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Extraction of Iron from Aqueous Chloride Media in Presence of Aluminum

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Abstract

The extraction of iron from aqueous chloride media in presence of aluminum was studied at different kinds of extractants(cyclohexanone, tributyl phosphate, diethyl ketone), different values of normality (pH of the feed solution), agitation time, agitation speed, operating temperature, phase ratio (O/A), iron concentration in the feed, and extractant concentration]. The stripping of iron from organic solutions was also studied at different values of normality (pH of the strip solution) and phase ratio (A/O). Atomic absorption spectrophotometer was used to measure the concentration of iron and aluminum in the aqueous phase throughout the experiments. The best values of extraction coefficient and stripping coefficient are obtained under the conditions of operation given in the table below:

System	Operation	рН	Agitation Time (min.)	Agitation Speed (RPM)	Temperature °C	Phase ratio	Cyclohexanone conc.
cyclohexanone	exraction	1.5	10	400	30	3/1	50%
	stripping	1	10	400	30	3/1	50%

Introduction

Iron Extraction

Iron now is being removed from solutions in afew plants; the major reason for its elimination is only to remove it from the system so that the recovery of the major value metal may be facilitated in the subsequent operations. Al-Hemiri and Wadood (1) studied the extraction of iron from aqueous chloride media by cyclohexane and methyl iso-butyle ketone with different operating conditions. Their results showed that, the extraction coefficient increases with increasing extractant concentration. And decreases with increasing iron concentration in feed.The best values of extraction coefficient and stripping coefficient are obtained under the conditions of operating given in table below;

system	operating	Temp.C ⁰	Agitation	Agitation speed(R.P.M)	Phase ratio	normality
			time(min.)			
cyclohexanone	Extraction	25	10	300	3/1	4
	stripping	40	10	300	1/2	0.5
MIBK	Extraction	25	5	300	3/1	4
	stripping	35	20	300	1/1	0.5

Reddy and Saji (2), presented a paper reported on the investigations carried out to recover high purity iron (III), titanium (IV) and vanadium (V) from titania waste chloride liquors, based on their value and concentrations, in two stages solvent extraction process employing commercially available extractants. The first stage clearly shows that high purity iron (III) chloride can be selectivity separated from a multivalent metal chloride feed consist of: FeCl3, TiOCl2, MgCl2, AlCl3, VOCl3, CrCl3, and HCl by employing a TBP-MIBK mixed-solvent extraction system. In the second stage, 2ethyl hexyl phosphoric acidmono-2-ethyl hexyl ester [EHEHPA] in kerosene is used as an extractant to recover titanium (IV) and vanadium (V). This study clearly showed that high purity iron (III) chloride (purity 99.9%) could be selectively separated from a multivalent metal chloride feed.

Arifien et al. (3), studied the extraction of Fe(III), Co(II), Ni(II) and Cu(II) from aqueous media with thiourea monophosphazene (H2MPZ)[little information are available in using this compound as chelating and extracting agent] in chloroform at three degrees of temperature (20,25,and 30oC). It was found that the temperature has no effected on the stoichiometry of the extracted species.

The use of N,N'-tetrasubstituted malonamides such as N,N'-dimethyl-N,N'-diphenylmalonamide (DMDPHMA)

Iron Stripping

Stripping is the reverse process to extraction where problems can occur when the stability of the extracted complex is so great that even concentration acids will not allow the metal to be stripped. The stability of the extracted species will govern the type and concentration of strip solution required(6).

Stripping of vanadium(V) and titanium(IV) after selective recovery of iron(III) was studied by Reddy and Saji (2), vanadium(V) from the loaded organic phase was selectively stripped by using (0.5 mol.dm-3) H2SO4 as a stripping agent in three stages leaving behind titanium(IV) in the organic phase. Subsenquently, titanium (IV) was recovered from the loaded organic phase using a mixture consisting of (2 mol.dm-3) H2SO4 and (2%) H2O2 as a stripping agent in two stages.

Costa et al. (4), investigated the stripping of iron(III) from the loaded organic phases by a simple contact with water, and the selectivity towards iron(III) presented by both malonamide derivatives when several base metal cations co-exist in the acid chloride aqueous solutions can be considered very promising.

In this study, we attempt to extract the iron from aqueous chloride media in presence of aluminum (12).

and N,N'-dimethyl-N,N'-diphenyltetradecylmalonamide (DMDPHTDMA) for the extraction of iron(III) from acid chloride solutions was investigated by Costa et al.(4), in order to evaluate the possibility of using this family of compounds to extract base metal cations. In this investigation, a mechanism for iron(III) extraction from chloride media by DMDPHMA and DMDPHTDMA is proposed, a comparison between their extraction behavior and involved mechanisms for both chloride and nitrate media being considered as well. The results suggested that DMDPHMA and DMDPHTDMA extract iron(III) through different mechanisms, thus showing the influence of the chemical structure on the metal ion transfer reactions to the organic phase. For DMDPHMA, an anionic-pair mechanism involving iron(III) extraction as the chlorocomplex FeCl4- seems to occur, while for DMDPHTDMA a solvation mechanism appears to be the predominant one.

The extraction of magnesium(II), aluminum(III). vanadium(V), titanium(IV), chromium(III), manganese(II), and iron(III) from hydrochloric acid solutions (0.01-2.0 mol dm-3) using various 3-phenyl-4acyl-5-isoxazolones, namely, 3-phenyl-4-(4fluorobenzoyl)-5-isoxazolone (HFBPI), 3-phenyl-4benzoyl-5-isoxazolone (HPBI), and 3-phenyl-4-(4toluoyl)-5-isoxazolone (HTPI) was investigated by Remva et al. (5).

Experimental Work

Experimentation

Chemicals

The following substances are used for carrying out the experiments:

- 1. Hydrochloric acid.5. Cyclohexanone.
- 2. Ferric chloride.6. Tributyl phosphate
- 3. Aluminum chloride. 7. Diethyl ketone
- 4. Benzene.

Equipment:

The equipments used were:

- 1. Water bath equipped with temperature controller (Baird and Tatlock ,Unitemp water bath).
- 2. Electrical mixer with a two blade glass impeller (Janke and Kunkle, IKA-WERK)
- 3. Atomic absorption spectrophotometer (AA-670, Shimadzu corporation, Japan)
- 4. Glass ware (round bottom).
- 5. Separating funnel. The operation steps are shown in Fig. (1)



Fig. (1) Operation steps

Experimental

Extraction Operation

Extractants Selectivity:

In this set of experiments, the effect of selectivity of the extractant on the extraction coefficient was investigated. The aqueous phase was prepared by dissolving FeCL3 and AlCl3 in HCl acid solution to reach the concentration of iron and aluminum of about (4g/l) for each metal. The organic phase used was (cyclohexanone, diethyl ether, tributyl phosphate, diethyl ketone). A volume of the aqueous phase mixed with the specified volume of the organic phase according to the phase ratio. The other variables (pH=1.5, agitation time=10 min., agitation speed=400 RPM, operating temp. =30oC, iron conc.

=4g/l, aluminum conc. =4g/l and phase ratio=3/1) being kept constants. A sample of the raffinate was measured in petrochemicals company – Basrah by using the atomic absorption spectrophotometer (AA-670, Shimadzu Corporation, Japan) for the iron and aluminum concentration.

 $E = \frac{\text{Concentration of metalin organic phase}}{\text{Concentration of metalin aqueous phase}}$

% metal removed = <u>Initial conc. of metal In aqueous phase final conc. of metal in aqueou</u> Initial conc. of metal In aqueous phase

Effect of pH of the Feed Solution

In this set of experiments, the effect of pH of the aqueous solution on the extraction coefficient was investigated. Varied values of pH of the feed solution, which were (1, 1.5, 2, 2.5,3 and 3.5). The organic phase achieved from the previous section and diluted with benzene to 50% vol. /vol. The other variables (agitation time=10 min., agitation speed=400 RPM, operating temp. =30oC, iron conc. =4g/l, aluminum conc. =4 g/l and phase ratio=3/1) being kept constants. A sample of the solution was taken at the end of each period (3, 5, 10, 15 and 20 min.) to determine the iron and aluminum concentrations.

Effect of Agitation Time

In this set of experiments, the effect of agitation time was investigated. The extractant and pH value of the feed were achieved from the previous sections and other variables (agitation speed=400 RPM, operating temp.=30oC, iron conc.=4g/l, aluminum conc.=4g/l and phase ratio=3/1) being kept constants.

Effect of Agitation Speed

In this set of experiments, the effect of agitation speed was investigated. A sample of the solution was taken at the end of each run of different speeds (200,300,400 and 500 RPM) to determine the iron and aluminum concentration. The variables (operating temp.=30oC, iron conc.=4g/l, aluminum conc.=4g/l and phase ratio=3/1) being kept constants. The extractant, pH value of the feed and agitation time were achieved from the previous sections.

Effect of Temperature

In this set of experiments, the effect of temperature was investigated. i.e (30, 35, 40, 45 and 50oC) to determine the iron and aluminum concentration. The extractant, pH value of the feed, agitation time and agitation speed were achieved from the previous sections. And the other variables (iron conc.=4g/l, aluminum conc.=4g/l and phase ratio=3/1) being kept constants.

Effect of Phase Ratio (O/A)

The effect of phase ratio on the extraction coefficient was investigated in this set of experiments. The phase ratios (O/A) used were (1/1, 2/1, 3/1 and 4/1). The other variables (iron conc.=4g/l, and aluminum conc.=4g/l) being kept constants. The extractant, pH value of the feed, agitation time, agitation speed and operating temp. were achieved from the previous sections. The aqueous phase mixed with the specified amount of the organic phase according to the ratio.

Effect of Iron Concentration

In this set of experiments, the effect of initial iron concentration on the extraction coefficient is studied. The extractant, pH value of the feed, agitation time, agitation speed, operating temp. and phase ratio were achieved from the previous sections and aluminum conc.=4g/l being kept constant.

Effect of Cyclohexanone Concentration

The effect of extractant concentration on the extraction coefficient was studied in this set of experiments. The ranges of the concentration of the extractant used is (25, 50, 75, and 100 % vol. /vol.). The extractant, pH value of the feed, agitation time, agitation speed, operating temp. phase ratio and iron conc. were achieved from the previous sections and aluminum conc.=4g/l being kept constant.

Stripping Operation

From the previous studies, the following values are fixed (agitation time =10 min., agitation speed =400 r.p.m., temp.=30 oC).

Effect of pH of the Strip Solution

The effect of pH of the strip solution on the stripping coefficient was studied in this set of runs. The pH varied (0, 1, 1.5, 2 and 2.5). The organic phase (loaded solvent) used was prepared from the extract of extraction stage. The other variable (phase ratio=3/1) being kept constant.

Effect of phase Ratio (A/O)

In this set of experiments, the effect of phase ratio (A/O) on the stripping coefficient was investigated. The organic phase (loaded solvent) used was prepared as in the previous set, pH value of the strip solution was achieved from the previous section. The phase ratio (A/O) used in this set varied (1/1, 2/1, 3/1, and 4/1).

Results and Discussion

Extraction Operation

Effect of Selectivity

The results obtained from this set are listed in Table (1), showing that using (cyclohexanone) as an extractant gave the highest value of extraction coefficient. Cyclohexanone has good kinetic of extraction, the axial overlap of the two (sp3) atomic orbitals from a strong σ bond between them. The carbon-carbon bond length in saturated compounds is found to be pretty constant (0.154 nm (1.54 A)). This refers, however to a carbon-carbon

single bond between (sp3) hybridized carbons. For two carbon atoms bonded to each other the nuclei are drawn inexorably closer together on going from

$$SP3-SP3 \rightarrow SP2-SP2 \rightarrow SP1-SP1$$

The rigidity that take place because of the formation of the ring (7).

Extractants	Percenta	ge Removal	eta^*			
	Fe.	Al.				
Cyclohexanone	90.2	6.9	126.814			
Tributyl phosphate	82	12	33.4094			
Diethyl ketone	75	10	27.0004			

Table 1 the result of extractants selectivity

 $\beta = \frac{\frac{Wt \text{ fraction Fe in solvent}}{Wt \text{ fraction Al in solvent}}}{\frac{Wt \text{ fraction Fe in aqueous}}{Wt \text{ fraction Al in aqueous}}$

Effect of pH of the feed solution

The results as shown in Figs. (2 and 3) indicate that increasing the pH lead to an increase in the extraction coefficient up to a limit then decrease. Thus, pH=1.5 gave the highest value of extraction coefficient. However the general effect of increasing the pH is as shown in the Figs., where the recovery increase with increasing pH values then levels off and finally decreases. The decrease in recovery is due to hydrolysis of the metal (6).



Fig. (2) Extraction coefficient vs. pH, at (t=10 min., speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (3) Percentage removal vs. pH, at (t=10 min., speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)

Effect of Agitation Time

The results clearly demonstrated that using an agitation time of (10min.) gave the highest extraction coefficient and percentage of iron removal as shown in Figs. (4 and 5). The solvent extraction is an equilibrium process, it found that the equilibrium is completed at (10 min.) of contact time and the increasing in the agitation time has no influence on the loading of the organic phase. Similar results were obtained by Al-hemiri and wadood(1), in the extraction of iron from aqueous chloride solutions by cyclohexane and MIBK.

Effect of Agitation Speed

The results, as shown in Figs. (6 and 7), indicate that the agitation speed of (400 RPM) gave the highest values of extraction coefficient and percentage of iron removal.

The surface area of the dispersed phase will depend on the amount of the agitation speed. However, it should not be thought that the greater the agitation speed the greater the rate of metal extraction. Too much agitation speed can result in the formation of a solute or semi-stable emulations. Furthermore, decreasing the drop size of dispersed phase can result in making the drop resemble rigid spheres. In this condition there is no internal movement with in the spheres, no new surfaces are produced, and the extractant with in the sphere cannot get to the surface to reach with metal ions. Consequently, the extraction rate is slow (8).



Fig. (4) Extraction coefficient vs. agitation time, at (pH=1.5, speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (5) Percentage removal vs. agitation time, at (pH=1.5, speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (6) Extraction coefficient vs. agitation speed, at (pH=1.5, t=10 min., T=30oC, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Al conc.=4g/l)

Effect of Operating Temperature

The results plotted in Figs. (8 and 9) indicate that increasing the temperature lead to an increase in the extraction coefficient and percentage of iron removal. Arifien et al. (3), obtained the similar results in the extraction of Fe (μ), Co (Π), Ni (Π) and Cu (Π) with thiourea monophosphazene.

24 Effect of Phase Ratio (O/A)

As indicated in Fig.(10), that increasing the phase ratio (O/A) cause a significant increase in the extraction coefficient and the phase ratio of (3/1) gave the highest value for the system. This might be attributed to the increase in the quantity of transferred metal which is related with quantity of extraction that will furnish the necessary molecules to form the complex to reach the equilibrium state. The behavior is valid to an extent beyond it the extraction coefficient will decrease, because the quantity or volume of the organic phase increases, the amount of metal transferred will undergo somewhat smaller increase leading to a decrease in the concentration of metal, hence causing a decrease in the extraction coefficient(8).

Fig. (11) further support the above conclusion where this figure represents the percentage removal of iron and aluminum, the percentage of iron removal increases up to a limit then it remains nearly constant.



Fig. (8) Extraction coefficient vs. operating temperature, at (pH=1.5, t=10 min., speed=400 RPM, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (9) Percentage removal vs. operating temperature, at (pH=1.5, t=10 min., speed=400 RPM, R=3/1, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (10) Extraction coefficient vs. phase ratio, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, Fe conc.=4g/l, and Al conc.=4g/l)



Fig. (11) Percentage removal vs. phase ratio, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, Fe conc.=4g/l, and Al conc.=4g/l)

Effect of Iron Concentration

The results obtained, here, indicating that increasing the iron concentration in the feed causes the percentage iron removed to decrease as shown in Figs.(12 and 13) knowing that a certain number of extraction molecules associated with each metal ion, thus upon keeping other variables constants, the metal concentration in the solvent will remain almost constant in spite of its increase in the aqueous phase. This will cause a reduction in the extraction coefficient. Similar results were obtained by Islam et al. (9), in the extraction of titanium and iron from acidic sulphate medium.



Fig. (12) Extraction coefficient vs. iron concentration in the feed, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, R=3/1)



Fig. (13) Percentage removal vs. iron concentration in the feed, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, R=3/1)

Effect of Cyclohexanone Concentration

The results showed that increasing the extractant concentration the iron removed increase. This could be seen in Fig.(14 and 15). The wide range of values of extraction coefficient obtained using different concentration (25, 50, 75, and 100%) may be due to the diluent used which could affects the solvation of the extractant and hence, its extractive properties. This influence could be due to the polar nature of the diluent, which might cause an interaction between the diluent and the extractant. Thus, the formation of an extractantdiluent species in the organic phase will produce a lower concentration of the free extractant, with a consequent decrease in the iron removed (10). Similar results were obtained by Islam et al. (11), in the extraction of Ti (IV), Fe (III) and Fe (II) by D2EHPA as the extractant



Fig. (14) Extraction coefficient vs. cyclohexanone concentration, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l)



Fig. (15) Percentage removal vs. cyclohexanone concentration, at (pH=1.5, t=10 min., speed=400 RPM, T=30oC, R=3/1, Fe conc.=4g/l)

Stripping Operation

Effect of pH of the Strip Solution

The results as shown in Figs. (16 and 17), indicate that using an acidic (hydrochloric acid) strip solution at pH=1 gave the highest value of the stripping coefficient. However increasing the pH of the strip solution beyond that causing a decrease in the stripping coefficient and percentage removal as noticed from the same figures. This might be attributed to a reverse action due to unstable complex formation between the extracted species and the acid which will lead to a reduction in the stripping coefficient.

Effect of Phase Ratio (A/O)

The results of this set which are plotted in Figs. (18 and 19). These figures indicate that increasing the phase ratio (A/O) lead to an increase in the stripping coefficient up to a limit then decreases in spite of the percentage of iron removal increases up to a limit then it remains nearly constant. Thus (3/1) is the best ratio for the cyclohexanone system. This could be attributed to the increase in the volume of aqueous phase which will consequently lead to decrease in the concentration of iron. Knowing that the amounts of iron transferred to the aqueous phase are almost constant as it does not require complex formation and therefore, is independent of the amount of aqueous phase used.



Fig. (16) Stripping coefficient vs. pH, at (R=3/1, Fe conc.=3g/l, and Al conc.=1g/l)



Fig. (17) Percentage removal vs. pH, at (R=3/1, Fe conc.=3g/l, and Al conc.=1g/l)



Fig. (18) Stripping coefficient vs. phase ratio, at (pH=1, Fe conc.=3g/l, and Al conc.=1g/l



Fig. (19) Percentage removal vs. phase ratio, at (pH=1, Fe conc.=3g/l, and Al conc.=1g/l)

Conclusions

The following conclusions are drawn from this investigation:

- 1. It was found that cyclohexanone is an active extractant for the iron recovery but it isn't a suitable for the aluminum recovery.
- 2. The separation of iron from aluminum can be obtained at pH =1.5. This point gave a highest value of extraction coefficient (1.4497) and the percentage of iron removal (81.3%).
- 3. A phase ratio (O/A) of (3/1) gave the highest extraction coefficient for the conditions (agitation time =10 min, agitation speed =400 RPM, operating temperature = 30° C) used for the system.
- 4. When the iron concentration decreased in the aqueous feed, the extraction coefficient increased.
- 5. For the given conditions, the extraction coefficient increased when the concentration of extractant increased.
- 6. In the stripping operation, it was observed, that using a phase ratio (A/O) of about (3/1) and pH=1, the strip solution gave the highest stripping coefficient, for the conditions used in the system.

Nomenclature

E= Extraction coefficient O/A= Phase ratio (organic/aqueous) β = Selectivity

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استخلاص الحديد من وسط كلوريد بوجود الالمينوم

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الخلاصة

اهتم هذا البحث ىبراسة استخلاص الحديد من وسط كلوريدي مائي بوجود الألمنيوم بأستخدام انواع مختلفة من المستخلصات(سايكلو هكسانون, تراينيوتايل فوسفات, داي ايثايل كيتون) وبمختلف قيم(درجة الحامضية, زمن المزج, سرعة المزج, نسبة الطور, تركيز الحديد في اللقيم وتركيز المادة المستخلصة). عملية انتزاع الحديد من المحاليل العضوية دُرست ايضا بمختلف قيم درجة الحامضية للمحلول المنتزع ونسبة الطور. استعمل جهاز الأمتصاص الذري لقياس تركيز كل من الحديد والألمنيوم في الطور المائي ولجمية المعامي المربع المور. افضل القيم لمعامل الاستخلاص ومعامل الانتزاع تم الحصول عليها تحت ظروف المائي ولجميع التجارب.

تركيز المادة المشتخلصية	نسبة الطور (عضوي/مائي)	درجة الحرارة (م)	سرعة المزج (دورة \دقيقة)	زمن المزج (دقيقة)	درجة الحامضية	العمل	النظام
%50	1/3	30	400	10	1,5	الاستخلاص	سايكلو هكسانون
%50	3/1				1	الانتزاع	