

# IFA Technical Conference

Marrakech, Morocco

28 September-1 October 1998

## OPERATING EXPERIENCE OF PROCESSING LOW GRADE JORDANIAN PHOSPHATES IN THE MANUFACTURE OF WPA PRODUCTION<sup>1</sup>

K. Halaseh and H. Dukhgan

Jordan Phosphate Mines Company Ltd., Jordan

### SUMMARY

This article focuses on the production of wet process phosphoric acid (WPA) using different blended low grades of Jordanian phosphates. Studied the impact of F, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> contents in raw materials on the specific filtration capacity (SFC), qualities of downstream products, calcium sulfate (CS) crystals and P<sub>2</sub>O<sub>5</sub> recovery. The aim of the rock impurities study is to obtain data on the influence of F, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> on the SFC in WPA production and also to optimize their contents in blended 66-68% BPL grade. Studied influence of inorganic modifiers on the SFC when using low SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> phosphate and evaluate different types of filter cloths used for PA production. Investigate the effect of slurry temperature, permeability, mechanical strength and the tonnage of P<sub>2</sub>O<sub>5</sub> produced on the cloth lifetime.

### RESUME

*Cet article se focalise sur la production d'acide phosphorique de voie humide, utilisant des mélanges de phosphates jordaniens à bas titre. On étudie l'impact des teneurs en F, Al<sub>2</sub>O<sub>3</sub> et SiO<sub>2</sub> dans les matières premières sur la capacité spécifique de filtration (CSF), les qualités des produits d'aval, les cristaux de sulfate de calcium et le rendement en P<sub>2</sub>O<sub>5</sub>. Le but de l'étude des impuretés du phosphate est d'obtenir des données sur l'influence de F, Al<sub>2</sub>O<sub>3</sub> et SiO<sub>2</sub> sur le CSF dans la production de l'acide phosphorique et aussi pour optimiser leurs teneurs dans les mélanges à 66-68 % BPL. On étudie l'influence de modificateurs inorganiques sur le CSF quand on emploie des phosphates pauvres en SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> et on évalue différents types de toiles filtrantes utilisées pour la production de l'acide phosphorique. On étudie l'effet de la température, de la perméabilité, de la résistance mécanique et du tonnage de P<sub>2</sub>O<sub>5</sub> sur la longévité de la toile.*



### Introduction

Aqaba Industrial Complex has the following international scale process and production facilities:

- SA: Monsanto Four Beds - Double Conversion, 1.485.000 MTPY.
- PA: Dihydrate Rhone Poulenc & Prayon Processes, 432.000 MTPY.
- DAP: Gulf Process, Badger INC, 800.000 MTPY.
- ALF<sub>3</sub>: Alusuisse, 20.000 MTPY.

PA Plant (PAP) started life as a Rhone-Poulenc dihydrate unit, originally built in the beginning of 1980's with a capacity of 1250 MTPD P<sub>2</sub>O<sub>5</sub>, designed to operate on 73 - 75% BPL Jordanian phosphate [1].

The plant is characterized by a large table filter with an effective area of 205 m<sup>2</sup> and single compartment tank reactor with central agitator having rotation speed of about 31 rpm and power absorbed 0.52 kw/m<sup>3</sup> slurry. The specific reaction volume (SRV) is 1 m<sup>3</sup> /tpd P<sub>2</sub>O<sub>5</sub> thus, the reactor would have a holding time of about 165-170 minutes and maintaining a crystallization rate of calcium sulphate at level 0.22-0.24 t/m<sup>3</sup>hr. 80% SA is introduced onto a disc on the shaft of a surface cooler and is thus sprayed across the reactor surface together with the droplets of slurry. The reactor slurry temperature is kept 82.5°C by means of drawing air across the surface of the reactor. Moreover, maximum feed rate to reaction volume ratio is 0.14 with overall plant efficiency of about 95±0.5%. It has been established, that with smaller SRV higher supersaturation will take place, higher nucleation rate will occur. This reduces crystal sizes and thus filtration quality [ 2 ].

In 1993 major debottle-necking in the reaction, concentration and as well as cooling pond sections were undertaken which has substantially increased capacity to 1310 MTPD .

---

<sup>1</sup> *Expérience opératoire dans le traitement des phosphates jordaniens de différents titres dans la fabrication d'acide phosphorique*

For a detailed review of PAP revamp see Rehabilitation of JPMC - PAP in Aqaba IFA Technical Conference 1994 Amman - Jordan [3].

Figure 1 is a flow sheet showing the dihydrate PAP at Aqaba complex.

The revamp included the addition of three digesters and a separate Prayon II compartment reactor with two 98.5% sulfuric - recycled acids mixers; a low level flash a cooler and axial flow circulator creating a very high rate of slurry circulation 8500 m<sup>3</sup>/h across the LLFC itself as well as throughout the reactors, so as to maintain a temperature of 76-78°C in the reactor and 74-75°C at the filter. All these provide numerous benefits to JPMC including relatively high phosphate rock processing flexibility.

The total reaction volume, nominal feed rate to reaction volume ratio and SRV are 2618 m<sup>3</sup>, 0.068 and 2 m<sup>3</sup>/tpd P<sub>2</sub>O<sub>5</sub> respectively. The latter is equivalent to some 6 hours retention time. Becker [4] reported, that when the SRV is in this case within the range 1.8 to 2 m<sup>3</sup>/tpd P<sub>2</sub>O<sub>5</sub>, ideal conditions for crystallization are achieved at 0.09-0.125 t/m<sup>3</sup> hr. However, the average energy consumption of the total reaction tanks increased to some 0.62 Kw/ m<sup>3</sup> slurry.

Beside, the rock grind required for Rhone - Poulenc process works on a specification of 75% passing 200 mesh initially but this specification was changed to 50% minus 100 mesh.

A further important point to note is that , the plant has excellent production results and efficiencies even operating above design rate. The annual global efficiency is within the range of 95-95.12%.

Finally, the PAP modification was changed from a single reactor dihydrate Rhone-Poulenc process route to a multicompartment reactor and three digesters of **Prayon Mark IV** dihydrate process.

## 1. Quality of Jordanian phosphates

Essentially, the BPL percentage determines the price of phosphate rock but the quality of phosphate cannot be measured in terms of the P<sub>2</sub>O<sub>5</sub> content or BPL grade. The type of impurities and their effect on the downstream chemical process are far more important than the concentration of P<sub>2</sub>O<sub>5</sub> in the plant operation and final PA production. As example, CS filterability is a major economical factor when selecting commercial phosphate rock, since filtration is the most critical and expensive step in the process [4,5].

The author [6] studied the effect of impurities on WPA production and comparative evaluation of Jordanian phosphate rock. Others [7] reported the flexibility in phosphate flowsheets not only to fulfill the needs of traditional and international customers but also to meet the specific requirements of individual plants.

On the bases of these studies, conclusively, have proved that Jordanian phosphate is soft, porous and friable rock of marine sedimentary origin having high surface area (16-22cm<sup>2</sup>/g). Chemical composition of various grades of the phosphate ores is given in Tables 1 and 2. As seen, Jordanian phosphates can be considered as a high quality and flexibility ranging started from low-grade 65 up to high grade 76.5% BPL [8,9]. The presence of impurities such as **Fe<sub>2</sub>O<sub>3</sub>**, **SrO**, **organic matters** and **rare earth element (Ln<sub>2</sub>O<sub>3</sub>)** are quite low.

The level of CO<sub>2</sub> is within the range of 3 to 4.8%, which gives an optimum rate for sedimentary phosphates in low foaming tendency. The content of alkaline oxides does not exceed 0.65%. Further, the MgO content of 0.2-0.3% is low enough not to cause problems with high acid viscosity and reduce filtration rate .

In terms of the potentially hazardous elements, (Table 2), Jordanian phosphates are characterized by low levels of **Cd, Pb, As, Mo, Cu, Mn, Cr, V** and the absence of Hg.

Taking PA technology into the consideration the quality of the Jordanian phosphate rock used can be considered satisfactorily performing by three key factors:

- High rate of slurry filtration,
- Acceptable level of SA consumption,
- Low rate of corrosion and abrasion.

The major factors affecting the above mentioned are:

### **F, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Cl and CaO/P<sub>2</sub>O<sub>5</sub> Ratio**

This article is a sequel to a previous work presented at the AFA Technical Conference - Jordan 1996 [10]. It presents additional information and data generated in the operation of Jordanian low grade phosphates at Aqaba PAP in recent years.

Table 1 shows the presence of fluorine and chlorine contents in phosphate ores are within the range 3.5 to 4.0% and 200 to 650 ppm respectively, whereas SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> contents fluctuates from very low of 1.95 up to 10.16%. Moreover, CaO/P<sub>2</sub>O<sub>5</sub> ratio ranges 1.457 to 1.64.

More than 16 years of extensive plant experience based on industrial scale experiments which have been systematically conducted at Aqaba PAP using various grades of phosphate have shown that the plant capacity and P<sub>2</sub>O<sub>5</sub> recovery rate is directly linked to the level of these impurities. Moreover, the extremely wide range of fluctuating causes affect production results, equipment's conditions and processing characteristics.

So the objectives of the rock impurities studies are to obtain data on the influence of F, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> on the SFC in WPA production and also to optimize their contents in blended 66-68% BPL grade in order to keep production rate, product specification, equipment's status within almost acceptable tolerances.

## **2. Influence of F, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> Contents**

The inorganic elements in sedimentary rock that are well known to affect considerably the crystal habit and size of CaSO<sub>4</sub> are F, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and to a certain extent MgO.

Becker [5] mentioned that these impurities in WPA, are of particular importance in that, they combined and formed complexes with fluorine ions which affect CaSO<sub>4</sub> crystallization.

However, a study of published literature [11,12,13] shows that, the presence of free fluorine ions leads to the formation of long thin needle crystals, which are difficult to filter. Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> are beneficial to crystallization because they reduce the free fluorine ions concentration and improve the form of CaSO<sub>4</sub> crystal. Moreover, it has been reported [4,11,14] that the presence of active silica has a positive effect on the reduction of corrosion and crystallization of CaSO<sub>4</sub>. By changing F/SiO<sub>2</sub> ratio, needle - type crystals can be modified to agglomerates. The best crystals look like balls, because of their surface to volume ratio is at minimum [4].

As reported by Kopilev [15], a high concentration of Al<sup>3+</sup> ions in slurry liquid phase regulates the CaSO<sub>4</sub> crystal growth uniformly in all direction yielding thicker crystals, with low surface - to volume ratio. It can also can promote the formation of cluster type crystals, which has a significant effect on CaSO<sub>4</sub> filterability [4,12]. Karmishov [16] found that the optimum condition for dihydrate CaSO<sub>4</sub> crystallization occurs when maintaining the level of free SO<sub>4</sub><sup>2-</sup> 3.5%, SiF<sub>6</sub><sup>2-</sup> 1.5% and Al<sub>2</sub>O<sub>3</sub> 0.8%.

As reported by researchers [5,17], displaying the effect of Al<sub>2</sub>O<sub>3</sub> and F on crystallization is explained with formation especially aluminum fluorides complexes type AlF<sub>3</sub><sup>x-</sup> where x has value ranges between 0 to 6 according to molar ratio Al/F dictates which type will be dominant. AlF<sub>5</sub><sup>2-</sup> is existing and can be substituted SO<sub>4</sub><sup>2-</sup> in the gypsum lattice. The solubility of these complexes depend on P<sub>2</sub>O<sub>5</sub>, free SO<sub>4</sub><sup>2-</sup>, reactive silica and REE contents [16].

Furthermore, important fundamental information about the effect of these impurities on the corrosion rates, crystallization of the precipitated CaSO<sub>4</sub> and the calculation method of fluoride complexing ratio (FCR) can be found in the literature by M.Schorr [13].

On the basis of this, FCR is calculated from the phosphates analysis data obtained taking into account the solubility factors of these impurities in PA and are presented on Table 3. As seen, the results of computation are highly fluctuating from between 1.378 to 0.286. Then, we have studied the effect of FCR on the SFC within this interval.

## 2.1 When the ratio of FCR higher than one:

### 2.1.1 Processing low silica - Alumina phosphate

Operating experience when treating several grades at Aqaba PAP has shown that using phosphate which contains extremely low levels of  $\text{Al}_2\text{O}_3$  (0.18 - 0.2%) and  $\text{SiO}_2$  (1.8 - 2.4%) suffers from lack of SFC and leads to the following observations:

- Poor slurry filtration characteristics, low filtration capacity and washing efficiency. When increasing the SFC higher than  $4.6 \text{ t/m}^2 \text{ pd P}_2\text{O}_5$  and after hours of retention time, the quality of slurry filtration deteriorates, completely uncontrollable, and consequently the attack. On account of bad filtration, the water - soluble  $\text{P}_2\text{O}_5$  losses in gypsum increase asymptotically to higher than 5.0%. Microscopic examination discloses that the gypsum consist of long thin needle shaped crystals: (length 250-300; width 30-50 and thickness 2-3 microns). Beside it is in dark colour, very sticky with 33-37% of free water content and high thixotropic properties. Surface area of cake is about  $3400 \text{ cm}^2/\text{g}$ .
- The maximum  $\text{P}_2\text{O}_5$  content attainable of the filtrate acid is between 25.5 to 26% of  $\text{P}_2\text{O}_5$ . Further, it is becoming difficult to filter the slurry while increasing the  $\text{P}_2\text{O}_5$  higher than 26% because of high slurry viscosity.

Interesting data are given in unpublished report [9] about treating the same type of phosphate but using single reactor having  $2 \text{ m}^3/\text{tpd P}_2\text{O}_5$  of SRV. The phosphate was ground to 59% minus 200 mesh. The average  $\text{P}_2\text{O}_5$  and free sulfate content of the filter acid were 28.2 and 2.4% respectively, whereas, the SFC was reached only  $3.9 \text{ t/m}^2 \text{pd P}_2\text{O}_5$ . The dominant length and width of the dihydrate  $\text{CaSO}_4$  crystals were 40 to 130 and 10 to 30 microns, which gives an average L:W ratio of 5.9:1 and an average surface area of  $3044 \text{ cm}^2/\text{g}$ .

Moreover, it is important to mention, that the  $\text{Al}_2\text{O}_3$  content in the PA produced is found to be less than 0.05%, which seems to be low proportion to the acid available for the formation of complex salts.

The unusually high corrosion on equipment is due to high F/ $\text{SiO}_2$  ratio (1.7 - 2).

### 2.1.2 Application of Inorganic Modifiers

The above mentioned process problems have been successfully overcome by international and local experiences, study and research activities. Consequently, from industrial experience, as an improvement,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  contents can be adjusted in a proper ratio by adding:

(a) Aluminum hydroxide  $\text{Al}(\text{OH})_3$ , (b) clay and kaolin minerals, (c)  $\text{SiO}_2$  as cheaper by product from the  $\text{AlF}_3$  plant, (d) in the case of high silica content, blending various grades at different ratios is more favourable and applicable.

In addition, it has been investigated the effect of the addition of various inorganic modifiers on the SFC for the phosphate containing low  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ .

The trials involved the addition of reactive  $\text{SiO}_2$  and clay minerals. (Jordanian clay contains on dry basis, wt.% 14.8 - 21.1  $\text{Al}_2\text{O}_3$ , 6.4 - 7.8  $\text{Fe}_2\text{O}_3$ , 54 - 61.3T.  $\text{SiO}_2$ , 35.3  $\text{SiO}_2$  reactive 35.3 and 1.2 - 1.8  $\text{TiO}_2$ ).

Treatment took place in a reactor having  $1 \text{ m}^3/\text{tpd P}_2\text{O}_5$  of SRV. The  $\text{P}_2\text{O}_5$  concentration of the filtrate acid was controlled at the level of 27%. The results listed below showed, that there were changes in physico-chemical properties of dihydrate  $\text{CaSO}_4$  crystals in particular, increased the surface area and its permeability, so as the SFC increased as much as 1.35-1.5 times. Moreover it must be pointed out, that in this way the unusually high maintenance costs have been reduced considerably.

SFC t/m <sup>2</sup> pd P <sub>2</sub> O <sub>5</sub>	Permeability cm <sup>2</sup> X 10 <sup>-9</sup>	Porosity %	Surface Area cm <sup>2</sup> /g	Raw Material
4.3	24.55	70	3397.7	low Al <sub>2</sub> O <sub>3</sub> and SiO <sub>2</sub> Phosphate
7.6	36.6	62	1278.7	Adding SiO <sub>2</sub>
8.5	53.77	64	1193	Adding Clay Mineral
>7.0	35 - 56	58 - 63	1027 - 1155	high Al <sub>2</sub> O <sub>3</sub> and SiO <sub>2</sub> Phosphate

## 2.2 Reduction of FCR to 0.83 in the phosphate used

This causes some improvements with plant performance:

- Increase the SFC from 4.6 to 5.5 t/m<sup>2</sup> pd P<sub>2</sub>O<sub>5</sub>.
- Increase acid concentration of the filtrate acid to 27% P<sub>2</sub>O<sub>5</sub>.
- Improve marginally the filterability and washability of gypsum. Washing efficiency can be maintained at the level of 98.5%.
- Produced gypsum containing big rhombic shaped crystals with 26-28% free water content.

Moreover, it has been observed that, when Al<sub>2</sub>O<sub>3</sub> content in slurry liquid phase is less than about 0.08%, the gypsum becomes waxy, soft and white in colour consists of needle shaped crystals. Further increases in the content to higher 0.1% changes crystals habits from needle to rhombic shape.

## 3. Processing blended low grade rock

Today different phosphate grades come to supplement Aqaba PAP as a blended 66-68% BPL containing such as 60-62% fines phosphate, 58-60% high carbonate, 60-62% high insoluble silica phosphate, 68-70% and 70-72% grades from different mines. The latter grade is a master controller of blending the rock.

The summaries of their chemical compositions are (on dry basis, wt.%): 27 - 32.2 P<sub>2</sub>O<sub>5</sub>, 42.5 - 50 CaO , 4.0 - 14.1 SiO<sub>2</sub>, 0.35 - 1.5 Al<sub>2</sub>O<sub>3</sub>, 3.4 - 3.8 F<sup>-</sup> and 1.475 - 1.818 CaO/P<sub>2</sub>O<sub>5</sub>.

Blending the above mentioned grades could be done in various proportions at the plant site, according to qualities of the final blended rock and economic consideration (P<sub>2</sub>O<sub>5</sub>, CaO/P<sub>2</sub>O<sub>5</sub> ratios) or plant capacity, qualities of downstream product or economic flexibility: P<sub>2</sub>O<sub>5</sub> prices, SA consumption and P<sub>2</sub>O<sub>5</sub> recovery. For these reasons, blending is one of the problems standing in the way of the utilization of such blended phosphate at Aqaba PAP.

### 3.1 Reduction of FCR to 0.42

This ratio offers certain advantages over the other three. In this case, the results of processing blended rocks show that a SFC is reached higher than 7.4 t/m<sup>2</sup> pd P<sub>2</sub>O<sub>5</sub> with a 27-28% of P<sub>2</sub>O<sub>5</sub> content of the filtrate acid. This has been possible, in part, by using a phosphate with high content of Al<sub>2</sub>O<sub>3</sub>, by keeping the solid content in reactors slurry at high levels 37-39%, adjusting the flow quantities of phosphate and SA distribution through the reactors, replacing the old filter perforated sheets and reducing the clearance between gypsum screw and **UCEGO** filter table which should be lower than 10 mm. In addition to that, the rotation speed of the table filter can be kept at higher rate. The technological indicators such as P<sub>2</sub>O<sub>5</sub> extraction and cake washing efficiencies are maintained at the level of 95.8-96.3% and 99-99.3% respectively. Thus, the plant efficiency in excess of 95.0 up to 95.5.

Moreover, gypsum contains about 23-26% free water with low surface area 1200-1500 cm<sup>2</sup>/g. Gypsum crystals are either in big rhombic and twin crystals or mostly granular, sandy and cream in colour consisting of prism hexagons crystals and mainly small spheres which agglomerate to form cluster type crystals having surface area of about 1027 - 1155. cm<sup>2</sup>/g .

For a brief period, while using low grade 68% BPL with the composition (wt.%) of 67.7 BPL, 30.98 P<sub>2</sub>O<sub>5</sub>, 49.5 CaO, 5.78 SiO<sub>2</sub>, 3.7 F and 0.45 Al<sub>2</sub>O<sub>3</sub> a rate equivalent to 1525 t/d was achieved, (or 7.44 t/m<sup>2</sup> pd P<sub>2</sub>O<sub>5</sub>) which means 16.4% in excess of guaranteed output rate. The results of industrial trails are listed below. As seen it is easy to understand why water soluble P<sub>2</sub>O<sub>5</sub> is a little high 0.44%.

R-6301	Flash cooler		YIELD %	P <sub>2</sub> O <sub>5</sub> Losses in gypsum				MTPD P <sub>2</sub> O <sub>5</sub>	Plant Capacity%	P <sub>2</sub> O <sub>5</sub> in acid, %
	C	T		Pr mmHg	w.s%	Co.Cry%	Unr. %			
76.40	1.96	334	95.033	0.20	0.82	0.25	1.07	1430	109.2	27.18
75.80	1.89	331	95.029	0.16	0.88	0.19	1.07	1350	103.1	27.77
76.82	2.07	349	95.172	0.14	0.86	0.18	1.04	1380	106.5	27.81
77.3	2.18	275	>94.5	0.44	0.817	0.13	0.947	1525	116.4	~27.00

### 3.2 When Blending with Fines Phosphate

It is more complicated dependency of SFC upon the FCR when further reducing below 0.32. The difficulties are certainly in relationship with mineralogical structure of phosphate containing high SiO<sub>2</sub> (soluble and insoluble forms).

Operation of blending the fines at unloading station and grinding section causes serious problems from the economical and as well as environmental point of view because of spillage. According to that, it has been decided to introduce this type of material once the plant on stream across the ground silo in order to avoid the mentioned problems and to keep away from developing overgrinding phenomena. The fines contain 10-15% particle size minus 50 microns and characterized by low P<sub>2</sub>O<sub>5</sub> content (20 to may be 7%), high SiO<sub>2</sub> content (25 up to 50%), kaolinite and carbonate as CaCO<sub>3</sub> contents ranging from 5 up to 13% and 6 up to 10.5% respectively. Moreover the specific surface area of these particles is between 37.7 up to 59.2 cm<sup>2</sup>/g. It has been confirmed that phosphate rock containing fines higher than 10% < 50 microns has viscosity which does not follow Poiseuille laws [9]. Also, it has been established that increasing the percentage of the smaller sized crystals below minus 40 microns from 20 to 40% will show 50% worse filtration rate [2].

A number of PA production industrial trials were carried out at PAP to treat low grade blending with fines at different feed rate with the composition of 65.1 BPL, 29.8% P<sub>2</sub>O<sub>5</sub>, 47.5% CaO, 9.5% SiO<sub>2</sub> and 0.66% Al<sub>2</sub>O<sub>3</sub>. Serious technical problems were faced when 13-15 t/hr of fines have to be blended. The bad filtration of the slurry could be attributed to the high viscosity and high percentage of SiO<sub>2</sub> fines (minus 50 mic). In the other respects, increase the P<sub>2</sub>O<sub>5</sub> in downstream product seems to be impossible. It was found that even a reduction of dosing rate to 3-4 t/hr was not sufficient to obtain a slurry that could be filtered properly. Moreover, experience and studies have indicated the following:

Extremely low SFC 3.5 - 3.7 t/m<sup>2</sup>pd P<sub>2</sub>O<sub>5</sub> because of poor slurry filtration characteristics. In this case, the filtration step becomes the process bottle-neck of PA production. The gypsum consist of a very small, broken mixed between rhombic and amorphous crystals would normally result in lower cake washing efficiency 97%. Even so the concentration of P<sub>2</sub>O<sub>5</sub> in acid was reduced to less than 25%. High SA consumption (2.98- 3.2 t salt P<sub>2</sub>O<sub>5</sub>) owing to high CaO/P<sub>2</sub>O<sub>5</sub> ratio and low overall P<sub>2</sub>O<sub>5</sub> recovery 92-93%. Also, the high stability of foam which it was very difficult to destroy on the surface of slurry. These reasons can may be explained either because of the presence of SiO<sub>2</sub> and kaolinite or in connection with specific area. It is obvious, that fine particles of SiO<sub>2</sub> which interfere by increasing the slurry filtration time, disturb the filtration operation by developing blocking phenomena the pores of gypsum cake and increasing the viscosity of the slurry. The practical use of this blended phosphate seems not to be possible. Moreover, blending was discontinued because of lack of adequate equipment and finally, decision has been taken to stop feeding the fines.

### 3.3 When Blending with High Insoluble Silica Phosphate

Another example of blending is using a low grade but containing high abrasive silica of about 64.5 BPL, 29.5% P<sub>2</sub>O<sub>5</sub> and 7 to 9% SiO<sub>2</sub> or higher. The SFC was fluctuating between 4.8 - 5.2 t/m<sup>2</sup>pd P<sub>2</sub>O<sub>5</sub>. On the other hand, it must be pointed out that blended phosphate needs high grind. However, the presence of silica in the insoluble quartz form or chert causes a serious erosion problem similar to that encountered in PAP.

- Increased erosion of the rotating equipment. It has been reported that 904 SSL agitator blade of the second digester was damaged due to high abrasion.
- Further difficulties were due to the sedimentation of quartz to the bottom of the reactors, digesters, flash cooler and on the surface of reactor, scrubbing fans depending on particle size of SiO<sub>2</sub>.

The average SFC is to some extent improved due to the adjusting of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> contents in the blended rock by 70-72% grade. The high content of Al<sub>2</sub>O<sub>3</sub> and acceptable level of SiO<sub>2</sub> resulted in quite a high SFC and further a high P<sub>2</sub>O<sub>5</sub> in the down stream product.

Consequently, full SFC of the plant 6.4 t/m<sup>2</sup> pd P<sub>2</sub>O<sub>5</sub> was obtained from using low blended grade rock containing (on dry basis wt.%) 66.8 PBL, 30.55 P<sub>2</sub>O<sub>5</sub>, 50.01 CaO, 5.9 SiO<sub>2</sub>, 0.4 Al<sub>2</sub>O<sub>3</sub> and 0.21 Fe<sub>2</sub>O<sub>3</sub>. In this case the gypsum consisted of very small (5-10 microns) crystals which form large (40-80 microns) cluster. The water soluble, cocrystallized and unreacted P<sub>2</sub>O<sub>5</sub> losses in gypsum were respectively 0.39, 0.67 and 0.04 in gypsum.

The plant has performed satisfactorily with this feed stock, thanks to a multicompartment reactor and three digesters system.

As a result of employing 66-68% BPL grades, unfortunately, the SA consumptions are high 2.90-2.98 depending on CaO/P<sub>2</sub>O<sub>5</sub> ratio, SO<sub>3</sub> content in the rock and P<sub>2</sub>O<sub>5</sub> recovery.

#### **4. Effect of Filter Cloth**

An additional factor influencing slurry filtration and plant performance is the filter cloth. Over the years, a number of filter cloth types including polyester and monofilament polypropylenes, have been employed and tested at Aqaba PAP.

Polypropylene cloth is relatively commonly utilized, but some several are unsuitable for huge plants because of the short durability caused by weakness in mechanical strength. The choice of a correct filter cloth poses difficult problems on account of the corrosive and scaling properties of PA slurry.

Consequently, when evaluating the economics and operating life of such a cloth, various points have to be taken into consideration and weighted according to each particular situation.

##### **- Material**

Polypropylene monofilament cloth is the most suitable. Though polyester is suitable for P<sub>2</sub>O<sub>5</sub> filtration and has better mechanical features than polypropylene, the relative price to usage length makes polyester expensive, especially for table filter.

##### **- Weaving Pattern**

Should provide a smooth and flat surface on the cake side so as to make the cake discharge as good and easy as possible.

##### **- Calendering**

Calendering is required for thermofixation of the cloth and also to help to obtain a smoother surface and if necessary to reduce the permeability. But calendering reduces the resistance of the cloth drastically. This is why a cloth with calendering should rarely be chosen.

Calendering should be just a finishing touch. The reason why many filter cloths are very much calendered is purely economical. It is possible to take a light and very open cloth (Table - 4, type A), thus very cheap but such a cloth has a very poor mechanical resistance .

##### **- Permeability**

In general, the permeability of cloth used for PAP is within the range of 4000-8000 m<sup>3</sup>/m<sup>2</sup>.hr to air under 20 mm of H<sub>2</sub>O gauge [4]. According to our experience, the permeability must be at least 6500 m<sup>3</sup>/m<sup>2</sup> hr.



### **- Wire Diameter**

To obtain the required permeability and filtration rate features mostly by weaving pattern with only little calendering, the cloth must be made with wires of not less than 0.35 mm (35/100) diameter. A good mechanical resistance also requires wires of minimum 0.35 mm to may be 0.4 mm.

### **- Weight**

For the same reasons as those mentioned here above, the cloth may not have a weight less than 400 gr/m<sup>2</sup>.

### **- Mechanical Characteristics**

Tensile strength and elongation depends on wire diameter, quality and number of wires and calendering. Actually, the resistance of the filter cloth is a result, of these mentioned items. The cloth should have as much as possible mechanical resistance in order to have the longest possible life span. Thus, it should have a tensile strength of not less than 390 in warp and not less than 240 da N/5cm in weft.

### **- Flexibility**

The cloth should show enough flexibility to be fitted and maintained in the groves of the **UCEGO** filter. Finally, the cloth should provide the best relation price to durability.

Technical specification of various types of polypropylene monofilament woven cloth utilized for **UCEGO** Filter are presented in Table 4.

### **Factors Influencing Filter Cloth Life Span**

On the basis of the results obtained from numerous different cloths trials have shown that three major factors are responsible for filter cloth life span.

- Filter slurry temperature,
- Permeability and mechanical strength of the cloth used,
- The tonnage of P<sub>2</sub>O<sub>5</sub> produced per total filter surface area.

#### **4.1 Filter Slurry Temperature**

Until 1993 PAP operated at extremely high filter slurry temperature in excess of 82 up to 83.5°C. The reason, however, was to achieve the designed daily production of P<sub>2</sub>O<sub>5</sub>.

Thereby, the scaling tendency depends not only on the levels of impurities from the rock employed, but mostly on the filter slurry temperature. Moreover, the latter is one of the most important factor governing the rate of scale formation.

The dependence of the slurry temperature on the filter cloth life span was studied with a range of temperature intervals from 74 up to 83.5°C, depending on the adopted process route and differences in process conditions. The rate of scaling increases as the filter slurry temperature rises simultaneously, the cloth life span drops (Table 5). Our results conclusively show that the basic reason for the short of operating time is the clogging by chukhrovite and alkaline fluosilicates. Therewith, the plugging itself is caused by the excessively high slurry temperature.

Furthermore, as a result, after a couple of months there is a high risk of considerable scaling of the filter grids and 30% product pipes which are the most sensitive to scaling.

A series of experiments were carried out to study the impact of temperature on various types of cloth utilized. Clearly (Figure 2), it has been confirmed that any new cloth used had to be taken off after approximately 12 days only, because of quick plugging while still in perfect mechanical conditions.

However, to obviate a build up of encrustations a systematic daily short washing open cycle of about 2 hours was periodically carried out with hot water 75-80°C and at pressure 6.5 bars. This technique was recommended by Rhone-Poulenc.

Consequently, from the conventional filter cloth type A maximum achievable tonnages of the near 16479 tons of P<sub>2</sub>O<sub>5</sub> was obtained, from 316.4 hours compared with the average of 11676 tons during and 224 hours (Figure 2).

In addition to that, with the object of improving the washing condition, and eliminating the clogging of the filter grids and cloths an additional high pressure spray water oscillator was installed at the filter dam under 50 to 80 bar and consuming at most 5 m<sup>3</sup>/hr. In such cases of highly scaling slurry, it is necessary to use it continuously.

The performance had been described as good and the plugging problems was decelerated. As a result seen in Figure 2, Cloth types A, C and D produced 16479, 17414 and 18796 tons of P<sub>2</sub>O<sub>5</sub> during 316.4, 334.4 and 360.9 hours respectively (one hour = 52.08 ton) giving 40 to 60% longer services. But when the oscillator was out of order, which happened many times, therefore, any type of filter cloth employed had to be removed as fast as the conventional cloth type (A), after 12 to may be 14 days.

Eventually, it was decided to use basically Cloth A that was exceptionally cheaper in spite of poor mechanical resistance.

The main points of the PAP revamping is to reach the designed capacity while processing at low temperature so as to limit the degree of supersaturating of the filter acid. The rate of encrustation, thus, the degree of scale formation on the cloth, is progressively reduced with the decrease in slurry temperature from 83.5 to 74°C.

As shown from the comparison in Table 5, the cloth service increases sharply with temperature. Cloth type B at high temperature only produced 12329 tons 236.7 hours. Whereas, at 74°C the production increased by as much as 2.25 times.

Also the operation times for replacing the cloth reduced to approximately 50%. Therefore, it completely changes the washing cycle procedure.

#### **4.2 Permeability and Mechanical Strength**

Table 1 shows the blended phosphates of 66-68% BPL grades containing less in total cationic impurities such as iron, aluminum and magnesium. Relatively the produced PA is of the highest quality. Moreover, the average solid content is less than 0.8% passing with filter acid.

To investigate the effect of cloth permeability on the gypsum washing efficiency a series of industrial trials were performed with permeability between 2500-3500 and 6500-7500 m<sup>3</sup>/m<sup>2</sup>.hr. The results showed (Figure 3), a cloth with high permeability gave better filter performance and higher washing efficiency 99-99.5%.

Moreover, it is easy to obtain up to 28% P<sub>2</sub>O<sub>5</sub> of the filter acid while maintaining high gypsum washing efficiency at level of about 99.1% as compared with filter cloth having low permeability. Thereby, average washing efficiency was between 98.59-98.7. Increasing the P<sub>2</sub>O<sub>5</sub> content to 27.69 in the produced acid caused higher, water soluble P<sub>2</sub>O<sub>5</sub> and 0.86% in gypsum level. In this case washing efficiency dropped to 96.43%.

Thus, our philosophy is to use filter cloth with high permeability with as much as 6500-7500 m<sup>3</sup>/m<sup>2</sup> hr.

The mechanical resistance is another important property of filter cloth. The effect of strength was studied on the cloth life span at constant temperature.

The results of comparative testing based on utilized phosphate grade 66-68% are given here below. On account of outstanding mechanical resistance and to illustrate its long service, the new cloth type B can produce 50% higher compared with the light weight cloth A.

The test was considered satisfactory and the cloth gave 4 weeks of service.

Type of cloth	weight g/cm <sup>2</sup>	mechanical strength daN/5cm		Tonnage of P <sub>2</sub> O <sub>5</sub>	Running Hrs
		warp	welf		
A	260	235	135	13208	242
B	440	460	225	> 27721	> 507.9

Providing such an efficient, high performance and good physico-mechanical characteristics of cloth type B thereafter, have been approved as new standard cloth for Aqaba PAP. The annual order was reduced from 900 to 400-450 pieces.

### 4.3 Tonnage of P<sub>2</sub>O<sub>5</sub> Produced

According to reference [4], it demonstrated that rotary table filter have a shorter cloth life of 700 hours on account of the gypsum removing scrolls. It has recommended that when the longevities of filter cloths from two different types of filters are compared, the correct index is the tonnage produced of P<sub>2</sub>O<sub>5</sub> per unit of total filter surface area (total surface area of Ucego 12 is 255 m<sup>2</sup>).

A number of studies have been performed on the tonnage of P<sub>2</sub>O<sub>5</sub> produced of several types of cloth. Consequently, whereas Figure 4 shows that maximum obtainable tonnage of P<sub>2</sub>O<sub>5</sub> produced is 27721 tons which equivalent to 108.7 ton per unit of total filter surface area.

Finally, whatever the washing cycle used, at the end of a certain period of operation the cloth become unserviceable owing to blocking, mechanical wear (perforation or tears due to abrasion) and thermal deterioration.

### Conclusion

The most important conclusion that can be drawn from the results of industrial experiments is that the F, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> affect the quality criteria of Jordanian phosphates and are confirmed by our experiments.

The required minimum level of alumina in the slurry liquid phase for agglomerate formation of dihydrate CS should be higher than 0.15 Al<sub>2</sub>O<sub>3</sub>.wt. %.

Depending on the content of F, Al<sub>2</sub>O<sub>3</sub> and type of SiO<sub>2</sub> present in the phosphate rock the filter loading requirement should be in the range of 3.5 to 7.4 t/m<sup>2</sup>pd P<sub>2</sub>O<sub>5</sub>.

In order to reach the highest figure of SFC with minimum corrosion and erosion problems, the FCR is found to be within the range of 0.75-0.54.

Keeping the high SFC by using 66-68% BPL phosphate the FCR can be reduced from 0.54 till 0.47 with acceptable level of erosion rate.

Working with phosphate containing 0.327-0.286 FCR is the worst condition from the economic point of view in PAP.

### References

1. Rhone-Poulenc improves the single tank PA process, Phosphate & Potassium No.88 1977, 147, 1987.
2. Becker P., 1994, Check-up for PA Units. IFA Tech. Conference, Jordan.
3. Hummadi N., Smith P.A., P. Pluvinage P., 1994, Rehabilitation of JPMC-PAP in Aqaba. IFA Technical Conference, Amman - Jordan.

4. Becker P., 1989, Phosphates and phosphoric acid 1st and 2nd Editions New York, Marcel Dekker Inc.1983.
5. Becker P., 1997, Phosphate Raw Materials, their Impurities and Related Effects on Commercial Product. AFA Tenth Annual Tech. Conference Jordan.
6. Halaseh K.Gh., Laboyko A.Y, Toshinsky V.I, 1993, Comparative Evaluation of Jordanian phosphate rock for WPA production. Him Prom N.12, P. 624-632 Moscow.
7. Oweis I., Hummadi N. and Mubaideen M., 1994, Flexibility in rock phosphate flowsheets to meet the specific requirements of individual plants IFA Technical Conference, Jordan.
8. JPMC booklet 1996.
9. JPMC. Documents and PA manual, Raw material specifications. 1979 /1980.
10. Halaseh K.Gh., 1997, WPA using Jordanian Phosphate rocks AFA Tenth Annual Tech. Conference, Jordan.
11. Schorr M., 1996, Mineral modifiers improve WPA production, Phos & Potassium No.187-1993 and No. 202.
12. Smith P.A., What is a high grade phosphate, PAS/910927.
13. Schorr M., 1993, WPA production using Phalaborwa phosphate rocks. Phos & Potassium No.184.
14. Phosphate rock grade and quality. Phosphate & Potassium No.178-1992.
15. Kopilev A.B., 1972, Technology of Phosphoric Acid. Moscow.
16. Karmishov V.E., 1983, Chemical Processing of Phosphorites Khimiya publishers Moscow.
17. Cadmium issue, Phosphate & Potassium No.1995
18. Slack A.V., 1968 PA. Marcel Dekker Inc. New York.

**Table - 1 Analysis of Different Grades of Jordanian phosphate Rock , Wt% .**

<b>BPL</b>	73.85	74.00	72.98	74.99	74.99	74.51	76.48	70.01	72.00	71.01	72.11	65.11	65.83	69.46	67.69	66.08
<b>P2O5</b>	33.80	33.87	33.40	34.32	34.32	34.10	35.00	32.04	32.95	32.50	33.00	29.8	30.13	31.79	30.98	30.55
<b>CaO</b>	50.50	52.00	51.00	53.00	50.00	51.00	52.00	49.50	49.00	49.00	51.00	47.5	48.52	48.2	49.5	50.01
<b>SO<sub>3</sub></b>	1.15	1.20	1.40	1.10	1.00	1.20	0.80	1.40	1.10	1.35	1.20	...	1.37	1.08	...	...
<b>SiO<sub>2</sub></b>	6.10	2.60	3.50	2.4	3.00	4.00	2.00	6.50	4.50	5.50	4.20	9.5	8.34	7.12	5.78	5.90
<b>CO<sub>2</sub></b>	3.42	4.00	5.00	4.00	3.00	4.00	3.00	5.00	4.00	5.50	4.00	...	5.28	4.45	...	...
<b>F</b>	3.74	3.50	3.90	3.60	3.50	4.00	3.80	3.80	3.50	3.90	3.60	3.65	3.65	3.80	3.7	3.74
<b>Al<sub>2</sub>O<sub>3</sub></b>	0.39	0.28	0.20	0.18	0.20	0.30	0.20	0.40	0.25	0.40	0.30	0.66	0.53	0.52	0.45	0.40
<b>Fe<sub>2</sub>O<sub>3</sub></b>	0.26	0.18	0.32	0.20	0.15	0.40	0.20	0.35	0.15	0.32	0.23	...	0.34	0.26	0.24	0.21
<b>MgO</b>	0.25	0.25	0.40	0.20	0.10	0.20	0.10	0.45	0.30	0.40	0.30	...	0.28	0.20	...	...
<b>Na<sub>2</sub>O</b>	0.54	0.40	0.60	0.40	0.40	0.60	0.40	0.60	0.40	0.60	0.40	...	0.52	0.52	...	...
<b>CaO/P<sub>2</sub>O<sub>5</sub></b>	1.494	1.535	1.527	1.544	1.457	1.466	1.486	1.545	1.487	1.487	1.545	1.594	1.61	1.516	1.598	1.64



**Table - 3 Fluoride Complexing Ratio For Jordanian Phosphate Rocks, Wt %**

SiO <sub>2</sub>	F	Al <sub>2</sub> O <sub>3</sub>	Weight Ratio % F/SiO <sub>2</sub>	Combined Equivalent Ratio F/(SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> )
6.10	3.74	0.39	0.613	0.458
2.60	3.50	0.28	1.346	0.971
3.50	3.90	0.20	1.114	0.837
2.40	3.60	0.18	1.500	1.111
3.00	3.50	0.20	1.167	0.870
4.00	4.00	0.30	1.000	0.740
2.00	3.80	0.20	1.900	1.378
6.50	3.80	0.40	0.585	0.438
4.50	3.50	0.25	0.778	0.585
5.50	3.90	0.40	0.709	0.526
4.20	3.60	0.30	0.857	0.637
9.50	3.65	0.66	0.384	0.286
8.34	3.65	0.53	0.438	0.327
7.12	3.80	0.52	0.534	0.396
5.78	3.70	0.45	0.640	0.473
5.90	3.74	0.40	0.634	0.472

**Table - 4 Technical Specification of different types of 100%Polypropylene Monofilament Cloth used for Ucego Filter .**

Type	Weight g/m <sup>2</sup>	Wire Thicknesses, mm		Resistance,dan/5cm		Mesh Opening Microns	Air permeability under 20mm of H <sub>2</sub> O gauge(m <sup>3</sup> /m <sup>2</sup> .hr)
		along wrap	across weft	warp	welf		
A	260	30	100	235	235	40x500	6500 - 8500
B	420 - 440	40	100	485	225 - 235	20x500	5000 - 7500
H	435	40	100	460	190	20x500	1700 - 3800
R	290	30	100	333	140	....	2500 - 3500

**Table - 5 Effect of filter slurry temperature on the cloth lifetime and PAP performance .**

Cloth Type	Tonnage of P2O <sub>5</sub> Produced	Filter slurry temperature °C	Filter cloth lifetime hours	Time for cloth removing & replacing	Procedure of washing Cycle	Scale Tendency	Filter cloth Condition
B (1)	12329	82 - 83.5 °C	236.7	1-2 ; 3.5 - 4 Hrs	a systematic daily wash of about 2 - Hrs duration	high scale formation	Completely blinded & thermal deterioration
B (2)	27721	74 - 75.5°C	507.9	30 min. , 3Hrs	a systematic wash 6 - 8Hrs once a two weeks	Relatively little scale formation	Tear due to abrasion & perforation

(1) one hour = 52.08 Ton , (single tank reactor ) .

(2) one hour = 54.58 Ton , (multicompart ment reactor & three digesters) .



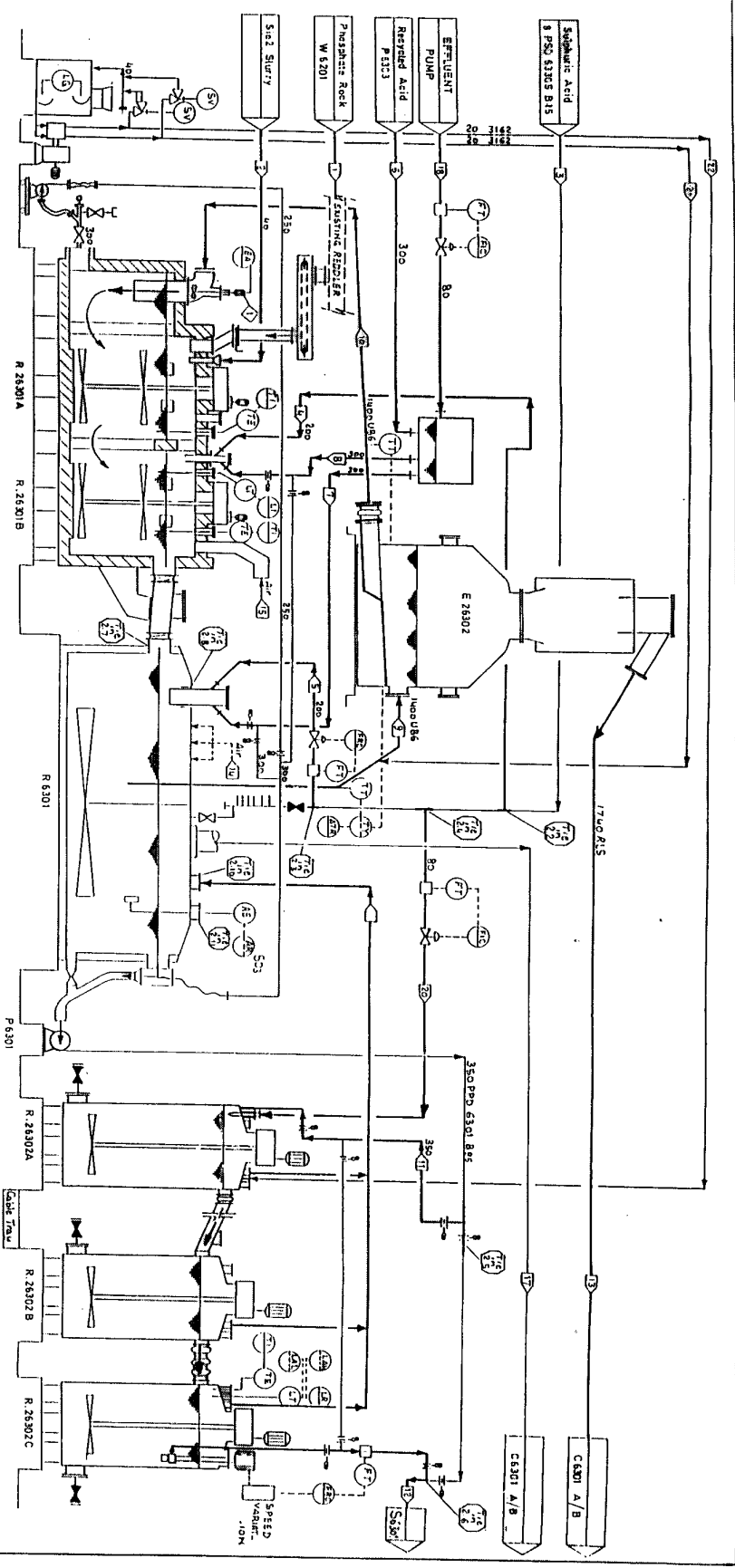


Fig-1 Process Flow Sheet of Aqua PAP.

NOTES: 1- FLOW AFTER SCRUBBER (FEEDWITH WATER A 20CL = 240000 m<sup>3</sup>/h)  
2- DESIGN FLOW 22 MASH

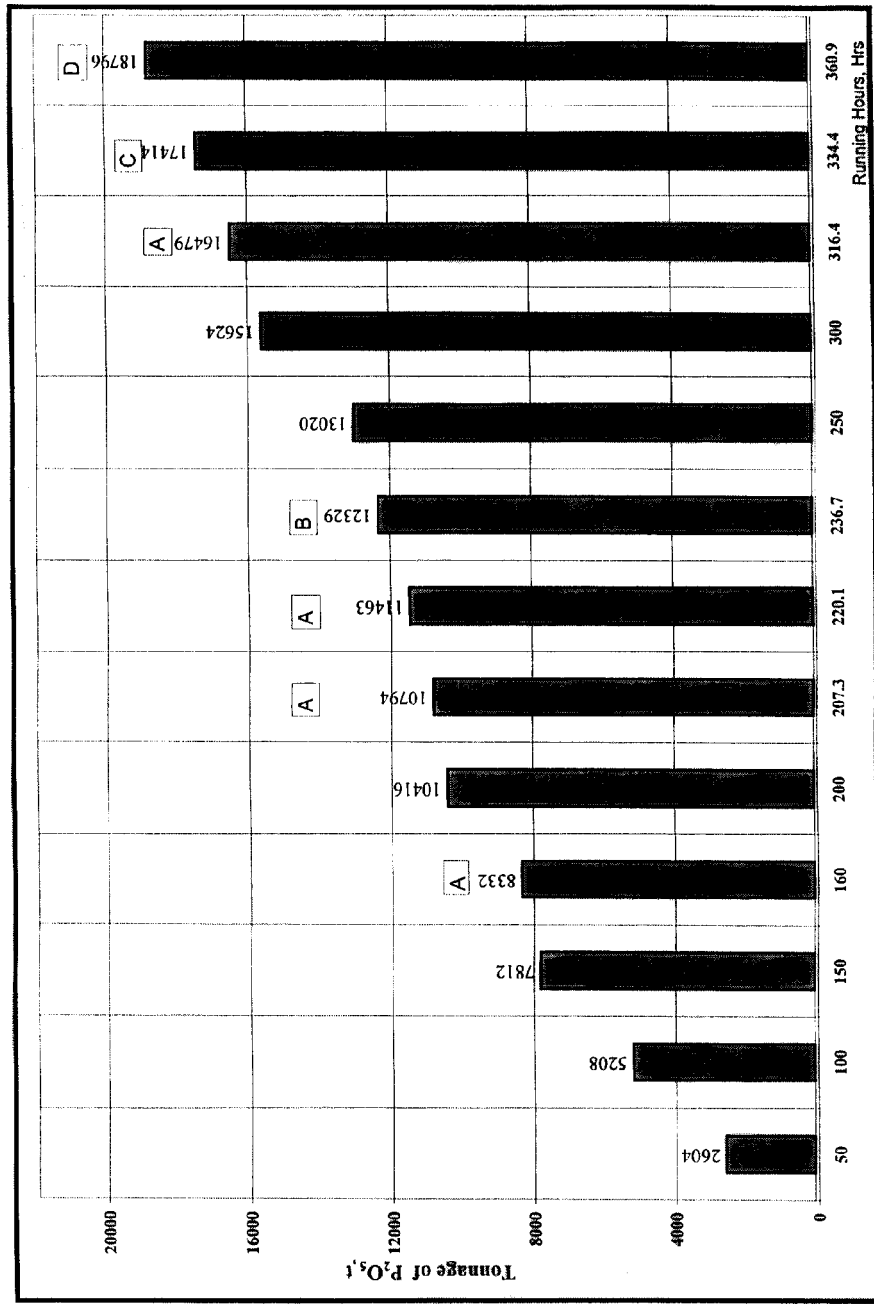
	UNITS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	
SOLIDS	TON	178810	120700									286857	286857										
LIQUIDS	TON		4800									509613	509613										
AIR	TON													202500	5000	2000	210000						
H <sub>2</sub> O	TON	3480												42055	6773	155	500	37048	15000				
TOTAL FLOW	TON	182290	6072	162703	60301	60301	58902	58902	58902	1392000	1360795	786270	786270	42055	206775	5155	3250	247248	165000			5.6	5.6
TEMPERATURE	°C	40	50	45	45	45	68	68	68	76.5	74	74	74	45	45	63	65.4	40	40			40	40
PRESSURE	Bar Abs	600	1140	1020	1020	1020	1180	1160	1160	1560	1552	1552	1552	0.240	ATM	ATM	ATM					1000	
DENSITY	kg/m <sup>3</sup>	800	1140	1020	1020	1020	1180	1160	1160	1560	1552	1552	1552	0.240	ATM	ATM	ATM					1000	
VOLUME FLOW	m <sup>3</sup> /h	578	80.3	44.15	44.15	44.15	44.8	232.3	232.3	8000	8063	513	513	282319	18220	4800	2000	281000	13			4.3	

JORDAN PHOSPHATE MINES CP. LTD.  
FERTILIZER COMPLEX  
AQABA - JORDAN

PAP REVAMP PROJECT  
PROCESS FLOW SHEET REACTION  
1310 MTPD PPS D

Fig-2. Effect of Different Type of Filter Cloth on The Tonnage of  $P_2O_5$  Produced, (One Hr=52,08 Ton).

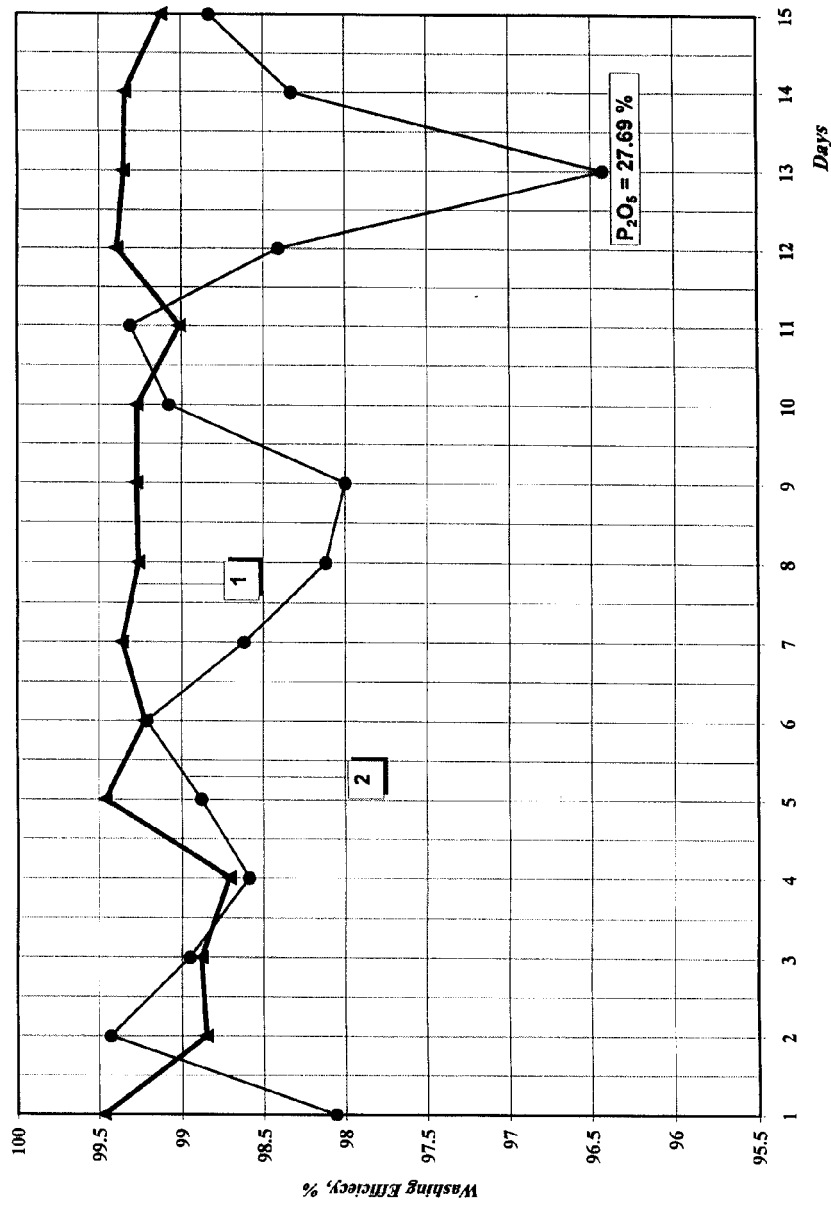
R.Hrs	Prod.	Cloth Type
50	2604	
100	5208	
150	7812	
160	8332	A
200	10416	A
207.3	10794	A
220.1	11463	A
236.7	12329	B
250	13020	
300	15624	
316.4	16479	A
334.4	17414	C
360.9	18796	D



Single Tank Reactor, RSV =  $1m^3$  /tpd  $P_2O_5$

Washing Efficiency, %	
Days	Cloth B
1	99.47
2	98.85
3	98.88
4	98.71
5	99.46
6	99.22
7	99.36
8	99.26
9	99.27
10	99.27
11	99.01
12	99.39
13	99.35
14	99.34
15	99.12
16	99.46
17	99.15
18	99.48
19	98.95
20	99.13
21	99.11
22	99.24
23	99.12
24	99.05
25	98.67

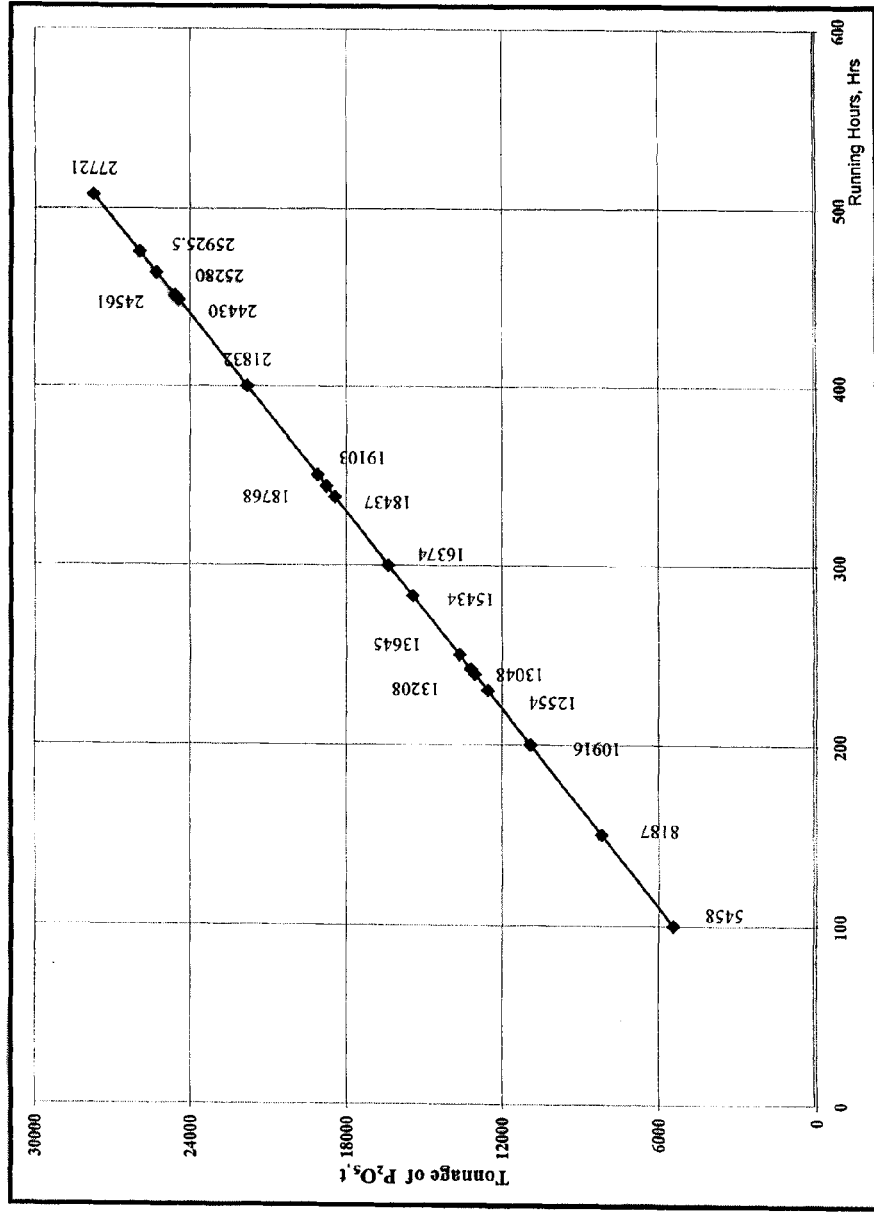
Fig 3 . Effect of Permeability of Filter Cloth on The Gypsum Washing Efficiency.



1- Permeability of Cloth B =  $6500 \text{ m}^3 / \text{m}^2 \text{ hr}$

2- Permeability of Cloth R =  $2500 \text{ m}^3 / \text{m}^2 \text{ hs}$

Fig-4. Effect of Different Type of Filter Cloth on The Tonnage of  $P_2O_5$  Produced, (One Hr=54,58 Ton).



R.Hrs	Prod.	Cloth Type
100	5458	
150	8187	
200	10916	
230	12554	A
242	13208	A
239.1	13048	R
250	13645	
282.8	15434	H
300	16374	
337.8	18437	F
343.9	18768	R
350	19103	
400	21832	
447.6	24430	G
450	24561	
463.2	25280	B
475	25926	B
507.9	27721	B

Multicompartment Reactor And Three Digesters ; RSV = 2m<sup>3</sup> /tpd  $P_2O_5$