

Study on the Solvent Extraction of Phosphoric Acid from Phosphate Ore-HCl Digestion Liquor and Pre-treatment with Amines—Part II

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The purpose of this work to study the effect of iron content on the solubility of the phosphoric acid produced by dissolution of Abu-Tartor phosphate ore in HCl at the optimum conditions of dissolution. Also, this work extends to study the pre-treatment of the produced liquor with amines to trap out the iron before extracting and stripping the phosphoric acid. Phase diagrams of organic solvent- H_3PO_4 - H_2O systems show that as the iron content increases the solubility of phosphoric acid in the organic solvent decreases. Trioctyl amine (TOA) and tridodecyl amine (TDA) were used to extract iron from the medium of the dissolution. The obtained results indicate that the efficiency of amines to extract iron depends on the molarity of amine, the nature of the diluent, and the concentration of HCl and the length of the hydrocarbon chain of the amine. The mechanism of removing iron by amines in HCl medium was suggested. The analysis of the obtained data show that both trioctyl amine and tridodecyl amine are good extractant to remove about 95% of iron content and TDA is preferable to TOA because it has low concentration (O.I.M./diluent). This may be attributed to the long chain of the hydrocarbon which leads to relatively high amounts of the donor groups. The aromatic diluent is more suitable than the aliphatic one to avoid emulsion formation. It is found that the order of efficiency of the different solvents to extract H_3PO_4 is:

Tret-amyl alcohol > TBP > Bu-OH > DIPA

and the order of efficiency of the different reagents to remove iron is:
TDA > TOA > DIPA > MIBK > Diethyl ether > (DIPA, 20%).

INTRODUCTION

In our previous studies^{1, 2} on the dissolution, extraction and stripping of Sabyia and Abu-Tartor phosphate ores, it was found that the degree of solubility and the efficiency of extraction as well as the stripping were lower in the case of Abu-Tartor phosphate ore than those for Sabyia one. The obtained results were attributed to the high content of iron in the Abu-Tartor ore as well as the nature of iron as shown from the mineralogical study which showed that 40% of the iron content is in the form of Fe_2O_3 and the remaining of iron is in the form of sulphides.

In 1996, Hanna *et al.*^{3, 4} reduced the iron content in the wet-process phosphoric acid produced from the dissolution of Abu-Tartor phosphate in sulphuric acid by either precipitation with Na_2CO_3 , KOH, NaOH and $(COONa)_2$ or solvent extraction with n-butyl alcohol and tributyl phosphate. The results of both methods show that the reducing of the iron content depends on the nature of the

precipitating agent or the used solvent as well as the optimum condition of the method.

The purpose of the present work was investigation of the effect of the iron content on the solubility of the phosphoric acid produced by dissolution of Abu-Tartor phosphate in HCl. Also, this work extended to study the pre-treatment of the produced phosphoric acid with amines to trap the iron before using the solvent extraction.

EXPERIMENTAL

The starting materials of this work are Abu-Tartor phosphate ore and HCl. Tri-*n*-octylamine (TOA), *n*-butyl alcohol and tridodecyl amine (TDA) were used to trap the iron from the medium of the dissolution. Tributyl phosphate (TBP) and tert-amyl alcohol were used to extract H₃PO₄ from the medium of the dissolution, and H₂O, KOH and Na₂CO₃ solutions were used to strip the acid.

The methods of chemical analysis, the preparation of phosphoric acid, the solvent extraction and the stripping of pure H₃PO₄ by different agents were mentioned elsewhere.^{5,6}

To study the effect of iron content on the solubility of H₃PO₄ in the organic solvents, different concentrations 10, 15, 25 and 30 of H₃PO₄ were prepared. A volume of 25 mL from each sample was taken and the phase diagram of the organic-phosphoric acid-water system was constructed. Three percentages of iron, 2, 3 and 4% were considered.

Preliminary study was carried out to reduce the iron content from an artificial solution containing phosphoric acid (42.5%) and iron (5.7 g FeCl₃·6H₂O, *i.e.*, 2.3% Fe) using 0.2 M tri-*n*-octylamine dissolved in different organic non-polar diluents. The effect of the HCl acid concentration and the variation in the nature of the diluent and amines on the efficiency of the amine to reduce the iron content were investigated. Different concentrations of HCl, 0.48, 0.96, 1.92, 2.40 and 3.00 M, solutions were used.

RESULTS AND DISCUSSION

The phase diagrams of *n*-butyl alcohol-phosphoric acid-water, tert-amyl alcohol-phosphoric acid-water, and tributyl phosphate-phosphoric acid-water systems at room temperature are shown in Fig. 1. From these figures, it is clear that as the percentage of iron increases, the area of the homogeneous layers decreases indicating a decrease in the solubility of phosphoric acid in the organic layer. Fig. 2 represents the results of reduction of iron content in H₃PO₄ by 0.2 M. trioctylamine at different normalities of HCl. The results indicate the percentage of the iron uptake (contained in H₃PO₄) using the organic layer (0.2 M TOA/xylene) depends on the normality of HCl. The percentage of the iron removal increases from 55 to 79.9% as the normality of HCl increases from 0.48 to 3.00 M; on the other hand, in the absence of HCl the percentage of the iron removal equals to 54.1%. Also it is found that the iron extractability by 0.2 M TOA/xylene system generally increases with the increase in HCl concentration (Fig. 2). This result is consistent with the increasing amounts of the extractable

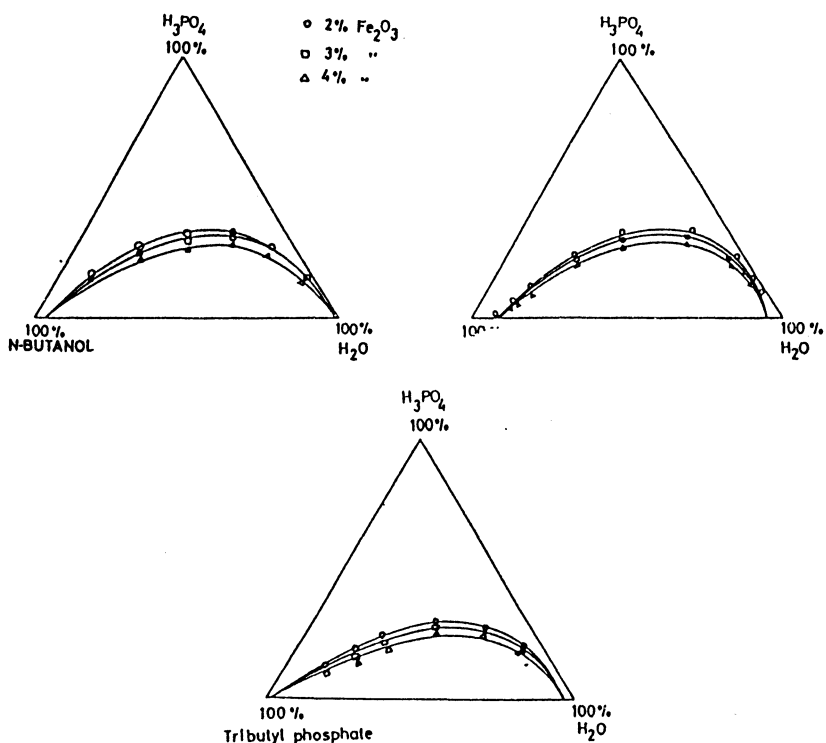


Fig. 1. The phase diagram of: N-Butanol-phosphoric acid water, Tert-amyl alcohol-phosphoric acid water and Tributyl phosphate-phosphoric acid water

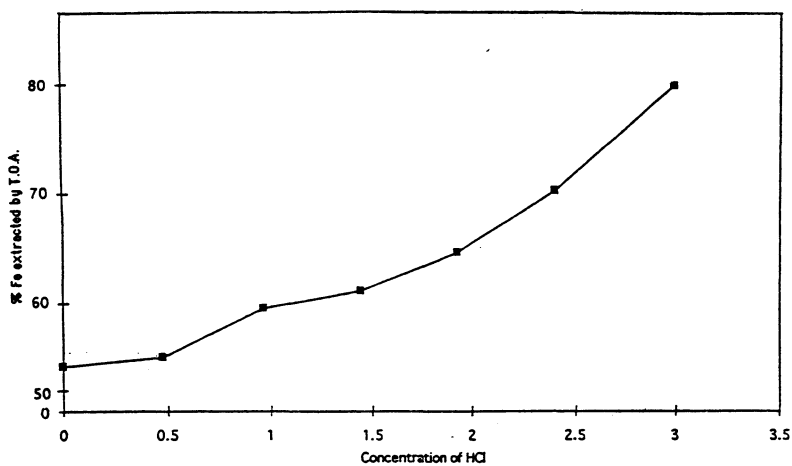


Fig. 2. The variation of Fe% extracted by TOA/xylene with different HCl molarities

species FeCl_4^- , as observed by Good and Bryan⁷. The extraction curve did not have a maximum but shows a tapering off at high concentration (3.0 M HCl), as indicated by Ilic and Catrall⁸. This tapering off is explained by two reasons, namely, the protonation of FeCl_4^- species and the extraction of H_3PO_4 by the amine salt which competes with the extraction of iron⁹. While the mechanism of extraction of iron from its aqueous chloride solution by high molecular weight amines in low dielectric constant diluents has been widely studied¹⁰⁻¹², the mechanism of extraction of iron from phosphoric acid by amines is limited.

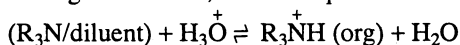
In the case of using tri-*n*-octylamine in reducing the iron content from the medium of dissolution of Abu-Tartor phosphate in HCl acid at the optimum conditions, it was found that the efficiency of amine depends on the concentration of TOA in xylene (Table-1). The results indicate that the percentage of the residual

TABLE-1
THE REDUCTION OF IRON CONTENT WITH DIFFERENT CONCENTRATION
USING TOA IN XYLENE AT DIFFERENT RATIOS (AQU./ORG.)

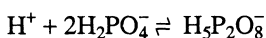
Vol. of aq. (filtrate)	Vol. of org. TOA in xylene	Aq./org. ratio	Remaining Fe% using different concentration of TOA		
			0.2 M	0.1 M	0.05 M
10 ml	10 ml	1 : 1	5.20	7.80	28.5
10 ml	20 ml	1 : 2	4.25	4.50	8.6
10 ml	30 ml	1 : 3	2.13	4.30	5.3

iron in the aqueous layer (acid layer) decreases from 5.20 to 2.13% as the ratio between the amine/xylene and the aqueous layer increases from 1 : 1 to 1 : 3 at 0.2 M amine, while it decreases from 28.5 to 5.3% when the molarity of amine increases from 0.05 to 0.20 M. Thus, in the case of dissolution of Abu-Tartor phosphate in excess HCl² of the stiochiometry of the equations, the best conditions to reduce iron from the filtrate is obtained at 0.2 M TOA/xylene and 1 : 3 ratio of (TOA/xylene) and aqueous layer. Also, the results indicate that the efficiency of TOA in removing the iron content reaches 97.87% from the medium of dissolution ($\text{H}_3\text{PO}_4 = 4.4\%$) while it equals 80% in the artificial phosphoric acid solution ($\text{H}_3\text{PO}_4 = 42.5\%$). This may be attributed to the concentration of H_3PO_4 in both media, whereas the distribution coefficient of Fe between the organic and aqueous phases increases with the decrease of the phosphoric acid content or decreases with the viscosity which enhances the diffusion of amine to trap the iron species.¹³

The suggested mechanism of this route may include a reaction between the amine dissolved in an organic diluent, and the aqueous acid solution as :

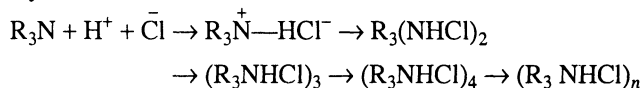


The role of HCl (excess HCl) in the case of using (TOA/diluent) to reduce Fe can be explained as:

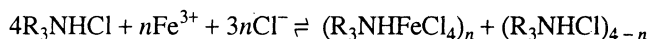




where the phosphate radical can be found in the solution in the form of the previous species or sometimes in most of them. Thus the removal of iron can be explained by:

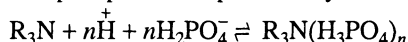


The $(\text{R}_3\text{NHCl})_n$ forms with iron ions a complex:



where $\text{R} = \text{C}_8\text{H}_{17}$ and $n = 1, 2, 3$.

In general, the removal of iron from the phosphoric acid is related to the presence of the iron ions in the form of Fe^{3+} as a complex with phosphates, fluorides or other impurities derived from the ore and/or the processing of the manufacture of H_3PO_4 . In the case of using HCl to dissolve the phosphate ore, some HCl (excess) reduces some ferrous ions to ferric ions and hence the complex is broken; then the iron can form new complexes with the amine species. Also in this process, some amino phosphate complexes may be formed.



The effects of diluent on the reduction of the iron content

In this set of experiments the effect of three diluents, namely, xylene, benzene, and kerosene, on the efficiency of trioctylamine in removing the iron content from the medium of dissolution (phosphate ore/HCl) was studied. After mixing and shaking the phosphoric acid filtrate with TOA/diluent at different ratios, two layers were separated and the iron and the P_2O_5 content were determined in the aqueous layer.

In the case of using benzene as a diluent (Table-2a) it is observed that

TABLE 2a
THE PERCENTAGE OF P_2O_5 AND IRON IN THE AQUEOUS LAYER USING
(0.2 M) TOA/BENZENE

Vol. of aq.	Vol. of org.	aq./org. ratio	Residual % in aq. layer.	
			P_2O_5	Fe
10 mL	10 mL	1 : 1	98.57	6.40
10 mL	20 mL	1 : 2	97.19	4.29
10 mL	30 mL	1 : 3	95.24	4.06

tri-*n*-octylamine removes 95.4% of the iron originally present in the organic phase with a loss of P_2O_5 equals to 1.43%. The efficiency of TOA/ benzene to remove iron from the medium of dissolution increases slightly as the ratio aq./org. layers increases from 1 : 1 to 1 : 3 while the loss in the P_2O_5 increases to reach 4.76%. The same efficiency is obtained by using xylene as a diluent under the same conditions with P_2O_5 loss =57.1% from the organic phase (Tabl- 2b). Benzene and xylene are non-polar solvent ($\mu = 0$) and have nearly the same value of

TABLE 2b
THE PERCENTAGE OF P₂O₅ AND IRON IN THE AQUEOUS LAYER USING (0.2 M)
TOA/XYLENE

Vol. of aq.	Vol. of org.	aq./org. ratio	Residual % in aq. layer.	
			P ₂ O ₅	Fe
10 mL	10 mL	1 : 1	98.00	5.20
10 mL	20 mL	1 : 2	96.66	4.25
10 mL	30 mL	1 : 3	94.29	2.20

dielectric constant ϵ is ca. 2, and so no effect on the efficiency of extraction is observed. In the case of using kerosene (Table-2c) as a diluent, it is observed that as the ratio between the aqueous and organic layers increases from 1 : 1 to 1 : 3,

TABLE 2c
THE PERCENTAGE OF P₂O₅ AND IRON IN THE AQUEOUS LAYER USING (0.2 M)
TOA/KEROSENE

Vol. of aq.	Vol. of org.	aq./org. ratio	Residual % in aq. layer.	
			P ₂ O ₅	Fe
10 mL	10 mL	1 : 1	90.15	6.80
10 mL	20 mL	1 : 2	86.20	6.48
10 mL	30 mL	1 : 3	82.60	13.73

the losses in P₂O₅ increase from 9.85% to 17.40% (organic phase) while the efficiency of the solvent to extract iron decreases from 93.20 to 86.27% with the appearance of an emulsion at the two layers interface. The complete separation of the two layers needs about 24 h. From these results it may be recommended that aromatic hydrocarbon diluents are preferable to the aliphatic ones for the following reasons:

1. The high solubility of amine (TOA) in the aromatic hydrocarbon.
2. The separation of phase during the extraction process is faster and easier.
3. No emulsion formation is observed.
4. No loss in the volume of the diluent.
5. The losses in P₂O₅ content is small, about 2.0% in the case of xylene and 1.43% in the case of benzene (within acceptable experimental error).

This study is in agreement with the conclusion obtained by Krsiura¹⁴ who extracted more than 41 elements from hydrochloric acid solution by 2% (v/v) TOA in CCl₄. He found that the distribution coefficient of phosphorus compounds between the amine/CCl₄ and the aqueous layers is $< 2 \times 10^{-3}$, while the curve of distribution coefficients of iron in HCl medium exhibits a maximum value. The role of the diluent can be explained on the basis that the active organic extractant (amine species) is almost always dissolved in a carrier solvent, which for most purposes is chemically inert with respect to the amine and the acid. The primary function of the carrier as a diluent is to provide suitable viscosity in order to obtain maximum mass transfer of the metal of iron from the aqueous to the organic phase.

The effect of hydrocarbon chain on the reduction of the iron content

To study the effect of the hydrocarbon chain on the efficiency of the amine to extract iron from the medium of the dissolution of Abu-Tartor phosphate in HCl, tridodecylamine (TDA) was used as an extracting agent. A series of experiments similar to those in the case of TOA were carried out; 0.05, 0.10 and 0.20 M of TDA in xylene solutions were used at 1 : 1, 1 : 2 and 1 : 3 ratios of aqueous acid filtrate. The results show that as the ratio between the amine and filtrate (aqueous) increases the efficiency of the amine to extract the iron increases considering all molarities of TDA amine in xylene (Table-3). The highest efficiency is observed at 0.1 M and 1 : 3 ratio where the reduction of the iron reaches 99.54%.

TABLE 3
THE PERCENTAGE OF THE RESIDUAL IRON IN AQUEOUS LAYER USING
0.2 M, 0.1 M AND 0.05 M TDA/XYLENE

Vol. of aq.	Vol. of org..	aq./org. ratio	Residual % Fe in aq. layer		
			0.2 M	0.1 M	0.05 M
10 mL	10 mL	1 : 1	4.10	2.03	24.4
10 mL	20 mL	1 : 2	2.88	0.65	2.19
10 mL	30 mL	1 : 3	2.74	0.54	1.22

By comparing this data with that obtained using TOA it is clear that in case of TOA, the efficiency increases by an amount equal to 1.59% and using 0.1 M instead of 0.2 M TOA. This may be explained according to the suggestion point out by Marcilla *et al.*,¹⁵ as they observed that when the length of the hydrocarbon chain of the amine increases, the solubility in phosphoric acid decreases, and therefore no losses in the amine and the trapped iron are observed.

Extraction of the phosphoric acid after treatment with amines

In this stage, after dissolution of the phosphate ore in HCl at the optimum conditions the following steps were carried out:

1. Filtration of the medium of phosphoric acid dissolution to remove the insoluble and suspended materials.
2. Removal of iron from the filtrate using TOA and TDA, each at the optimum ratio and concentration.
3. Extraction of the phosphoric acid with TBP or tert-amyl alcohol from the aqueous layer at their optimum conditions.
4. Stripping H_3PO_4 from TBP or tert-amyl alcohol with H_2O .
5. Stripping the iron content from the amine layer by H_2O , 5% Na_2CO_3 , 5% NaOH and 5% KOH.
6. Complete chemical analysis of the produced H_3PO_4 .

Following the previous six steps to extract pure phosphoric acid from the dissolution of Abu-Tartor phosphate ore in HCl (6 M), the results (Table-4) can be summarized as follows:

1. The treatment of phosphoric acid filtrate by (0.2 M TOA/xylene) causes a reduction in the iron content to 2.2% from the original value while the amine extracts about 5.70%, P_2O_5 at ratio 1 : 3 aqueous to organic layer (Table 4-1).

2. The extraction of phosphoric acid from the treated chloride medium from step 1 by TBP at ratio of 1 : 1 through three steps, shows that the efficiency of TBP to extract P_2O_5 reaches 65.56% (Table- 4-2).
3. The stripping of H_3PO_4 from the organic layer (H_3PO_4 in TBP) in three steps by H_2O as stripping agent at 1 : 2 aq./org. ratio gives an efficiency of the stripping equal to 93.5% whereas the iron content is reduced to reach about 0.38% of the original amount (Table-4-3).

TABLE 4
EXTRACTION OF THE ABU-TARTOR PHOSPHORIC ACID FROM THE MEDIUM OF DISSOLUTION BY 0.2 M TOA/XYLENE

(1) Pre-treatment of filtrate to remove iron by TOA

Vol. of aq.	Vol. of org.	aq./org. ratio	Residual % in aq. phase	
			P_2O_5	Fe
10 mL	30 mL	1 : 3	94.30	2.20

(2) Extraction of the phosphoric acid by TBP

No. of steps	Aq./org. ratio	P_2O_5 ext. % in org.
1st	1 : 1	31.10
2nd	1 : 1	21.60
3rd	1 : 1	12.86

Total $P_2O_5\%$ = 65.56%

(3) Stripping of the phosphoric acid by H_2O

No. of steps	aq./org. ratio	Percentage stripped	
		P_2O_5	Fe
1st	1 : 2		
2nd	1 : 2	93.50	0.38
3rd	1 : 2		

4. The stripping of iron from 0.2 M TOA/xylene layer by H_2O ¹⁶ at different ratios, 1 : 2, 3/2 : 1, and 3 : 1 aqueous to amine layer shows that the efficiency of H_2O extract iron increases from 45.50% at 2 : 1 ratio to 93.57% at ratio 3 : 1. Using 5% Na_2CO_3 ¹⁷ solution to strip iron from the amine layer at 1 : 2 ratio of amine to sodium carbonate, the efficiency of stripping becomes 77.97%, while using 5% KOH solution for stripping gives an efficiency equal to 86.42% at 1 : 1 ratio (Table-5). Complete chemical analysis of the obtained H_3PO_4 is shown in Table-6. The results of using T.D.A. in stripping iron and tributylphosphate for extracting pure H_3PO_4 by using H_2O , 5% Na_2CO_3 and 5% KOH for stripping Fe (Table-7) showed that:
 - (a) The efficiency of TDA to extract iron = 99.46%
 - (b) The loss in P_2O_5 = 7.80%
 - (c) The efficiency of TBP to extract P_2O_5 = 62.50%
 - (d) The stripping of H_3PO_4 from organic layer (H_3PO_4 /TBP) = 96.10%

(e) The stripping of iron from TDA/xylene layer by:

$H_2O = 94.79$ (1 : 3, org./aq).

5% $Na_2CO_3 = 80 : 33$ (1 : 3) org./aq. containing Na_2CO_3 .

5% $Na_2CO_3 = 74 : 80$ (1 : 3) org./aq. containing Na_2CO_3

5% KOH = 85.33 (1 : 3) org./aq. containing KOH

TABLE 5
STRIPPING OF IRON FROM 0.2 M TOA/XYLENE BY
(1) H_2O , (2) Na_2CO_3 and (3) 5% KOH

(1) H_2O			
Volume of amine	Volume of H_2O	Amine/ H_2O	Fe% stripped by H_2O
20 mL	10 mL	2 : 1	45.50
20 mL	30 mL	3/2 : 1	49.38
20 mL	60 mL	3 : 1	93.57
(2) 5% Na_2CO_3			
Volume of amine	Volume of Na_2CO_3	Amine/ Na_2CO_3 ratio	Fe% stripped by Na_2CO_3
20 mL	40 mL	2 : 1	77.97
(3) 5% KOH			
Volume of amine	Volume of KOH	Amine/KOH ratio	Fe% stripped by KOH
20 mL	20 mL	1 : 1	86.42

TABLE 6
THE CHEMICAL ANALYSIS OF THE EXTRACTED H_3PO_4 BY DIFFERENT SOLVENTS
AFTER TREATMENT WITH AMINE.

Component	Percentage	
	TOA/xylene + TBP + H_2O	TDA/xylene + TBP + H_2O
P_2O_5	18.850	18.620
F	0.330	0.360
SO_4^{2-}	1.880	1.750
SiO_2	0.022	0.020
Fe_2O_3	0.400	0.147
CaO	0.011	0.020
MgO	0.022	0.026

The obtained results indicate that using benzene as a diluent for the amine gives better results in the extraction of H_3PO_4 beside reducing the iron content to the lowest value (0.28%) than in case of using kerosene as a diluent. Also, formation of an emulsion was noticed in the case of kerosene.

TABLE 7
EXTRACTION OF PHOSPHORIC ACID FROM THE MEDIUM OF DISSOLUTION
USING 0.1 M TDA/XYLENE

(1) Dissolution				
Vol. of aq.	Vol. of amine	aq./amine ratio	Residual % in aq. layer	
			P ₂ O ₅	Fe
5 mL	15 mL	1 : 3	92.2	0.54
(2) Extraction of phosphoric acid by TBP at 1 : 3 ratio (aq./org.)				
No of steps		aq./org. ratio	P ₂ O ₅ % extr.	
1st		1 : 3	26.66	
2nd		1 : 3	19.20	
3rd		1 : 3	16.65	
Total P ₂ O ₅ % ext. = 62.5				
(3) Stripping of phosphoric acid by H ₂ O				
No. of steps	aq./org. ratio	Percentage stripped		
		P ₂ O ₅		Fe
1st	1 : 2			
2nd	1 : 2	96.10	0.103	
3rd	1 : 2			
(4) Stripping of iron				
By H ₂ O	(amine/H ₂ O = 1 : 3)		= 94.79	
By 5% KOH	(amine/KOH = 1 : 1)		= 85.33	
By 5% Na ₂ CO ₃	(amine/Na ₂ CO ₃ = 1 : 1)		= 74.80	
By 5% Na ₂ CO ₃	(amine/Na ₂ CO ₃ = 1 : 3)		= 80.33	

A complete study on the extraction of H₃PO₄ and iron content using TDA, TOA, DIPA, DEE (diethyl ether) and 20% DIPA/benzene was carried out and the results are shown in Table-8. From the obtained results of extraction, stripping and chemical analysis of the produced H₃PO₄, it may be concluded that:

TABLE-8
EXTRACTION OF PHOSPHORIC ACID AND IRON USING DIFFERENT SOLVENTS

Solvent	Vol. of aq.	Vol. of org.	aq./org. ratio	Residual % in aq. layer	
				Fe	P ₂ O ₅
0.1 M TDA	10 mL	30 mL	1 : 3	0.540	92.20
0.2 TOA	10 mL	30 mL	1 : 3	2.130	94.29
DIPA	10 mL	30 mL	1 : 3	11.094	83.34
MIBK	10 mL	30 mL	1 : 3	20.950	90.34
DEE (Diethyl ether)	10 mL	30 mL	1 : 3	81.780	95.00
DIPA	10 mL	30 mL	1 : 3	87.300	96.90

- Both TDA and TOA are good extractants to remove about 95% of iron content in crude phosphoric acid. It is worth noting that in the case of using the precipitation method in reducing the iron content, there is a loss of about 25% of P₂O₅ content when the iron content is reduced to about 50% from its original value. Hanna *et al.*^{3, 4} observed that the removing of the iron from phosphoric acid by precipitation depends on the nature

and amount of the precipitating agent. Also, they found that using solvent extraction to remove iron from wet-process, phosphoric acid is more expensive and leads to pollution of the environment. However, solvent extraction method by amines is still considered more efficient than ion exchange technique in removing iron, for the later method requires many pretreatment processes.

2. TDA is preferable to TOA in pretreatment of the filtrate from the dissolution of phosphate ore in HCl medium because it has low concentration (0.1 M/diluent). This may be attributed to the long chain of hydrocarbons which leads to high amounts of donor groups.
3. Benzene and xylene are more suitable diluents of amines compared to kerosene due to the formation of an emulsion layer which appears and creates some problems during the separation procedure using kerosene as a diluent.
4. 0.2 M TDA/xylene gives low efficiency compared to 0.1 M TDA/xylene during the removal of the iron content. This may be due to the low viscosity of the organic layer in the case of using dilute solution.
5. TBP is more efficient in extracting H_3PO_4 acid compared to TOA because TBP is less soluble in H_3PO_4 ; hence there is small loss in the solvent during the course of separation⁽¹⁸⁾.
6. The order of efficiency of the different solvents to extract H_3PO_4 acid is:
TAA > TBP > Bu-OH > DIPA
7. The order of efficiency of the different solvents to remove iron is:
TDA > TOA > DIPA > MIBK > Diethyl ether > (DIPA 20%).

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