

SOLVENT EXTRACTION OF IRON IONS FROM HYDROCHLORIC ACID SOLUTIONS

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ABSTRACT

In this study Methyl Isobutyl Ketone (MIBK) and acetophenone (Methyl Phenyl Ketone) are presented as suitable agents for removal of Fe(III) from concentrated hydrochloric acid solutions.

An extensive study applying 4 parameters was carried out: Time (5, 10, 15, 20, 30 and 60 minutes), Stirring rate (200, 300, 400 and 500 r.p.m), Solvent to HCl Ratio (5%, 10%, 14% and 16%), and initial concentration of Fe ions in crude HCl (95.24, 126 ppm, 330 ppm and 1955 ppm). The best Condition was found to be 30 min – 400 r.p.m – 14 %.

The distilled water was used to strip Fe ions out of MIBK; In order to get most of the MIBK dissolved in both the concentrated HCl and water. Diluents were used such as benzene, kerosene and xylene. Xylene gives better recovery of about 96%.

Key words: Fe(III); Solvation mechanism; Hydrochloric acid; MIBK ;acetophenone

1. INTRODUCTION

Heavy metal pollution is a serious environmental problem. Iron ions are one of the heavy metals that cause serious problems in the aqueous streams especially at high levels concentration [1]

Iron (III) is present in the solutions after hydrometallurgical processes of recovering a number of nonferrous metal ions. Hydrochloric acid (HCl) enriched with iron ions is produced during the synthesis of PVC; hence has lower value than traditional hydrochloric acid. In order to produce high purity hydrochloric acid; an effective means to remove it from the pregnant solutions is often Desirable. [2]

Traditional processes used for treating effluents with heavy metals, include: ion exchange, reverse osmosis, chemical coagulation and precipitation, ultrafiltration, and adsorption. [3-6]

It was found that the adsorption process can be effective in heavy metal removal. In addition to that, the natural adsorbents are environmental friend, existent in large quantities and have good adsorption properties. [7-8]

From literature it was found that solvent extraction and ion exchange are being employed for iron control. [9-10] However solvent extraction processes have become major purification operations in practice [11-13] There are a few research reports on the use of a solvent extraction approach to recover Fe (III) from chloride solutions; the solvent includes various amines and quaternary ammonium salts; Extraction using immiscible solvents containing carbonyl group or Polyethers give very good results but it dissolves to higher rate starting from 6 N concentrations hydrochloric acid. [14-19]

The present work examines the possibility of iron (III) separation from hydrochloric acid solution by solvent extraction using MIBK at various initial metal concentrations and MIBK ratios.

2. Chemicals & Reagents

All chemicals were used as delivered without any purification.

Two liquids, i.e., MIBK (Methyl Isobutyl Ketone: 4-Methyl 2- Pentanone) From SDFCL s d fine chemicals (India) Assay: 99.0 min. Sp.Gr. : 0.80 g/ml and acetophenone (Methyl Phenyl Ketone From Alpha chemicals. Assay: 98% min. Sp.Gr:1.026; were used as metal ion carriers in solvent extraction; benzene, kerosene and xylene as diluents. MIBK was used as 10, 12, 14 and 16 vol. % solution.

The spent acid was a yellow solution with the concentration 11 M and iron concentration range from 95 to 1955 mg/L

3. Liquid-liquid extraction procedure

Extraction was carried out in a typical way: aqueous sample, containing metal ions in 11 M HCl were mechanically shaken with MIBK for different time periods at (25±1°C) and then transferred into glass separator funnels and then allowed to stand for phase separation of suitable volume.

Scrubbing of loaded MIBK, was carried out with deionized water. Stripping was carried out four times consecutively using fresh water each time. Aqueous phases after each stage were taken for analysis.

Benzene, kerosene and xylene were applied as diluents. All tests were carried out at room temperature (25±1°C) to eliminate any temperature effects.

Percentage extraction (E) was calculated from the contents of metal ions in the aqueous Phases before (C₀) and after (C) extraction:

$$C^* = \frac{(V_i \cdot C_i) - (V_w^* \cdot C_w^*)}{V_0^*} \quad [20]$$

Concentrations of iron ions in the organic phase were calculated from mass balance taking into account the change of phase volume, if necessary:

$$E = \frac{C_0 - C}{C_0} \cdot 100\% \quad [16]$$

4. RESULTS AND DISCUSSION

Batch experiments of liquid-liquid extraction of Fe (III) were carried out to verify transport of the iron species with MIBK and acetophenone.

4.1. Effect of different time intervals on % extraction

The percentage extractions of the Fe³⁺ ion from the high-level (125 ppm) aqueous solution for different time periods of MIBK are given in Table 1.

Table 1. Percentage extraction of Fe ions at different time period of MIBK.

Time	Ce	% extraction
5	5.27	98.39
10	4.95	98.49
20	1.66	99.49
30	3.04	99.58
60	3.09	99.54

As shown from table increasing the time from 30 to 60 min has slight effect on % extraction; so time 20 was chosen as optimum time period.

4.2. Effect of different MIBK ratios

The percentage extraction of the Fe³⁺ ion from the high-level (125 ppm) aqueous solution using different concentrations of MIBK (10,12,14 and 16%) at constant contact time 15 and 60 min are given in Table 2.

Table 2. Percentage extraction of Fe ions using different MIBK ratios.

MIBK concentration	%extraction (at 15 min)	%extraction (at 60 min)
10%	89.08	91.98
12%	94.75	96.92
14%	94.83	97.54
16%	94.98	97.26

It is observed that from the MIBK ratio of 12% that the extraction is nearly constant; However MIBK ratio of 14% was chosen as optimum concentration.

4.3. Effect of different stirring speed

The Percentage extractions of the Fe³⁺ ion from the high-level (125ppm) aqueous solution for different stirring speed of MIBK are given in Table 3.

Table 3. Percentage extraction of Fe ions at different stirring speed with MIBK.

r.p.m	Ce	% extraction
100	13.53	95.89
200	12.58	96.18
300	4.79	98.54
400	2.95	99.10
500	11.21	96.60

As shown from table increasing the R.P.M from 100 to 400 increases % extraction then decreases; so time 400 R.P.M was chosen as optimum speed of agitation.

4.4. Effect of initial concentrations of iron (III)

Table 4. Percentage extraction at different initial concentrations of iron (III).

Ci(mg/L)	Ce(mg/L)	% extraction
95.24	2.45	97.42
125.9	3.04	97.58
329.7	2.95	99.10
1955	0.43	99.97

As shown from table 4 increasing the initial concentrations of iron (III) from 95.24 to 1955 ppm has increased % extraction.

4.5. Effect of the diluents on the extraction of iron (III)

In order to get most of the MIBK dissolved in both Conc. HCl and water we used diluents such as kerosene, Benzene and Xylene. Table 5 shows the percent of iron (III) carried out in several combinations of the MIBK and the diluent.

Table 5. Percentage extraction of different combinations of the MIBK and the diluents.

Diluent	Volume before extraction	Volume after extraction	%loss	%Extraction
Kerosene	20 mL	9 mL	52.5%	92.11
Benzene	150 mL	129mL	14%	99.10
Xylene	250 mL	240 mL	4%	99.47

By comparing total volume of the solvent with the different diluents, it is observed that Xylene gives better recovery of about 96% of MIBK with high percent of extraction.

4.6. Comparison of the extraction of iron (III) using Acetophenone and MIBK

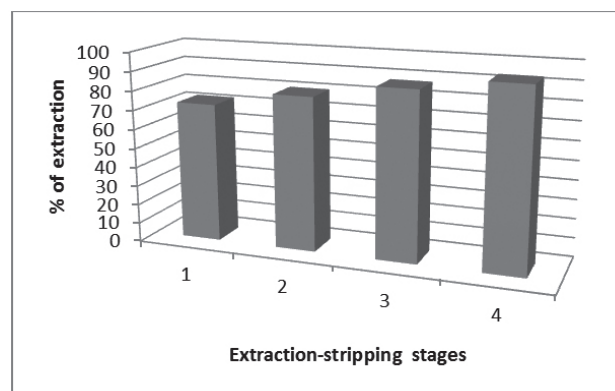
Two successive extraction-stripping stages were carried out with MIBK and Acetophenone using fresh aqueous solutions of about 2000 mg/L Fe (III) in HCl 11 M.

Table 6. The extraction of iron (III) using Acetophenone and MIBK.

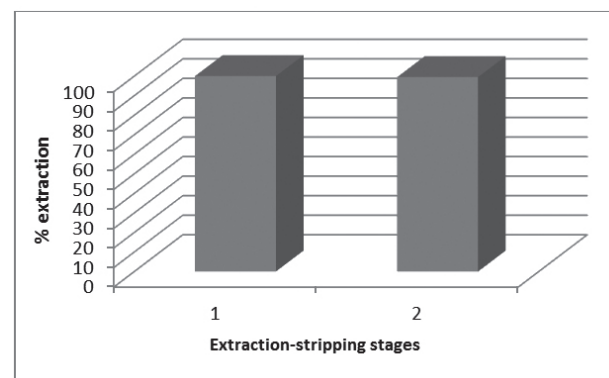
Stages	Acetophenone	MIBK
1	99.95	99.97
2	99.45	99.04

As shown from Table 6 MIBK extraction is slightly higher than for Acetophenone for iron (III) concentration about 2000 mg/L HCl. Therefore, the substitution of MIBK with acetophenone, as an iron (III) extractant from hydrochloric acid solutions, seems to be useless. Moreover, acetophenone is more expensive than MIBK.

4.7. Extractant re-utilization

**Fig.1.** Fe (III) extraction from HCl in four successive extraction-water stripping stages by MIBK.

In order to collect information on the stability of the MIBK for the adopted experimental conditions, four successive extraction-stripping stages were carried out with MIBK, using fresh aqueous solutions of a 95.24mg/L Fe (III) in HCl 11 M. The results obtained for the Fe (III) extraction in each stage, Fig. 1. Clearly denote that the re-utilization of the MIBK is feasible, since no significant decrease of Fe (III) extraction is observed after the successive contacts. A similar behavior is expected for the acetophenone extractant described in this work as shown in Fig. 2.

**Fig. 2.** Fe (III) extraction from HCl in four successive extraction-water stripping stages by acetophenone.

5. Conclusion

The solvent extraction of iron (III) was carried out from hydrochloric acid media using MIBK and acetophenone. Both solvents can be successfully used for removing iron(III) ions from aqueous solution. The maximum removal

percentages of iron(III) ions using both solvents are achieved within the first 20 min. The removal percentages of iron(III) ions increase sharply by increasing solvent ratios up to 14%. As the initial concentration of ions increases the percentage removal using MIBK increases at stirring speed 400 r.p.m.

Using Xylene as solvent stripper giving better result than benzene but it is more expensive.

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