PURIFICATION OF COMMERCIAL PHOSPHORIC ACID TO THE FOOD GRADE QUALITY BY LIQUID/LIQUID SOLVENT EXTRACTION

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ABSTRACT

Commercial phosphoric acid (45% P_2O_5), obtained from Polyserve Fertilizer Company, Egypt, was presently used. This work is divided into several steps, where it begins with the pretreatment step. In this step, desulphurization of phosphoric acid by phosphate ore addition is performed, then defluorination by the addition of sodium carbonate, which is followed by the removal of soluble and insoluble matter by activated clay addition, filtration and passing through carbon columns. Cations are removed by extraction with n-butanol. Different factors affecting the extraction, scrubbing and stripping steps have been studied. In the post treatment step, the purified acid (37% P_2O_5) is concentrated under vacuum to 85% P_2O_5 and then passed through activated carbon column to reach the water white color. The obtained purified acid is found to be in good agreement with the international standard specifications for food quality. Finally, on the light of the obtained results a technological flow sheet is elucidated.

INTRODUCTION

The necessity of very pure phosphoric acid has increased in recent years because of its progressive need in foodstuffs, animal feed additives, and liquid fertilizers. Solvent extraction processes have attracted great attention for wet process phosphoric acid purification and a considerable number of solvents have been studied as extractants, including the alcohols [**Bergdorf & Fischer (1978); Ruiz et al. (1978);**

Ruiz et al. (1985) and Guirguis et al. (2006)], mixed alcohols and ketones [Marco et al. (1988) and Ruiz et al. (1998)], ketones [Marcilla et al. (1993), a & b and Fek et al. (1994)], long chain tertiary amines [Stentrom et al. (1986) and Stentrom & Wingefors (1988)], ethers [Ruiz et al. (1986), a & b; Ruiz et al. (1987) and Marcilla & Ruiz (1989)] and others [Ahmed et al. (2006) and Myriam et al. (2006)]. Some patented processes are described in the literature by authors related to the R&D departments of commercial firms involved with either phosphoric acid processing or associated equipment [Mc Cullough et al. (1976); Bergdorf & Fischer (1978) and Ruiz et al. (1978)], whereas a complete review of the commercial purifying wet process phosphoric acid is given by the Phosphorous Derivatives under ISO 9002 Standards (1985).

EXPERIMENTAL, RESULTS AND DISCUSSION

A commercial wet process phosphoric acid from the Polyserve Fertilizer Company, Egypt, with 45% P_2O_5 is the starting material for this work. The acid is almost dark brown to black in color due to the presence of organic matter, besides varying amount of suspended mineral solids due mainly to $CaSO_4$ escaping from the filter and to precipitation from supersaturated hot acid during its cooling. Therefore, the acid is normally considered as an unstable physico-chemical system. The obtained chemical analyses of this acid are shown in Table (1). The purification process is divided into three main steps namely; pretreatment, solvent extraction, solvent regeneration for recycling and post treatment.

Acid pre-treatment

The pre-treatment operation involves an extensive preparation including cooling, desaturation, desulphurization, defluorination and clay treatment, beside, filtration and adsorption through activated carbon.

Cooling and desaturation of the acid

The phosphoric acid as received was left to stay during cooling for about 24 hours in a slowly agitated tank to allow all the supersaturated salts to precipitate.

Desulphurization

Four grams of phosphate ore are added to one liter of phosphoric acid to react with its free sulphuric acid giving insoluble calcium sulphate according to the following reaction:

$$10H_2SO_4 + Ca_{10}(PO_4)_6F_2 + 10H_2O \longrightarrow 10CaSO_4.nH_2O + 6H_3PO_4 + 2HF$$

Table (1): Complete chemical analyses of the used industrial phosphoric acid (In-Put acid)

Major and minor elements		Trace elements	
Component	Assay (%)	Elements	Assay (ppm)
P_2O_5	45.00	U	74
SiO_2	0.13	Th	2
CaO	1.92	Zr	15
Fe_2O_3	3.56	Zn	140
Al_2O_3	0.25	Cd	4
MgO	0.491	Cu	8
MnO	0.039	As	3
TiO_2	0.062	Sr	1300
Na_2O	0.380	Ba	505
K_2O	0.201	Mo	5
F^-	0.81	V	113
Cl^-	0.0016	Cr	124
SO_4^-	5.65	Pb	6
H_2O combined	22.1	Co	8
H_2O free	27.04	Ni	16
		*REE+Y	359
		B	0.05

(*REE+Y: Rare Earth Elements and Yttrium)

Defluorination

Sodium carbonate (0.5g) was found suitable to react with the fluorine present in one liter acid giving insoluble sodium fluorosilicate according to the following equation:

$$H_2SiF_6 + Na_2CO_3 \longrightarrow Na_2SiF_6 + CO_2 + H_2O$$

Clay treatment

Local activated clay. provided by a Ceramic Company, was used to trap most of the soluble humic matter present in the acid. A quantity of

2g/l was found to be quite adequate, where an optical density of 0.2 at a wavelength of 405nm has been obtained.

Adsorption through activated carbon

In order to remove the soluble humic matter and organic material. the filtered acid was allowed to pass through activated carbon columns (60cm in length and 2cm in diameter). Each column was packed with 15g of locally produced activated carbon supplied by EL-Nasr Company for Coke and Basic Chemicals. Table (2) shows the complete chemical analysis of the obtained green acid after pretreatment step.

Table (2): Chemical analysis of Polyserve phosphoric acid after pre-treatment (green acid).

Major and Minor Elements (%)		Trace Elements (ppm)	
Component	Assay	Component	Assay
P_2O_5	45.5%	U	74
SiO_2	*U.D.L	Th	2
MgO	0.20	Co	U.D.L
CaO	0.14	Pb	U.D.L
Fe_2O_3	1.22	Be	U.D.L
Al_2O_3	0.30	Ba	U.D.L
Na_2O	0.05	Sr	30
K_2O	0.03	B	0.02
MnO	0.03	V	80
TiO_2	0.01	Cd	4
F^-	0.005	Cu	8
Cl^-	U.D.L	As	2
SO_4^-	0.10	Sr	30
H_2O combined	22.9	Mo	5
H_2O free	28.03	Ni	16
		Total REE	300
		Zr	5
		Zn	140

*U.D.L : Under Detection Limit

Solvent extraction

The process of solvent extraction is divided into three main steps namely; extraction. scrubbing and stripping besides solvent regeneration. Different solvents have been tested and indeed used commercially to extract phosphoric acid as shown in Table (3). It is clear that both acetone and methyl isobutyl ketone are the most promising solvents to extract the phosphoric acid. However, they are miscible with water and they have to be recovered by distillation. Moreover, the fraction percent of rejected component impurities is smaller than that of n-butanol. A compromise should be made between ease of separation, solubility of the solvent with the acid. stability of solvent, selectivity and extraction efficiency. Further experiments were carried out on n-butanol only, because it has lower solubility and higher stability. The relevant factors affecting the extraction process are studied below.

Table (3): Solvents tested for the extraction of phosphoric acid (45.5 % P_2O_5)

Solvents	P_2O_5 Extraction (%)	Fraction (%) of Rejected Components			
		Fe_2O_3	Al_2O_3	MgO	U
N-butanol	69	85	95	75	75
Iso-butanol	61	80	85	70	65
1-Heptanol	40	100	100	99	80
Acetone	80	55	55	43	50
Methyl isobutyl ketone (MIBK)	75	65	70	65	70
Tri-normal butyl phosphate (TBP)	52	75	78	69	60
TBP + isobutyl ketone	69	80	85	70	62
TBP + butanol	58	82	93	70	66

Effect of contact time

The effect of contact time on the attainment of equilibrium has been studied for the time intervals from 5 to 40 minutes and illustrated in (Fig. 1). It is obvious that a contact time of 20 minutes is quite adequate.

Effect of settling time

This effect was studied at constant agitation speed of 3000rpm at an initial time of 1, 3, 5, 7 and 10 minutes. It is clear from (Fig. 2) that a settling time of 5 minutes is quite reasonable.

Effect of temperature

For studying the effect of temperature on the distribution coefficients. which reflects its results on the percentage of P_2O_5 extraction from phosphoric acid, experiments were carried out using an electric thermostat, the temperature of which is regulated from $5^{\circ}C$ to $85^{\circ}C$, where the other extraction factors were kept constant at the obtained optimum value. It is clear from (Fig. 3) that temperature has little effect on the extraction results between $5^{\circ}C$ and ambient temperature. The reason for the decrease of the P_2O_5 extraction at higher temperatures between $45^{\circ}C$ and $85^{\circ}C$ could be due to the solvent dissociation or formation of different complexes. For practical reasons, room temperature ($25^{\circ}C$) could be used.

Effect of initial P_2O_5 concentration

Three different initial P_2O_5 concentrated samples were obtained from Polyserve Fertilizers Company, Egypt, namely: 30%. 45% and 56% P_2O_5. Each of these samples was pretreated to the green acid as mentioned before. The results obtained are given in Table (4). In general, P_2O_5 extraction increases with an increase in the initial P_2O_5 concentration. However, we used the 45.5% P_2O_5 acid concentration, as it is the one produced on a large scale by the company.

Effect of organic/aqueous phase ratio

The same adopted technique for the general extraction was followed at the optimum values previously determined except that the organic/aqueous phase ratio (w/w) was varied from 1:3 to 3:1. The results shown in (Fig. 4) indicate that the best organic to aqueous phase ratio is 1:1.
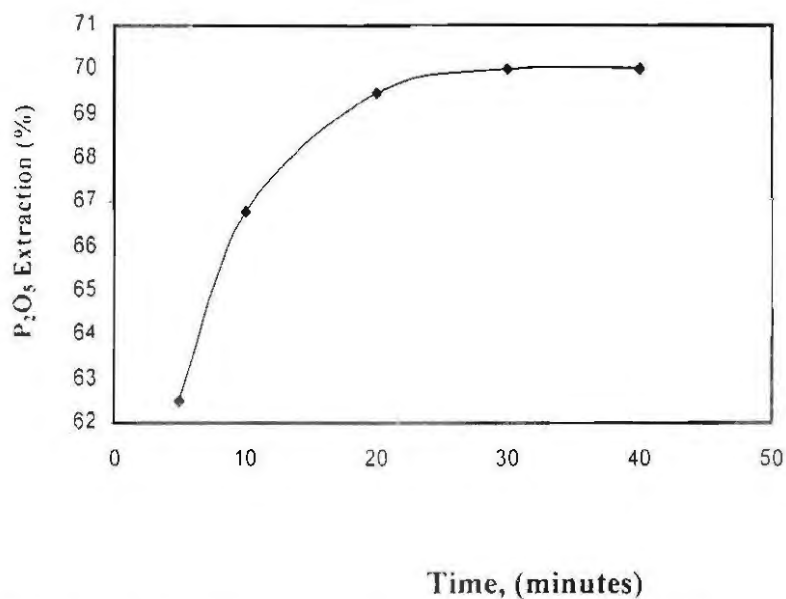
Time, (minutes)

Fig. (1): Effect of contact time on the extraction of P_2O_5 from phosphoric acid.

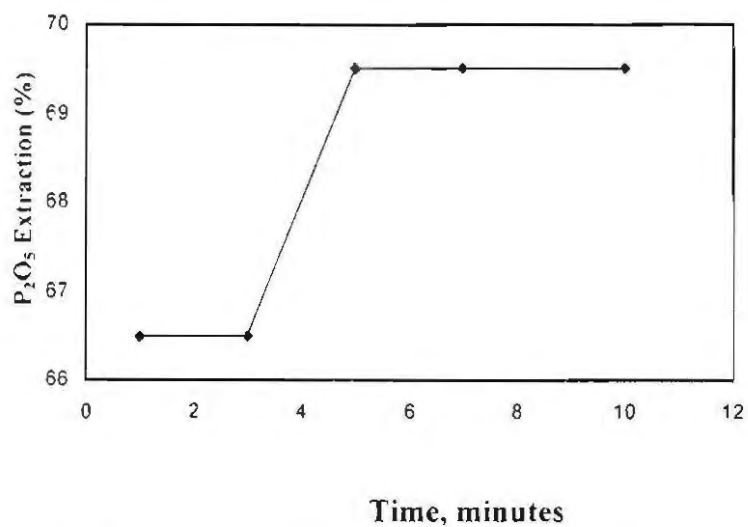

Time, minutes

Fig. (2): Effect of Settling Time on Extraction of P_2O_5 from phosphoric acid.
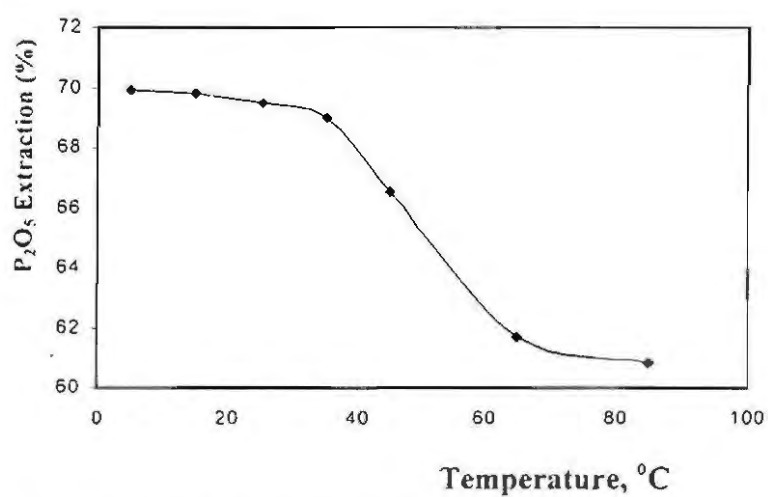
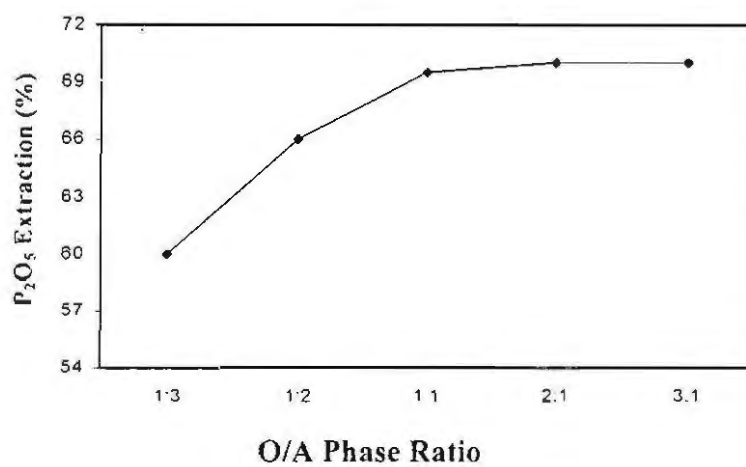**Fig. (3):** Effect of temperature on extraction of P_2O_5 from phosphoric acid.

Fig. (4): Effect of organic/aqueous phase ratio on the extraction of P_2O_5 from phosphoric acid.

Table (4): Effect of acid concentration on the extraction
of P_2O_5 from phosphoric acid

Acid Concentration, %	P_2O_5 Extraction, %
30	64
45	69.5
56	74

Experimental procedure

A mixture of 50g of the acid and 50g of the solvent was stirred vigorously with a magnetic stirrer and a thermostat, to control the temperature, for 20 minutes in a 400ml beaker. The mixture is then transferred to a separating funnel and allowed to settle for 5 minutes. The aqueous and the organic phases were separated, their weights and volumes were recorded and the sample of the aqueous phase was analyzed. Due to the mutual solubility of the alcohols and water there were great changes in the volumes of the organic and aqueous phases after equilibrium; this was taken into consideration when evaluating the results. Some tests were conducted using alcohol saturated with water to minimize changes in volume. In fact, the extraction coefficient is reflected on the P_2O_5 extraction percentage according to the following equation:

$$\%E = 100D\,(P) / (1+P)\,D, \text{ where;}$$

P (phase ratio) = volume of organic / volume of aqueous
D (extraction coefficient) = $[P_2O_5]_{organic} / [P_2O_5]_{aqueous}$

Distribution isotherm

A distribution isotherm is a plot of the equilibrium concentration of the extracted species in the extracted phase against its concentration in the raffinate layer at a given temperature. Distribution isotherm can be prepared for either the extraction or the stripping process. The former is called extraction isotherm and the latter is stripping isotherm. Data for the extraction isotherm can be easily obtained from either single contact of a fixed volume of aqueous feed (phosphoric acid) with different volumes of organic feed (n-butanol) or else by repeated contact of one and the same aliquot of the fresh organic feed (n-butanol) of equal volume.

Mc-Cabe Thiele diagram is a composite plot of the distribution isotherm and the operating line. The latter could readily be established by only one point, which corresponds to the final raffinate composition and the ratios of the aqueous to organic phases that determines the slope at the line, as it is a straight line. On constructing the diagram, the stages are stepped off by extending a horizontal line from the vertical extremely of the operating line to intersect the isotherm, then a vertical intercept to the operating line and so on until the other extremely of the operating line is intersected. The diagram can be used to approximate the number of theoretical stages required for a given process. Another importance of Mc-Cabe Thiele diagram is in evaluating the extraction results. It is clear from (Fig. 5) that four theoretical stages are quite adequate for P_2O_5 recovery, if the operating line is at an O/A phase ratio equals 1:1.

Scrubbing Process

Washing the organic phase (loaded solvent) containing purified phosphoric acid after extraction is carried out to eliminate metal impurities where water, water saturated butanol or an aqueous phosphoric acid of greater purity than the wet phosphoric acid can be used. In this study, aqueous phosphoric acid after stripping (as given herein after) is used for this purpose at a phase ratio of O/A of 40:1 for 20 minutes at room temperature [**Berkowitz et al. (1982)**]. As the concentration of phosphoric acid used for scrubbing is the same as that of the purified acid product, small amounts of P_2O_5 were transferred between the organic phase and the scrubbing phosphoric acid.
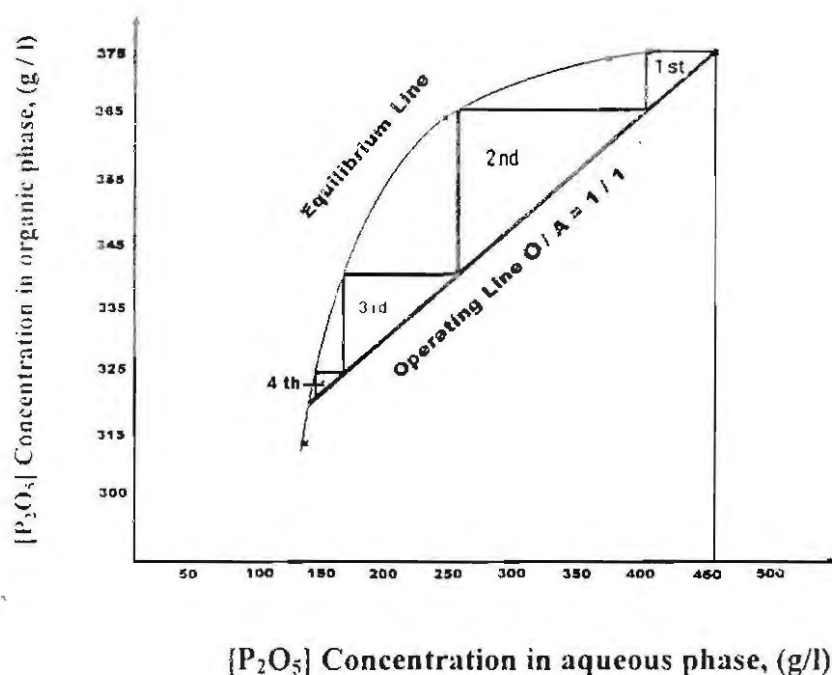
[P$_2$O$_5$] Concentration in aqueous phase, (g/l)

Fig. (5): Mc-Cabe Thiele diagram for P$_2$O$_5$ extraction.

Stripping Process

Stripping is the removal of the extracted solute from the organic phase, wherein the solute is obtained in an enriched and purified form and the solvent is simultaneously prepared for re-use (by distillation in the present study).

Effect of different stripping reagents

Five different reagents namely phosphoric acid, water contains salts of alkali metal carbonate or hydroxide, ordinary distilled water and water saturated butanol were tried to find out the most efficient reagent for the stripping process at an O/A phase ratio of 3:1, room temperature, 30 minutes contact time and 5 minutes settling time. It is clear from the obtained results shown in Table (5) that distilled water can be used for the stripping process since it is the cheapest and there will be no contamination from the added reagents.

Table (5): Effect of different reagents on the stripping process

Reagents	% S of P_2O_5
Phosphoric acid	79.2
Water contains sodium salts	77.5
Water contains 5% NaOH	78.6
Water saturated butanol	79.7
Distilled water	80

Effect of contact time on the stripping processes

The effect of contact time on the attained equilibrium state has been studied at intervals from 5 to 40 minutes. The other factors are kept at O/A phase ratio of 3:1. settling time 5 minutes and the experiments were carried out at room temperature. It is obvious from Table (6) that a contact time of 30 minutes is sufficient.

Table (6): Effect of contact time on the stripping process

Time, minutes	% S of P_2O_5
5	70
10	73.5
20	75
30	80
40	80

Effect of settling time on stripping process

The effect of settling time on P_2O_5 stripping from the organic phase was studied between 1 to 10 minutes while the other factors were kept at the optimum values. From the results shown in Table (7), a settling time of 5 minutes is satisfactory.

Table (7): Effect of settling time on the stripping process

Settling Time, minutes	% S of P_2O_5
1	72.6
3	73.4
5	80
7	80
10	80

Effect of temperature on the stripping process

It is clear from Table (8) that as the temperature increases from $25^{\circ}C$ to $85^{\circ}C$, the P_2O_5 stripping value decreases. So, room temperature could be used.

Table (8): Effect of temperature on the stripping of P_2O_5 process.

Temperature, ^0C	S %, P_2O_5
5	76.5
15	76.5
25	80
35	78.5
45	70.6
65	66.4
85	60

Effect of organic/aqueous phase ratio on the stripping process

Different O/A phase ratios from 1/3 to 4/1 (w/w) were used to get the optimum phase ratio for P_2O_5 stripping from the organic phases. The other factors are fixed at contact time 30 minutes, settling time 5 minutes, the acid concentration is 45.5% P_2O_5 and experiments are carried out at room temperature. From (Fig. 6) it is clear that the best organic to aqueous phase ratio is 3:1.
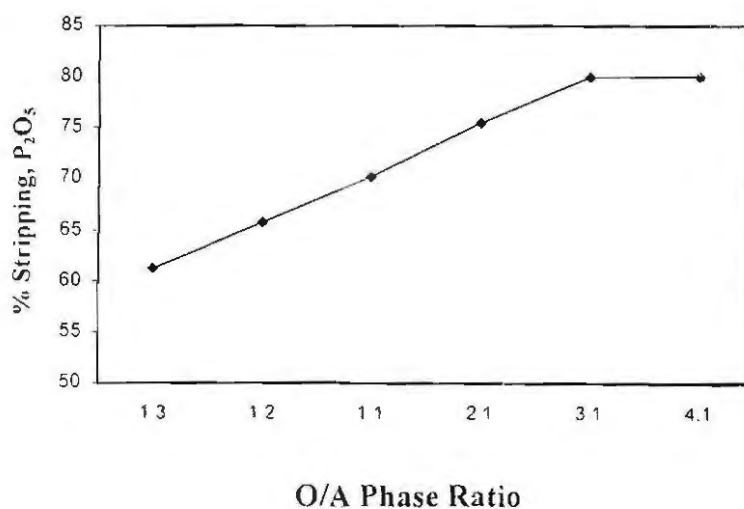

O/A Phase Ratio

Fig. (6): Effect of O/A phase ratio on the stripping of P_2O_5 process.

Equilibrium line and construction of Mc-Cabe Thiele diagram for the stripping process

It is clear from (Fig. 7) that four stages were found sufficient for the stripping of P_2O_5 from the organic phase at an organic to aqueous phase ratio of 3:1. A complete chemical analysis of the purified acid together with the start material (green acid) and the raffinate is shown in Table (9).
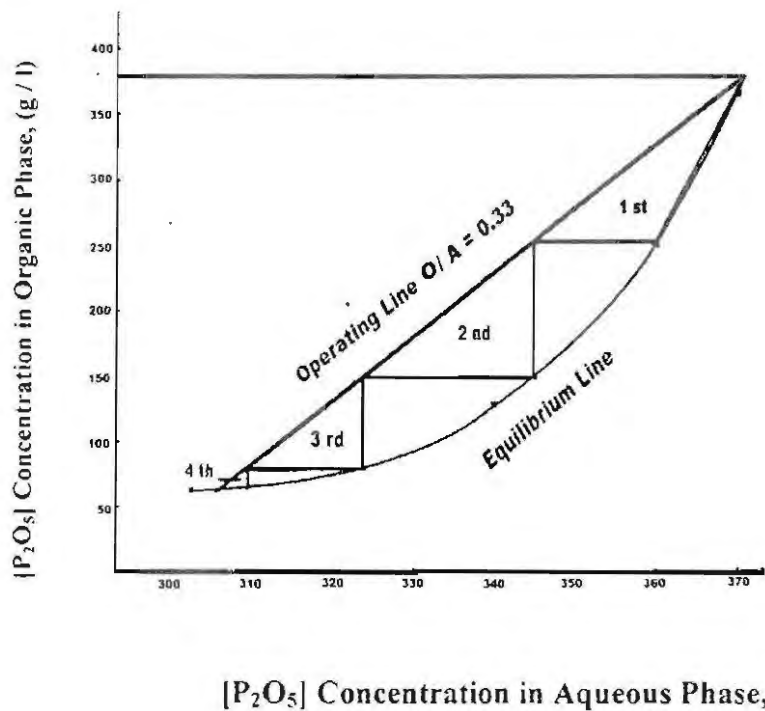

[P_2O_5] Concentration in Aqueous Phase, (g/l)

Fig. (7): Mc-Cabe Thiele Diagram for P_2O_5 Stripping.

Table (9): Purified acid analysis together with raffinate.

Component	Green Phosphoric Acid Before Purification	Phosphoric Acid After Purification	Raffinate
P_2O_5	45.5%	37%	7.5%
SiO_2	U.D.L	U.D.L	U.D.L
MgO	0.2%	U.D.L	0.2%
CaO	0.14%	42 ppm	0.13%
Fe_2O_3	1.22%	70 ppm	1.21%
Al_2O_3	0.3%	U.D.L	0.3%
Na_2O	0.05%	4 ppm	0.046%
K_2O	0.03%	5 ppm	0.015%
MnO	0.03%	6 ppm	0.024%
TiO_2	0.01%	U.D.L	0.01%
F^-	0.005%	U.D.L	0.005%
Cl^-	U.D.L	U.D.L	U.D.L
SO_4^{2-}	0.1%	5 ppm	0.099%
U	74 ppm	U.D.L	74 ppm
Co	U.D.L	U.D.L	U.D.L
Th	2 ppm	U.D.L	2 ppm
Pb	U.D.L	U.D.L	U.D.L
Be	U.D.L	U.D.L	U.D.L
Ba	U.D.L	U.D.L	U.D.L
Sr	30 ppm	U.D.L	30 ppm
B	0.02 ppm	U.D.L	0.02 ppm
V	80 ppm	U.D.L	80 ppm
Cd	4 ppm	0.02 ppm	3.8 ppm
Cu	8 ppm	0.9 ppm	7 ppm
As	2 ppm	U.D.L	2 ppm
Mo	5 ppm	U.D.L	5 ppm
Ni	16 ppm	U.D.L	16 ppm
$\sum$ REE	300 ppm	U.D.L	300 ppm
Zr	5 ppm	U.D.L	5 ppm

U.D.L: Under Detection Limit; REE: Rare Earth Elements

Post Treatment of the Purified Phosphoric Acid

The obtained purified acid after solvent extraction is 37% P_2O_5. accordingly it is concentrated by evaporation under reduced pressure to a P_2O_5 content in the range from 85% to 86% P_2O_5. The obtained concentrated phosphoric acid is passed through activated carbon column at a flow rate of 60ml/h to remove any residual contaminants.

The produced acid is water colorless and odorless and in good match with the international specifications for food and analar quality acid as shown in Table (10). It is worth mentioning in this regard that complete ·decolorization of purified wet process phosphoric acid is achieved when the transmission of phosphoric acid against distilled water in 5cm cells at 365nm is greater than 80% [Import & Export Co. Ltd. (2001)]. From Lambert-Beer law a transmission of 80% and 90% measured in a 5cm cells correspond to a transmission of 95.6cm and 97.6% respectively measured in 1cm cell. On the other hand, the West German additives control regulation of July 10th. 1984, Bundesg establish No. 30, 8897 to 901, states that the contents of the volatile acid calculated as acetic ·acid· must be less than 10ppm [Schimmel et al. (1990)]. Our prepared acid got a value of 3ppm. ·

Table (10): Chemical analysis of the purified phosphoric acid together with the international standards samples.

Elements	Purified Phosphoric acid, Present Work	International Standard Sample of Chemically Pure Acid|Import & Export Co. Ltd. (2001)		International Sample of Food Grade Purity Acid|Import & Export Co. Ltd. (2001)	
Purity	85.5	85 max	85.5typical	85max	85.3 typical
Color	10	20 max	10 typical	30 max	20 typical
Cl⁻	U.D.L	5 max	1 typical	5 max	1 typical
SO_4^{2-}	10	50max	30 typical	50 max	30 typical
F⁻	U.D.L	10 max	3 typical	20 max	3 typical
As	U.D.L	1 max	0.3 typical	50 max	40 typical
Heavy metal as Pb	4 ppm	10 max	5typical	10 max	5 typical

Industrial Grade Wet Process Phosphoric Acid

↓

P_2O_5 (45%), U (74ppm)

		24 hours
Supersaturated salts ←	**Cooling and Desaturation**	←
Sulphate ←	**Desulphurization**	← 4g phosphate ore
Prepurification ← Fluoride ←	**Defluorination**	← 0.9g Na_2CO_3
Hummic matter ←	**Clay Treatment**	← 2g/l $CaSO_4$
Organic matter ←	**Carbon Column**	← 60 ml/h

↓

Green Phosphoric Acid
45.5% P_2O_5 75ppm U

↓

		N-butanol O/A=1
Solvent recycle Organic phase	**Solvent Extraction** P_2O_5 (38%), U (undetected)	← Room Temperature
Purification ← Impurities ←	**Scrubbing**	← o-phosphoric acid (O/A =40), room temp.
Solvent Distillation —	**Stripping**	← Distilled water (O/A =3), room temp.

↓

Purified Phosphoric Acid
(37% P_2O_5)

↓

		Under vacuum
Post Purification ←	**Concentration**	←
	Carbon Column	← Flow rate 60ml/h

↓

Analar and food grade purified phosphoric acid 85.5% P_2O_5
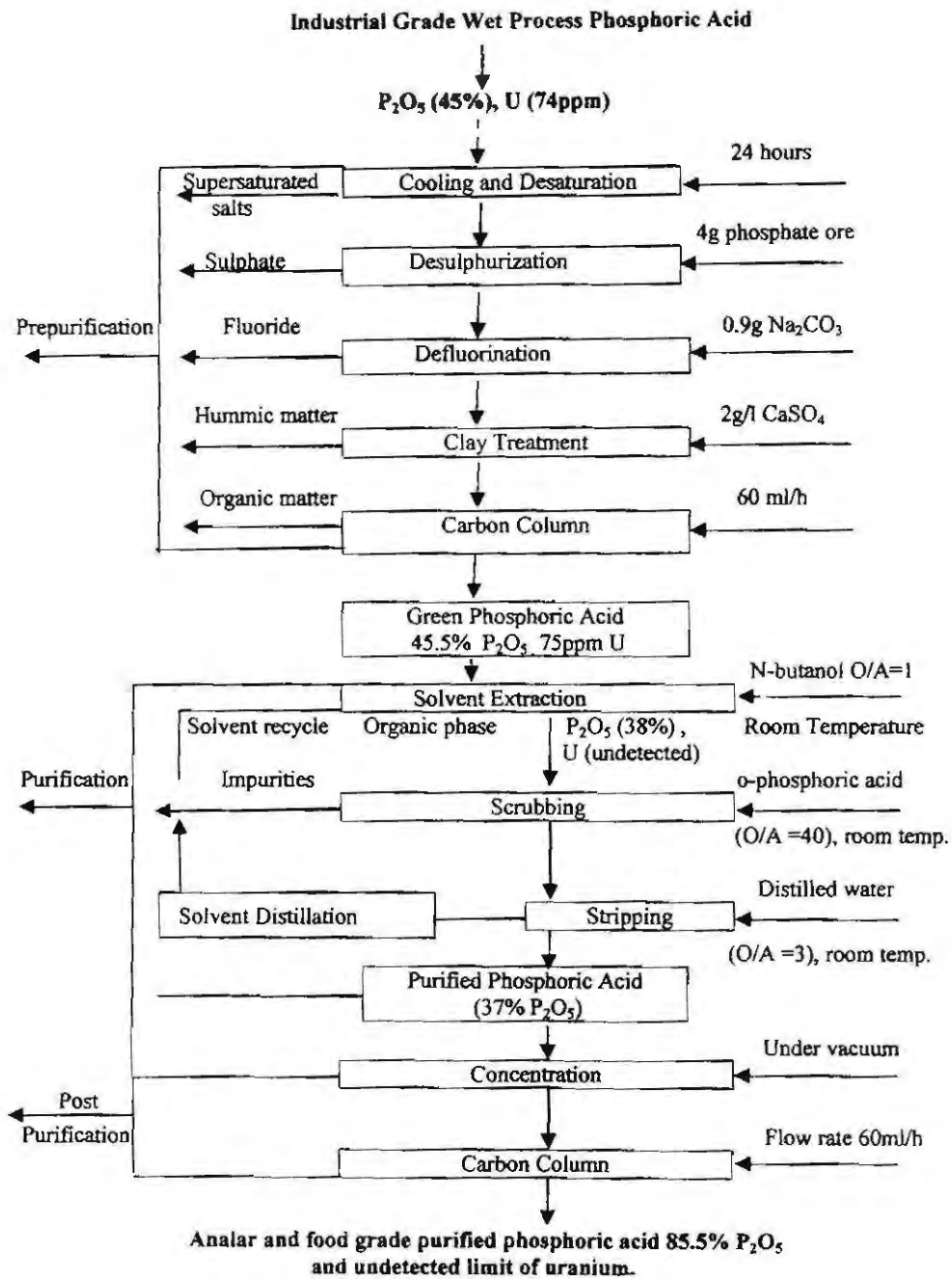**and undetected limit of uranium.**

Fig. (8): Proposed technical flow sheet for purification of industrial grade wet process phosphoric acid to analar and food grade quality.

CONCLUSIONS

The previous results show that:

1. The solvent is stable and selective to phosphoric acid; extraction of uranium and heavy metals are negligible. The bulk of impurities including uranium are left behind in the raffinate from which it can be recovered. However. fluorosilicic acid is co-extracted together with H_3PO_4. so it is advisable to do the pretreatment step before extraction.

2. The concentration of n-butanol has a positive effect on the extraction. so the solvent is used without diluents.

3. The concentration of phosphoric acid has a positive effect on the extraction. so it is recommended to use high strength phosphoric acid.

4. The temperature has a small effect on the extraction in the range studied. so extraction could be carried out at room temperatures.

5. The suggested flow sheet. Fig (8). and the solvent used allow the preparation of purified phosphoric acid of various grades. fertilizers. chemical, animal feed. food quality and analytical acid. This is very important as one can produce the quality of acid needed in the market.

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تنقية حامض الفوسفوريك التجارى الى درجة الأستخدام الغذائى بواسطة الأستخلاص
بالمذيبات بالطريقة السائلة

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هيئة المواد النووية، ص ب ٥٣٠ المعادى، القاهرة

يختص البحث الحالى بدراسة تنقية حمض الفوسفوريك إلى جودة الأستخدام الغذائى،
مبتدا من المنتج التجارى (٤٥% خامس أكسيد الفوسفات من إنتاج شـركة بـولى سـرف
للأسمدة– أبوزعبل– القاهرة– مصر). وقد تم تقسيم العمل إلى عدة خطوات تبدأ بالمعالجـة
الأولية للحامض حيث يتم ازالة الكبريتات باضافة خام الفوسفات منفـاعلا مـع حـامض
الكبريتيك لينتج كبريتات الكالسيوم غير الذائبة. وفى الخطوة التالية يتم التخلص من الفلورين
بإضافة كربونات الصوديوم، التى يستتبعها ازالة المواد الذائبة وغير الذائبة باضافة الطمـى
النشط، ثم التمرير خلال اعمدة الكربون. يتم تنقية حامض الفوسفوريك الأخضر فى الخطوة
التالية بطريقة الاستخلاص بالمذيبات العضوية المختلفة مثل ن–بيوتانول. وفى هذه الخطـوة
تنقسم عملية الاستخلاص بطريقة التنقية إلى ثلاث مراحل مختلفة وهى الاستخلاص، عمليـة
غسل المذيب ثم الاسترجاع. وأخيرا يتم تركيز الحامض المنقى (الذى يحتـوى علـى ٣٧%
من خامس أكسيد الفوسفور) بالبخر تحت ضغط منخفض ليعطى الحمض المركز(٨٥%خامس
أكسيد الفوسفور) الذى يمرر بدوره خلال عامود كربون نشط لإزالة أى مواد عضوية متبقية،
معطيا حامض عديم اللون والرائحة الذى وجد أنه متوافق بصورة جيـدة مـع المواصـفات
العالمية القياسية لحامض الفوسفوريك المستخدم للغذاء. وأخيرا فانه بناء على ما تم الحصول
عليه من نتائج فقد تم وضع وصف للوحة تشغيل تكنولوجية لتسليط الضوء على النتائج التـى
تم الحصول عليها.