Research Article

SOLVENT EXTRACTION OF IRON (III) IN BY TRI-CAPRYL AMINE OXIDE

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ABSTRACT

Extraction of Iron (III) from nitric and perchloric acid solutions with Tri-capryl amine Oxide (TCAO) in benzene has been studied. Effect of several variables like concentration of the extractant, metal ion, acidity, foreign ions etc. were studied on the extraction. The extractions from hydrochloric and sulphuric acid solutions are nearly quantitative and are partial from nitric acid solutions. Based on the results obtained, the probable extracted species are also suggested. Attempts were made to apply the method successfully for the estimation of iron in synthetic and slag samples.

Keywords: Extraction, Iron (III), Tri-Phenyl arsine Oxide (TCAO), Slag samples.

INTRODUCTION

Human beings contains about 4 grams of iron in thetwo proteins namely hemoglobin and myoglobin which play important roles in vertebrate metabolism, respectively oxygen transport by blood and oxygen storage in muscles.

Industrial wastewater is worldwide environmental problem with contamination due to heavy metals like iron, chromium, manganese, zinc etc. Iron (III) could be extracted successfully with reagents of different classification1-8. But there were no reports available in literature on the extraction of iron (III) from nitric acid media using amine oxides as extractants. The present communication describes a detailed study on the extraction of iron (III) by Tricapryl amine Oxide (TCAO) from nitric and perchloric acid solutions.

MATERIALS AND METHODS

TCAO was synthesized by N- oxidation of Tri capryl amine with hydrogen peroxide[9]. A stock solution of 0.45 M TCAO in benzene was prepared and diluted appropriately to get the required concentration. Iron (III) stock solution (0.35M) was prepared using Ferric sulphate (E.Merck) (Mol.Wt.270.3 g/mol) standard potassium dichromate solution volumetrically[9] .All other chemicals used were of Anala R grade and are purified by standard methods. Double distilled water was use throughout the course of these investigations.

Iron (III) extraction

Iron (III) distribution studies were made using appropriate concentrations of the iron salt and mineral acid by equilibrating with an equal volume (10ml) of TCAO in benzene (0.05M) pre-equilibrated with 0.1M mineral acid. The solution was shaken for 5 minutes. The two phases were allowed to settle and separate after equilibration. Iron (III) from the organic phase was stripped with 10ml of 1.0M HNO_3. The concentration of Iron (III) in both the phases was determined by AAS method.

RESULTS AND DISCUSSION

In the extraction of iron (III) by TCAO in benzene as a function of acidity, the distribution ratio (K_d) was found to increase with increase in acid concentration with all the acid systems studied. Maximum extraction efficiency at 9.5M acidity in hydrochloric and 8.5M in sulphuric acid solutions is observed respectively beyond which there is no change in efficiency. (Table.1).The extractions are nearly quantitative with both the acid solutions.

Composition of the extracted species

Composition of the extracted species was determined using extraction isotherm method[10] and distribution ratio methods[11]. In the extraction isotherm method the limiting ratio of the metal to TCA was found unity with both the acid systems. The log-log plots of K_d vs. TCAO from these acid solutions gave straight lines.The slope analysis of the distribution data in both the acid solutions indicate that the solvation number is unity which

is evidenced from the log-log plots with iron depending up on the acid media as per the mechanism mentioned below.

From nitric acid solutions

$$TCAO + H^+ + Fe^{3+} + 4NO_3^- \Leftrightarrow [TCAO.H^+Fe(NO_3^-)_4]_{org}$$

From perchloric acid solutions

$$TCAO + H^+ + Fe^{3+} + 4\ ClO_4^- \Leftrightarrow [TCAO\ H^+Fe(ClO_4^-)_4]_{org}$$

Effect of stripping agents

Iron (III) from the organic phase was stripped back with 20.0ml portions of various concentrations (0.1 – 2.0 M) of HCl, ACOH, H_2SO_4 and HNO_3 solutions. It was observed that 1.0 M HNO_3 alone is a good stripping agent. However in no case the acid strips out all the iron (III) in a single extraction 99.6%. Iron (III) could be recovered from organic phase by making contact three times with equal volumes of 1.0 M HNO_3.

Variation of diluents

Chloroform, carbon tetrachloride, toluene n-hexane, n-heptane cyclohexane, nitrobenzene, dichloromethane and xylene with wide verity in chemical nature and dielectric constant were used as diluents. Low % extraction was noticed from xylene to nitrobenzene (78.2 to 59.4 %). On the other hand maximum extraction efficiency was achieved with benzene as diluent (Table-2). Hence the same diluent was used in all these studies.

Analysis of iron in samples

Ferro alloys such as Ferrochrome are generally produced by electronic arc furnace from melting of mineral chromites[12]. Steel can be produced with different alloying metals such as nickel, molybdenum, vanadium etc. making it more passive and increasing its stainless steel properties. Slag consists of oxides of magnesium, iron chromium and alluminium of various oxidation states[13, 14].

Slag samples are obtained from Jindal Ferro Alloys Corporation, Kothavalasa, and Visakhapatnam Dt. with chemical composition: Cr_2O_3-10-17 %, FeO-2-6 %, SiO_2-25 – 28%, MgO-22-25 %, Al_2O_3 16-22% and CaO-1-3%.

Procedure

An exact weight (1.0 gm) of slag is dissolved in conc. HNO_3 and conc. HCl or mixture of two acids is then subjected to prolonged boiling and evaporation on a water bath. It is then diluted and filtered into a 100 ml flask, washed with distilled water and finally diluted up to the mark. The filtrate is discarded. An aqueous solution (10ml) of iron (III) has been equilibrated with an equal volume of TCAO (0.05 M) in benzene pre equilibrated with appropriate concentration of hydrochloric acid. The iron (III) concentration in the aqueous phase before and after extraction was estimated by AAS as described earlier. The results obtained in these studies were compared by extracting iron from synthetic samples with % composition Fe = 0.5-2.5 ppm and NO_3^- = 8.5 M. Results are presented in Tables - 3.

Conclusions

The proposed method is very simple, rapid and selective. It requires not more than 25 minutes time to extract and estimate iron content in synthetic and slag samples.

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Table 1: Variation of acidity with %extraction

[Fe (III)] = 1.04 x 10^{-4} M [TCAO] = 5.0 x 10^{-2} M

Acid Molarity (M)	HNO_3	$HClO_4$
0.75	75.05	72.82
1.0	75.58	82.58
1.5	80.95	81.95
2.0	81.22	87.45
3.0	84.68	89.26
4.0	85.25	91.83
5.0	90.35	92.80
6.5	90.75	94.55
7.0	91.74	96.42
8.0	94.95	97.75
8.5	96.86	98.87
9.0	98.75	97.61
9.5	98.92	96.15
10.0	98.92	96.05

Table 2: Effect of Diluents on Extraction

[Fe (III)] = 1.04 x 10^{-4} M ; [HNO_3] = 9.5M; [TCAO] = 5.0 x 10^{-2} M

Diluent	Dielectric constant	% extraction
Benzene	2.28	98.9
Chloroform	4.81	91.5
CCl_4	2.23	86.0
Xylene	2.56	78.2
n-Hexane	1.89	76.7
Cyclo hexane	2.0	75.2
Dichloro methane	8.08	68.5
Toluene	2.43	63.8
n-heptane	1.92	60.7
Nitrobenzene	34.82	59.4

Table 3: Analysis of iron (III) in synthetic and slag samples

S. No.	Synthetic sample	Extn method(ppm)	AAS method
1	1	2.05	2.01
2	2	2.14	2.16
3	3	1.96	1.98
4	4	2.14	2.09
5	5	2.11	2.14
6	6	1.92	1.87
7	7	0.75	0.77
8	**Slag sample** 1	1.66	1.68
9	**Slag sample 2**	0.54	0.51

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