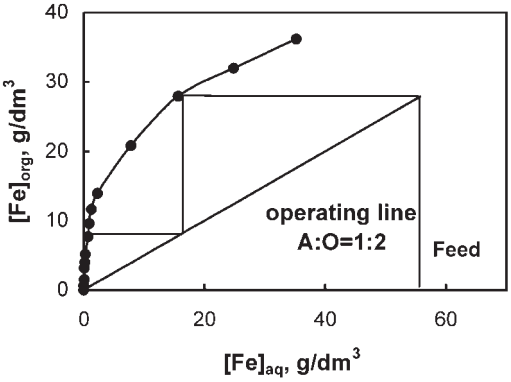

Figure 7. McCabe-Thiele plot for iron(III) extraction. Fe(III) = 55.85 g/dm^3; HCl = 2.0 mol/dm^3.

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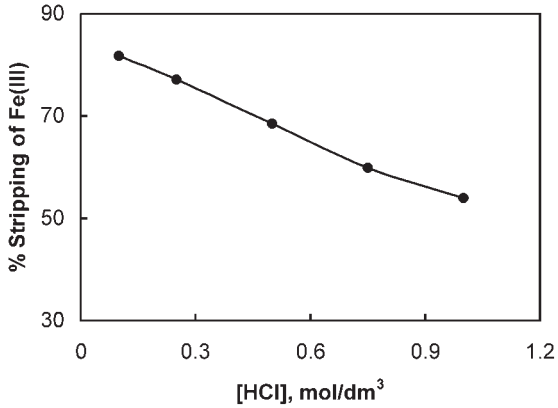

Figure 8. Stripping behavior of iron(III) using hydrochloric acid.

Stripping Isotherm

The loaded organic phase containing 27.92 g/dm^3 iron(III) was used to carry out the stripping studies using 0.1 mol/dm^3 hydrochloric acid and the results are shown in Fig. 9. It is clear from the McCabe-Thiele plot that the quantitative stripping is possible in three counter-current stages with an org : aq phase ratio of 1 : 1.5.

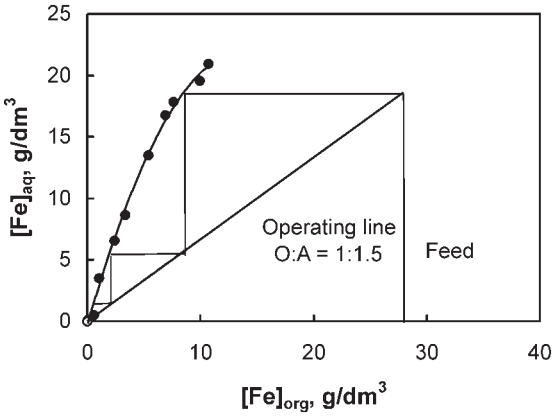

Figure 9. McCabe-Thiele plot for iron(III) stripping. Fe(III) = 27.92 g/dm^3; O : A = 1 : 1.5.

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Extraction and Separation of Iron(III) from Simulated Waste Chloride Liquors of the Titanium Minerals Processing Industry

Based on the above results, a simulated waste chloride liquor consisting of iron(III) (1.03 mol/dm^3), magnesium(II) (0.04 mol/dm^3), aluminum(III) (0.03 mol/dm^3), titanium(IV) (0.03 mol/dm^3), vanadium(V) (0.02 mol/dm^3), chromium(III) (0.01 mol/dm^3), and manganese(II) (0.03 mol/dm^3) in 2.0 mol/dm^3 hydrochloric acid was prepared and subjected to a batch type counter-current extraction process using 2.5 mol/dm^3 TBP in kerosene as an extractant (A : O = 1 : 2). Batch type counter-current extraction and stripping studies were performed at laboratory scale using separatory funnels of suitable volume at 303 $\pm$ 1 K. The loaded organic phase was then subjected to three stages of counter-current stripping by employing deionized water of pH = 1 at an O : A phase ratio of 1 : 1.5. Typical results of the process are given in Table 3. The present investigations revealed that TBP can be used as a selective extractant for the recovery of high purity iron(III) chloride (>99.9%) with an yield of 99% from the waste chloride liquors of the titanium minerals processing industry.

Hydrothermal Synthesis and Characterization of Iron Oxide Powders

The preparation of iron oxide powders comprises the steps of (1) selective extraction of iron(III) chloride from the simulated waste chloride liquor in

Table 3. Extraction of iron(III) chloride from simulated waste liquors of titanium minerals processing industry using 2.5 mol/dm^3 in kerosene as an extractant.

Constituent	Feed (mol/dm^3)	Raffinate (mol/dm^3)	Strip solution (mol/dm^3)
Fe(III)	1.03	0.01	0.34
Ti(IV)	0.03	0.03	ND
Mg(II)	0.04	0.04	ND
Mn(II)	0.03	0.03	ND
Al(III)	0.03	0.03	ND
V(V)	0.02	0.02	ND
Cr(III)	0.01	0.01	ND
HCl	2.00	1.20	0.36

Note: Purity of iron(III) chloride = 99.9%, yield = 99%.

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three stages of counter-current extraction (A : O = 1 : 2) using 2.5 mol/dm^3 TBP in kerosene as an extractant, (2) precipitation stripping of ferric hydroxide from the loaded organic phase obtained from the solvent extraction stream using sodium hydroxide (3.0 mol/dm^3) in an org : aq phase ratio of 1 : 2, and (3) hydrothermal synthesis of iron oxide powders using the alkaline suspension containing iron hydroxide as the precursor. The iron oxides were characterized by XRD, SEM and particle size. The schematic diagram of the process is shown in Fig. 10.

The XRD patterns of the iron oxide powders synthesized at different temperatures (120–180°C) and heating times (30, 60 and 120 min) are shown in Figs. 11 and 12. The diffraction patterns at 120–180°C are typical to the standard pattern of hematite powders (α-Fe_2O_3)[24] and the intensities of the peaks increases with increase in temperature indicating the growing volume fraction of the crystalline hematite. However, the heating time has only a marginal effect on the crystallinity of the hematite from 60 to 120 min (Fig. 12). On the other hand, poor crystallinity was observed when the heating time was 30 min. Figure 13 shows the XRD patterns of the

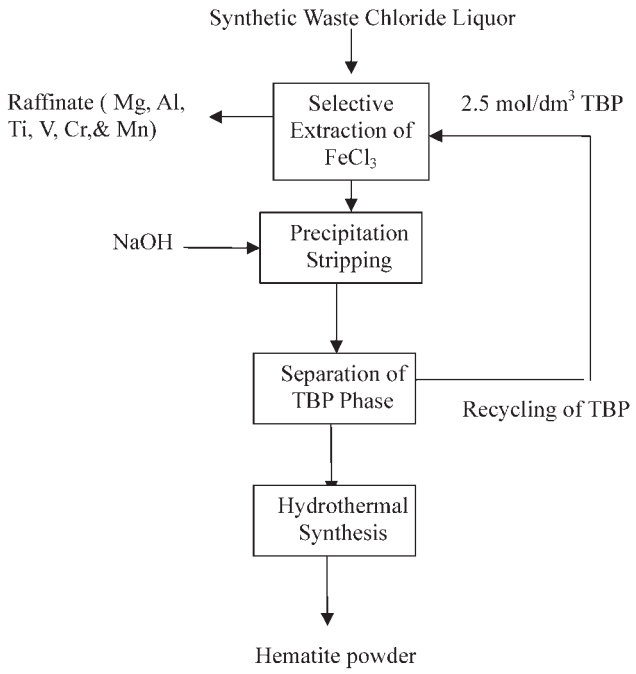

Figure 10. Schematic flow diagram for the preparation of hematite powders.

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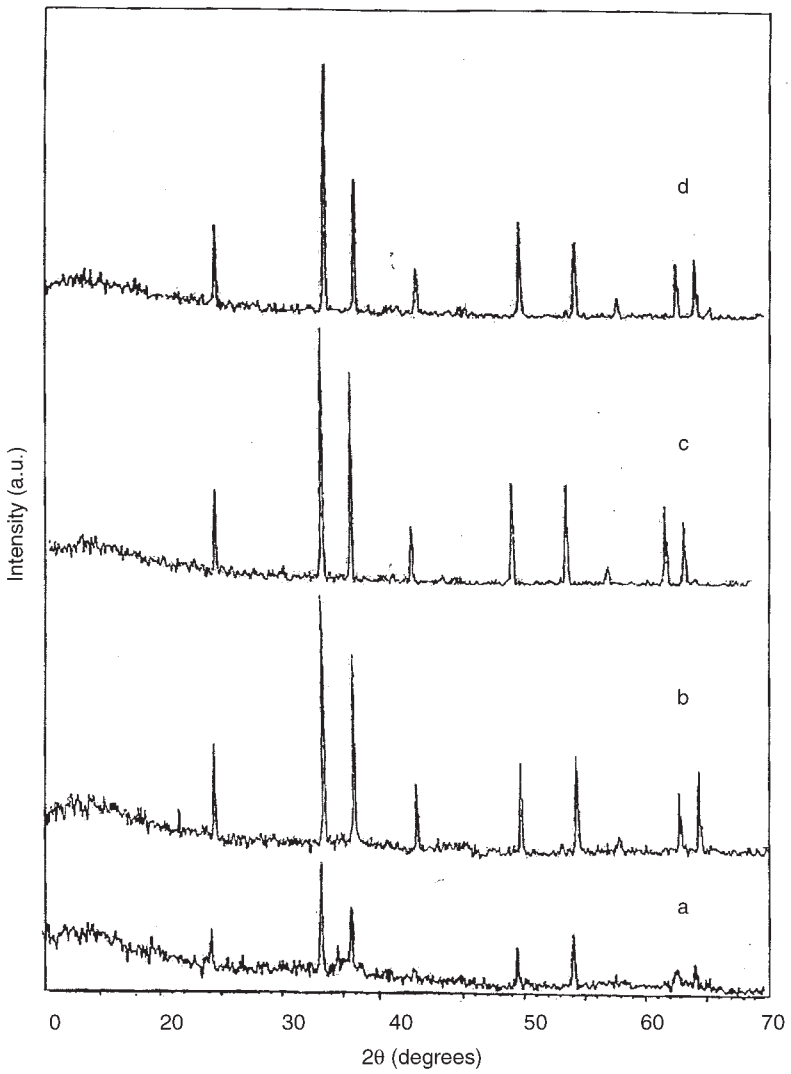

Figure 11. XRD pattern of hematite powders at different temperatures using 3.0 mol/dm^3 sodium hydroxide for precipitation stripping. Reaction time = 120 min; (a) 120°C, (b) 140°C, (c) 160°C, (d) 180°C.

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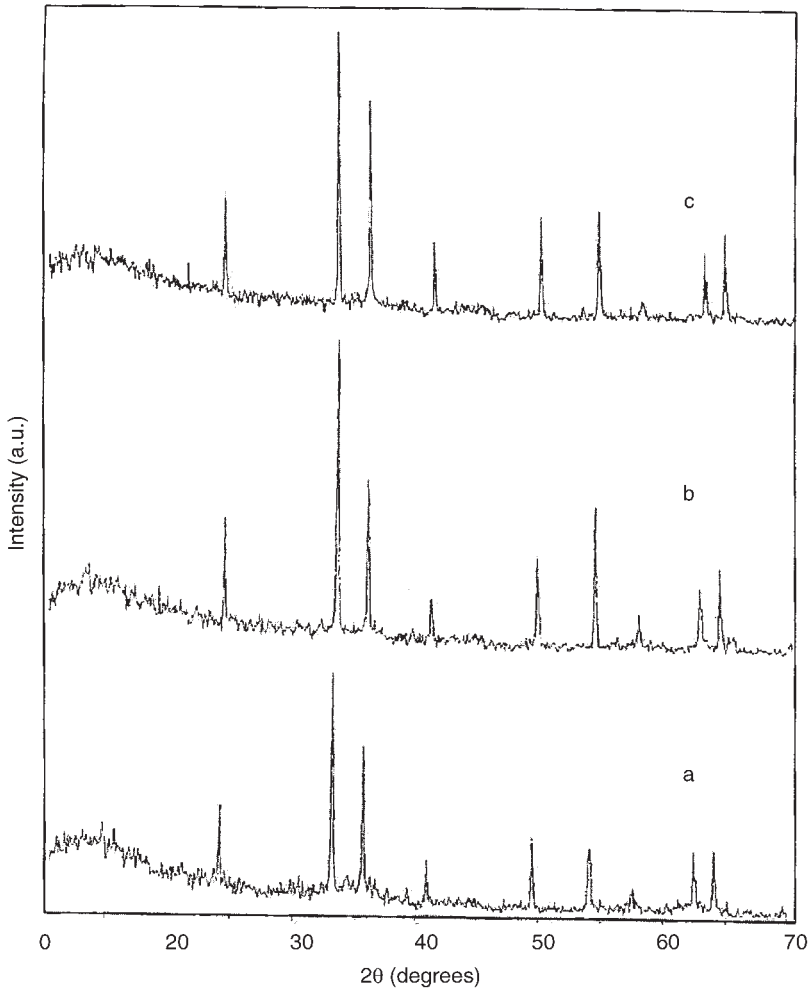

Figure 12. XRD pattern of hematite powders synthesized at different reaction times using 3.0 mol/dm^3 sodium hydroxide for precipitation stripping. Reaction temperature = 140°C; (a) 30 min, (b) 60 min, (c) 120 min.

hematite powders obtained by using different sodium hydroxide concentrations (1.5–3.0 mol/dm^3) for precipitation-stripping at 140°C and heating time of 120 min. The diffraction pattern obtained at 1.5 mol/dm^3 sodium hydroxide contains some iron hydroxide peaks, which indicates the poor conversion of iron hydroxide into hematite powders.

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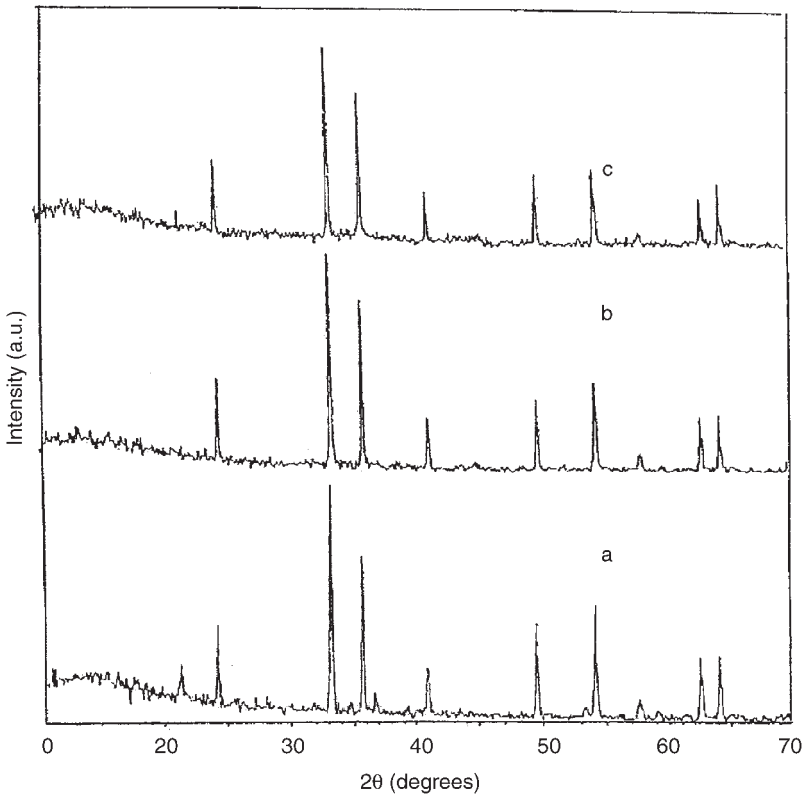

Figure 13. XRD pattern of hematite powders synthesized using different sodium hydroxide concentrations for precipitation stripping at 140°C. Reaction time = 120 min; (a) 1.5 mol/dm^3, (b) 2.5 mol/dm^3, (c) 3.0 mol/dm^3.

Figure 14 shows the scanning electron micrographs of iron oxides prepared at 120°C, 140°C, 160°C, and 180°C, for a heating time of 120 min. The SEM pictures of powders synthesized at 120°C show the presence of amorphous precipitate. On the other hand, the SEM pictures of powders synthesized at 140°C, 160°C, and 180°C show plate-like morphology. The average particle diameters of hematite powders synthesised at 120°C, 140°C, 160°C, and 180°C for 120 min using 3.0 mol/dm^3 sodium hydroxide for precipitation stripping are, 4.0, 4.4, 4.6, and 5.0 μm, respectively, and that of hematite powders prepared under the reaction times 30, 60, and 120 min at 140°C, using 3.0 mol/dm^3 sodium hydroxide for precipitation stripping are 3.0, 3.75, and 4.4 μm, respectively. The average particle diameters of hematite

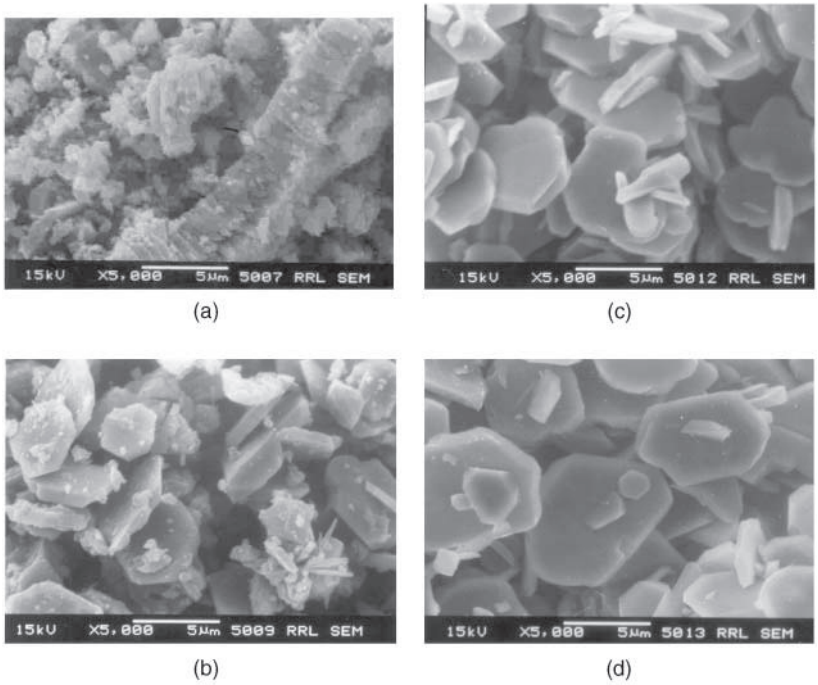

Figure 14. Scanning electron micrographs of hematite powders prepared at different temperatures for 120 min using 3.0 mol/dm^3 sodium hydroxide. (a) 120°C, (b) 140°C, (c) 160°C, (d) 180°C.

powders obtained by using 1.5, 2.5, and 3.0 mol/dm^3 sodium hydroxide for precipitation stripping at 140°C for 120 min are 2.75, 4.25, and 4.4 μm, respectively.

In the literature there have been several reports concerning the synthesis of hematite powders by various methods.[7–12] However, the use of hydrothermal techniques shows versatility for generating hematite powders of different shapes, such as acicular, plate-like, spherical, and polyhedral, which would have potential applications in magnetic recording media, pigment for anticorrosive protection, soft ferrites, inorganic pigment, catalysis, etc.[14] The shape and the size of hematite powders obtained through hydrothermal synthesis has been shown to depend on the precursor composition, the neutralizing media, temperature, reaction time, and the concentration ratio of Fe(II) to OH. The role of various process conditions cited has not been investigated in detail by earlier workers. In the present study, by varying the reaction temperature in the range 120–180°C, it was observed that plate-

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like morphology could be obtained even at the low temperature of 140°C with constant stirring. As a comparison, Lucian et al.[14] have reported plate-like structures only at a temperature of 180°C. Further, the present study has shown plate-like morphology could be achieved at a much lower NaOH concentration (2.5 mol/dm^3), in comparison with an earlier report[14] (4.0 mol/dm^3). Thus, the present investigation provides an optimum set of process conditions for the conversion of organic extractant loaded ferric chloride to hematite powders of plate-like morphology.

Recycling Capacity of the Solvent

The recycling capacity of the extractant was tested, first by extracting iron(III) using 2.5 mol/dm^3 TBP from 2.0 mol/dm^3 of hydrochloric acid solutions containing 1.0 mol/dm^3 ferric chloride and then stripping with 3.0 mol/dm^3 sodium hydroxide in an org : aq phase ratio of 1 : 2. The stripped organic phase was reused for extraction. The extraction efficiency was found to be the same as that of fresh extractant. Further, the recycling capacity was also confirmed by IR spectral data (Fig. 6). It is clear from the IR data that the P═O stretching frequency of TBP was not changed even after precipitation-stripping. The results revealed practically insignificant change in the extraction efficiency of the solvent even after five cycles of extraction and stripping processes.

CONCLUSION

The present investigations clearly highlight that hematite powders of plate-like morphology can be prepared from titania waste chloride liquors by solvent extraction using TBP as an extractant, followed by precipitation-stripping and hydrothermal synthesis, at moderate temperatures. The plate-like hematite powders may find potential application as pigments for anticorrosive protection.

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