
ESTIMATION OF IRON (III) IN INDUSTRIAL EFFLUENTS

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ABSTRACT

Solvent extraction of Iron (III) from aqueous ortho phosphoric and perchloric acid solutions by Tri-benzyl amine (TBA) in chloroform 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 are nearly quantitative from both the acid solutions employed in the study. Based on the results obtained, the probable extracted species are also suggested. The results were compared with the estimation of iron using other reagents. Attempts were made to apply the method successfully for the estimation of iron in alloys and industrial effluents.

KEYWORDS:

Iron (III);
Tri-Benzyl amine (TBA);
Extraction ;
Industrial effluents;

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1. INTRODUCTION

Heavy metal pollution is a serious environmental problem. Among heavy metals Iron ions cause serious problems in the aqueous streams especially at high level concentration [1]. Iron in red blood cells helps as oxygen carrier in human body and plays an important role in the immune system. Deficiency of iron results in memory loss and other related problems [2].

Iron (III) could be extracted successfully with reagents of different classification [3-8]. But there were no reports available in literature on the extraction of iron (III) from phosphoric acid media using amines. It is an observed fact that the free amine cannot extract iron (III) from aqueous acid solutions. Hence it will be pre-equilibrated with the corresponding acid used for the extraction. The object of the present study aims at the extraction of iron (III) by Tri-n-benzyl amine (TBA) from phosphoric and perchloric acid solutions and to establish the nature of extracted species. Attempts were also made to apply this method for the estimation of iron in synthetic and industrial samples.

2. RESEARCH METHOD:

Tri benzyl amine (molecular weight 287.39) was obtained by E. Merck Company used as such without any further purification. A stock solution of 0.35M TBA in chloroform was prepared and diluted appropriately to obtain the required. Ferric ammonium sulphate (E.Merck) was used for preparing iron (III) solution (0.5M) and was standardized using standard potassium dichromate solution volumetrically [9]. All other chemicals used were of AnalaR grade. Double distilled water was used throughout.

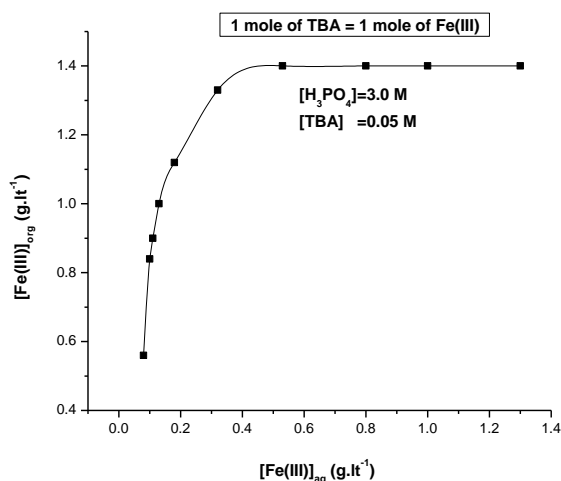
Iron (III) extraction:

In all the extraction studies the organic phase (20 ml) containing the required concentration of TBA 0.05M in chloroform was shaken in 150 ml separating funnel with an equal volume of aqueous phase containing appropriate concentration of for a time period of about 5 minutes. No emulsification was encountered in equilibration. The two phases were allowed to settle for five minutes after equilibration and were separated. Iron (III) from the organic phase was stripped with 10ml of 1.0M HNO₃. The concentration of Iron (III) in both the phases was determined by AAS method.

3. RESULTS AND ANALYSIS:

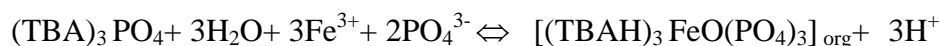
In the extraction of iron (III) by TBA in chloroform as a function of acidity, the distribution ratio (K_d) was found to increase with increase in phosphoric acid concentration i.e., from acid molarity 0.25M up to 2.5M and remained constant thereafter. Extractions are nearly quantitative with both the acid solutions In case of perchloric acid systems maximum extraction efficiency is obtained at 0.25M acidity followed by gradual fall in the extraction beyond this molarity (Table.1).

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 TBA was found unity with both the acid systems(Fig.1). The log-log plots of K_d vs. TBA gave straight lines indicating that the solvation number is unity in both the acid media.

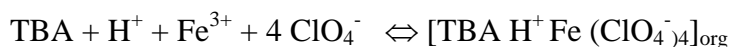


Based on the above information the following extraction mechanism has been proposed:

From phosphoric acid solutions:



From perchloric acid solutions:



3.1 EFFECT OF STRIPPING AGENTS:

20.0ml portions of various concentrations (0.1 – 2.0 M) of HCl, ACOH, H₂SO₄ and HNO₃ solutions were used to strip back Iron (III) from the organic phase. It was observed that 1.0 M HNO₃ alone is a good stripping agent. However in no case the acid strips out all the iron (III) in a single extraction 99.5%. Iron (III) could be recovered from organic phase by making contact three times with equal volumes of 1.0 M HNO₃.

3.2 VARIATION OF DILUENTS

Effect of diluents (Chloroform, carbon tetrachloride, dichloromethane n-hexane cyclohexane, n-heptane nitrobenzene, toluene and xylene) with different chemical nature and dielectric constant were used in these studies. % extraction was found to decrease from benzene to nitrobenzene (89.55 to 59.48). Maximum extraction efficiency was achieved with chloroform as diluent (Table-2). Hence the same diluent was used in all these studies.

3.3 ANALYSIS OF IRON IN SYNTHETIC AND INDUSTRIAL WASTE WATER SAMPLES

Further the results obtained in these studies were compared with those by direct determination method by AAS method. Representative data has been presented in Tables - 3.

The current method has been validated for recovery of iron by extraction in different samples. A known amount of the alloy samples containing iron in the range 0.5 – 1.0 g were weighed accurately (and finely powdered in a mortar. An exact weight of the powdered sample was dissolved in an aliquot of aquaregia. The solution was evaporated and extracted with dilute hydrochloric acid solution. The mixture was shaken well for about 15 min. and the mixture was diluted by 0.01 M HCl solution to the mark and then followed filtered by Whatmann filter paper No. 40. The first portion of filtrate was discarded. The clear solution obtained was made up to 100 ml and used as stock solution. 20 ml of this iron solution was shaken for five minutes with an equal volume of 2.5×10^{-2} M of TBA.

An aliquot (20 ml) of the filtered effluent sample was heated to $1/5^{\text{th}}$ of the initial volume. It was then made up to 100 ml. 20ml of this solution was extracted with an equal volume of 2.5×10^{-2} M TBA in chloform. After separation of two phases; Iron (III) (present in both natural as well as industrial sample) from the organic phase was stripped with 10 ml of

1.0M sulphuric acid and was determined by AAS as described earlier. The results obtained in these studies were compared by extracting iron from synthetic samples with % composition Fe = 0.50-2.50 ppm , pH =2.3 and NO₃⁻ = 1.0 M. Results are presented in Tables - 3& 4.

Table-1: Variation of acidity with %extraction

[Fe (III)] = 1.08 x 10⁻⁴ M

[TBA] = 5.0 x 10⁻² M

Acid (M)	H ₃ PO ₄	HClO ₄
0.25	92.48	82.66
0.5	93.74	80.43
1.0	96.49	79.28
1.5	97.25	72.97
1.75	97.62	70.81
2.0	97.80	70.44
2.5	98.26	69.26
2.75	98.26	67.83
3.0	98.26	62.84

Table-2: Effect of Diluents on Extraction

[Fe (III)] = 1.08 x 10⁻⁴ M ; [H₃PO₄] = 2.5M; [TBA] = 5.0 x

10⁻² M

Diluent	Dielectric constant	% extraction
Benzene	2.28	89.55
Chloroform	4.81	98.26
CCl ₄	2.23	88.40
Xylene	2.56	82.22

n-Hexane	1.89	76.74
Cyclo hexane	2.0	75.25
Dichloro methane	8.08	68.57
Toluene	2.43	68.22
n-heptane	1.92	64.27
Nitrobenzene	34.82	59.48

Table -3: Determination of Iron in Alloys

Material	C	Mn	S	P	Si	Iron	Amt of Iron(III) taken (ppm)	Amt of Iron(III) found (ppm)	% Recovery
Cast Iron	3.430	0.880	0.041	----	2.120	91 - 91.2	90.5	89.92	99.36
Carbon steel	0.007 -1.3	0.3-1.0	0.02-0.06	0.002-0.1	0.005-0.5	98.1-99.5	94.5	93.85	99.31
Wrought iron	0.05-0.25	0.01-0.1	0.02-0.1	0.05-0.2	0.02-0.2	99-99.8	96.5	95.76	99.23

Table-4. Analysis of iron (III) in synthetic and industrial effluents

S.No.	Date	Effluent sample	Before recycling Extn ethod(ppm)	AAS method	After recycling Extn method	AAS method
1	01-02-2019	1	2.15	2.11	0.67	0.64
2	15-02-2019	2	2.22	2.19	0.53	0.48
3	01-03-2019	3	1.98	1.87	0.44	0.47
4	15-03-2019	4	2.04	1.92	0.48	0.41
5	01-04-2019	5	2.13	2.09	0.54	0.44
6	15-04-2019	6	2.11	1.94	0.67	0.74

7	01-05-2019	7	1.94	1.88	0.64	0.72
8	15-05-2019	8	1.95	1.91	0.38	0.35
S.No.	Synthetic sample	Fe(III) added (g/Lt)	Fe(III) Extn method	AAS method	SD	----
1	1	0.10	0.098	0.088	0.018	----
2	2	0.25	0.246	0.239	0.024	----
3	3	0.50	0.492	0.495	0.016	----

4. 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 pharmaceutical samples.

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