

# **IFA Technical Conference**

Port El Kantaoui, Tunisia 12-15 September 1986 THIRTY YEARS OF THE S.I.A.P.E. PROCESS FOR THE PRODUCTION OF PHOSPHORIC ACID BY THE WET PROCESS AND EXPERIENCE IN THE REVAMPING OF PHOSPHORIC ACID AND FERTILIZER PLANTS

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### A - S.I.A.P.E. PROCESS

S.I.A.P.E. has developed a simple and efficient original process for upgrading low-grade phosphates and for the production of phosphoric acid with high yields.

The development of the process over the last thirty years took place in keeping with changes in the quality of the ore treated and in technological improvement with regard to materials as well as equipment.

The improvements of the process over the last thirty years may be summarized as follows:

modification of the process depending on the degree of impoverishment of the ore; optimization of water consumption; optimization of energy consumption; increasing the production capacities of new Units-

#### I - INTRODUCTION

The world production of phosphoric acid by the wet process from natural phosphate continues to increase. It has reached 20,000,000 tonnes of P<sub>2</sub>O<sub>5</sub> in 1982.

The producers of phosphoric acid are increasingly faced with the regular and continuous decrease in the P2Os content of phosphates and the corresponding increase in undesirable impurities during their conversion. In the long run, these problems will become more acute and phosphoric acid manufacturers must advance their tech+ nology in order to adapt to the new situation. In fact, a fair number of impurities in natural phosphates affect directly or indirectly the performance of a phosphoric acid production unit; the extent of influence depends not only on the individual concentrations of these impurities but also on their interactions.

.This situation can be remedied in two ways:

by attempts to purify and enrich the crude phosphate (more extensive, efficient washing, flotation, calcination etc.);

by development of the technologies for the conversion of phosphates so that they can be used for the treatment of relatively low-grade and impure phosphates.

It is in this latter context that S.I.A.P.E. has perfected and developed the process for the manufacture of 28% phosphoric acid by the wet process.

This process is designed for the treatment of low-grade phosphates containing significant levels of impurities.

The S.I.A.P.E. MULTITANK process which, since 1952, has been the basis for the production of phosphoric acid at the Plant "A", S.I.A.P.E. MAIN PLANT has developed a MONOTANK process in 1968.

The addition of a digestion tank which enables the performance of the process to be improved was carried out in 1978.

Since then, significant improvements have been introduced to the process. This has enabled the consumptions of inputs to be optimized.

This paper describes all innovations of the process, laying emphasis on the advantages achieved. But first of all, a brief account of the S.I.A.P.E. process with its history and development is presented.

II - DESCRIPTION OF THE S.I.A.P.E. PROCESS FOR THE MANUFACTURE OF PHOSPHORIC ACID

Phosphoric acid manufacture by sulphuric acid treatment, with the precipitation of calcium sulphate in the form of the dihydrate forms the chemical basis of the S.I.A.P.E. process.

## S.I.A.P.E. PROCESS

Phosphoric acid production in Plant "A", Main Plant of S.I.A.P.E. started in 1952 according to the S.I.A.P.E. multitank process, the acid treatment section of which consists of four stirred tanks placed in series and communicating between one another by overflow.

As the circulation of the mature sturry from the 4th to the 1st tank cannot be spontaneous, this is achieved by means of a high output vertical pump (SEE DIAGRAM No. 1 + PAGE - 25 = 0)

The tanks are made of lead, held in place with belts in the form of cage winding made of steel, internally lined with acid-proof bricks with a potassium silicate-based binder.

The tank cover is made of wood.

All the tanks are equipped with two-tier S.I.A.P.E. type stirrers which ensure the homogeneity of the reaction medium and enables the destruction of foams.

Measuring out the raw materials is done by means of dispensing wheels. The introduction of sulphuric acid and the recycled phosphoric acid into the treatment tank is carried out using S.I.A.P.E. type original injectors. This method of dilution and introduction makes it possible to avoid points of overheating and local over-concentrations of sulphuric acid which lead to decreases in the treatment yield.

The ground phosphate is introduced with a lifting screw into a wetter which receives the recycled slurry. This wetter enables the phosphate to be intimately mixed with the slurry before introduction into the treatment tank.

Although the suitability of this multitank process for the conversion of phosphates into 28% phosphoric acid has been proved for many years, some difficulties have appeared, namely:

the internal lining of the lead tanks needs frequent attention following its detachment; the reaction medium being corrosive (excess of sulphuric acid) promotes the corrosion of the stirrers;

the total absence of control and automation leads to frequent disorders in the treatment; the recycling of the slurry is inadequate.

These difficulties led us to design the new monoreactor S.I.A.P.E. process.

The Pilot unit which gave rise to the monoreactor S.I.A.P.E. process was built and installed at the phosphoric acid plant of the Plant "A" of S.I.A.P.E. in 1968. ItS industrial exploitation in parallel with the two existing multitank units (old S.I.A.P.E. process) made it possible to perfect the new S.I.A.P.E. process.

The use of the multitank process for several years and the various tests carried out with the pilot plant enabled us to make the choice of materials adequate for the process (P.V.C. - S.V.R., stainless steel, REIN-FORCED CEMENT etc.) (SEE DIAGRAM No. 2 - PAGE 26).

This process is used in the following industrial Units:

COMPANY	CAPACITY IN TONNES OF	TOWN (COUNTRY)	YEAR OF
	P <sub>2</sub> 0 <sub>5</sub> /0		PRODUCTION
I.C.M. 1	330	GABES (TUNISIA)	1971
S.I.A.P.E. A	320	SFAX (TUNISIA)	1973
I.C.M. 2	400	GABES (TUNISIA)	1974
AZOT SANAII	300	SAMSUN (TURKEY)	1974
S.I.A.P.E. 1	2 x 500	GABES (TUNISIA)	1979
C.I.C.	280	TURNU MAGRELLE (ROUMANIA)	1981
I.C.M. 3	500	GABES (TUNISIA)	1982
I.C.G.	500	GAFSA (TUNISIA)	1984
S.I.A.P.E. II CURRENTLY IN PROGRESS	2 x 600	LA SKHIRA (TUNISIA)	1987

# CHARACTERISTICS OF THE S.I.A.P.E. PROCESS

The S.I.A.P.E. process was developed initially for treating GAFSA phosphates which are well known for their low P205 content compared with the commercial phosphates of the largest producers in the world. However, its suitability for treating phosphates of all grades has been proved at the S.I.A.P.E. Plant itself and also in plants of overseas licensees, with very good yields. CONFIGURATION OF THE S.I.A.P.E. MONOREACTOR

A S.I.A.P.E. type monoreactor is in the form of a single tank consisting of a cylindrical central compartment and an annular compartment.

The raw materials are introduced into the central compartment.

The mixing of the concentrated (98%) sulphuric acid and phosphoric acid is ensured by an original, simple and efficient special mixer.

The central compartment communicates with the ring by two connections, an upper one which enables the evacuation into the ring of the slurry which is still in the form of an emulsion, and a lower one, which enables the mature slurry to be returned to the central compartment – this internal recycling is high and forms an important parameter.

The specific reaction volume is between 1.2 and 1.5  $\rm m^3/tonne$  of P2O5 day for GAFSA phosphate, depending on the quality treated.

The central compartment is equipped with a stirrer specially designed for playing two important roles — intimately mixing the reactants with the recycled sturry and assisting in the recirculation under the influence of the lower propeller which pumps the sturry upwards. The stirrers of the annular space are stirrers of the double turbine type, containing straight blades. The upper blades which are partially submerged in the pulp destroy the foams and disperse the sturry for heat removal by a stream of air at the surface. The lower blades generally of smaller dimensions, maintain the gypsum in suspension.

The stirrers of the ring are generally seven (7) in number. The effect of these stirrers combined with that of the central stirrer enable the slurry to be circulated intensely in the monoreactor. The energy consumed per  $m^3$  of slurry is 0.90 KW/ $m^3$ . The temperature gradient between the central compartment and the end of the ring is minimal.

The removal of heat of reaction is ensured by a slurry-gas surface contact by a stream of air in the empty part of the reactor above the slurry surface. The volume: surface area ratio of the slurry is optimized in order to obtain maximum cooling efficiency. This ratio, which is fixed when the reactor is designed, determines the slurry height which is, on average, three (3) metres for all the reactors in operation.

Before being discharged into the atmosphere, the reaction gases are scrubbed in scrubbing towers operating either with sea water or with well water depending on where the Plant is sited (SEE DIAGRAM NO. 3 - PAGE 27).

OPERATING PARAMETERS OF THE S.I.A.P.E. PROCESS

The temperature of the sturry is an important parameter of the process. In fact, the formation of dehydrated gypsum crystals which are perfectly stable and which are of dimensions well suited for filtering requires the sturry temperature to be maintained between 78 and  $80^{\circ}\mathrm{C}$ .

The  $P_2O_5$  concentration of the mother liquor is maintained between 27 and 30% according to the type of phosphate.

Depending on the quality of phosphate treated, the sulphuric acid concentration in the slurry is maintained between 0.8 and 1.2% of HoSO4.

The solids content of the slurry is an indication for the average phosphoric acid recirculation level required for the dissolution of the ore. This solids content is maintained at 30%, corresponding to a slurry density of 1.470 g/l.

The phosphates treated, in general, have a P205 concentration between 26 and 30% depending on the origin

(see appendix).

On a pilot scale, we have even been successful in converting phosphates with a low  $P_2O_5$  content and which are high in impurities (chemical analysis on page 31).

The yields obtained as well as the unit consumptions of sulphuric acid and phosphate are given in the table below (PAGE -8) for GAFSA phosphate.

% P <sub>2</sub> O <sub>5</sub> IN THE CRUDE PHOSPHATE AS SUCH	GUARAN- TEED TOTAL YIELD		CONSUMP- TION/T OF	H <sub>2</sub> SO <sub>4</sub> CON- SUMPTION/T OF P <sub>2</sub> O <sub>5</sub> PRODUCED
	IN %	DUCED IN % P205	PRODUCED	
> 29%	96	28	3.60	2.87
28.6 to 28.9	95.5	28	3.67	2.88
28.1 to 28.5	95.0	28	3.72	2.90
27.7 to 28.0	94.5	27.5	3.80	2.92
27.3 to 27.5	94.5	27.0	3.86	2.94

### ADVANTAGES OF THE PROCESS

Owing to its suitability for the conversion of low-grade phosphates with good yields, the S.I.A.P.E. process is known for:

a) - A simple design of its reactor and of its stirrers which ensure:

an automatic and intense natural circulation of the slurry in the reactor;

- an easy measuring out of raw materials;
- an intimate contact between the reactants;
- a foam destruction without the use of an adjuvant.
- b) Its ease of maintenance implying a long operational tife.
- c) Its low energy consumption.

In addition to these advantages and taking into account the operating conditions of the process, the investments required are not heavy.

### III - IMPROVEMENTS MADE TO THE S.I.A.P.E. PROCESS

Since 1975, phosphoric acid producers have met with problems of continuous deterioration in the quality of phosphate treated.

In fact, the  $P_2O_5$  content has dropped at the expense of the impurities which have substantially increased. This has manifested itself in a decrease in yield, in capacity and in the  $P_2O_5$  concentration of the acid produced.

In order to cope with this situation, S.I.A.P.E. has developed its process by the addition of an additional tank called the digestion tank.

This tank, the volume of which is 20% of the total reaction volume, receives the slurry by overflow from the monoreactor at the end of the peripheral ring and then returns it to the central treatment compartment.

The addition of this tank has significantly improved the crystallization, and consequently the filtration, maintaining the usual treatment temperature in the monoreactor at the same time.

The production capacity of the plant has been improved following the increase in the reaction volume. However, it should be noted that the specific reaction volume has remained constant, which confirms that the improvement achieved is a direct consequence of the temperature gradient of the slurry in the digestion tank.

Therefore, the addition of this tank requiring an additional investment which can be recovered in the short term due to the increase in production, has enabled the yields to be increased and the operation with all qualities of phosphate treated to be stabilized.

Further details may be found in the March 1982 paper (LEAFLET FROM CHEMICAL ENGINEERING PROGRESS).

OPTIMIZATION OF WATER CONSUMPTION IN THE S.I.A.P.E.

PROCESS

Within the context of studies on moistened phosphate with a view to the use beforehand of wet grinding in the phosphoric acid manufacturing plant of Industries Chimiques de GAFSA, trials have been carried out at Plant

"A" of S.I.A.P.E. in 1981.

The trials consisted in moistening the phosphate before its introduction into the reactor, at a rate of 30 to 35% by weight of water in the pulp.

The main difficulty to be overcome from the point of view of the process is the decrease in the quantity of water on the filter in order to maintain a constant overall water balance of the process for the plant as a whole.

It relates therefore, to moistening the phosphate to 35% water and thereby lower the water equivalent on the filter and to study the behaviour of the moistened phosphate in the reaction and the filtration stages.

These studies led to the following observations:

- a) \* A clear improvement in the filtration yield ranging from 0.5 to 1%.
- b) Maintenance of the treatment yield at its usual level, which consequently led to the improvement in the overall yield of the phosphoric acid plant.
- c) Increase in the mean strength of the recycled acid from 18 to 22% of P<sub>2</sub>O<sub>5</sub>, which manifests itself in a significant decrease in the output of the recycled phosphoric acid (from 2.8 to 2.1 m<sup>3</sup>/tonnes of phosphate).
- e) Improvement in the filtrability of the gypsum.

It should also be noted that the rotation speed of the filters decreased considerably, which enabled us to improve the mechanical performance of our filters and to lower significantly the gap between the concentration of the phosphoric slurry leaving the reactor and that of the acid stored. The capacity of the filters and, consequently, that of the plant, improved.

Moreover, it should be noted that the major part of the water used for washing the filter parts is recovered and used as water for the process.

### OPTIMIZATION OF THE ENERGY CONSUMPTION

A detailed study of the literature clearly shows the divergent viewpoints held by research workers with regard to the relationship between the reactivity of a phosphate and its physicochemical characteristics.

Some investigators agree that the main factor affecting the reactivity of phosphate is the external surface area of the phosphate, whereas others are of the opinion that particle size of the phosphate alone cannot be related to its reactivity index which depends on other important factors such as the chemical composition and the crystal structure of the ore.

A review of these studies therefore shows that the relationship between reactivity and particle size of phosphates is not yet well established and described in a suitable way.

Studies on reactivity have been carried out in our Plant on different varieties of phosphate which exist in TUNISIA.

Our studies were commenced with a first comparison of the particle size spectra of crude phosphates with those of the ground phosphate used in the phosphoric acid plant.

These spectra have revealed that, by grinding GAFSA phosphate, the increase in the fine particle size sizes is promoted, which, based on our experience in this field, favours an increase in the level of the co-crystallized material.

We know with certainty that at constant sulphuric acid level, the loss of co-crystallized material increases with the fineness of the phosphate.

Therefore, under conditions analogous to the manufacture of phosphoric acid from ground phosphate, the use of crude phosphate would enable the co-crystallized material formation to be decreased to some extent, with a small increase in the amount of material escaping treatment at the same time.

Based on these observations and considerations, we decided to try manufacturing phosphoric acid using

unground phosphate on an industrial scale. In fact, since October 1981, we have stopped grinding the phosphate.

The main results gathered over a trial period of three months lead to the following conclusions:

The total P205 losses using the unground phosphate are comparable to those obtained with the ground phosphate of the same quality, the chemical yields obtained therefore being comparable. In fact, although the losses due to the material escaping treatment were slightly greater with the crude phosphate, we have noticed a decrease in losses of co-crystallized material. This shows once again the antagonism which exists between the two forms of P205 losses.

The losses of washable P2O<sub>5</sub> were maintained at their usual levels, which enabled the filtration yield to be maintained.

The specific unit consumptions of raw materials remained constant.

In conclusion, these studies have enabled us to show clearly that our process is capable of treating GAFSA phosphates in the crude state for the manufacture of phosphoric acid without problem, with good yields. This innovation has contributed to the omission of the grinding plant, thus improving two important factors:

An energy saving, estimated approximately at 35% per tonne of  $P_2O_5$  produced.

A significant increase in the operational life of the phosphoric acid plant (fewer turning mechanical parts); it goes without saying that the working conditions in the phosphoric acid plant are also improved following the omission of the grinding section.

Finally, we believe that this improvement introduced to the process since 1981 is closely related to the use of moistened phosphate.

### INCREASING THE PRODUCTION CAPACITIES OF NEW UNITS

The mastery of an effective know-how in the field of phosphoric acid manufacture by S.I.A.P.E. and the

existence of a planning department with experience in the field enabled us to design large-capacity monoreactors.

In fact, the first S.I.A.P.E. monotank was designed only for the treatment of a capacity of 320 tonnes of  $P_2O_5/day$  whereas at present, we are able to build monotanks of a capacity ranging up to 1,000 tonnes  $P_2O_5/day$ .

By way of example, the two phosphoric acid manufacturing units currently under construction at SKHIRA are designed for treating (650 x 2) tonnes of  $P_2O_5/day_+$ 

In conclusion, it is seen from this paper that the S.I.A.P.E. process progresses in a very positive way at all levels, the results of some improvements presented here result from the efforts of all the staff of S.I.A.P.E. who have contributed to the various stages of the development of this process.

The staff have acquired a wide experience in the field of the manufacture of phosphoric acid in particular and that of fertilizers in general.

Our well-experienced staff who are often invited to give technical assistance, in TUNISIA as well as ABROAD, will, in future, contribute further to the development of our process which has been at the origin of the increase in the capacity of phosphate conversion in TUNISIA. In fact, the conversion capacity has increased from 500,000 tonnes in 1965 to approximately 4,000,000 tonnes in 1985 (SEE APPENDIX).

The mining capacity has followed the rate of conversion which has also enabled the mining sector to expand over the last twenty years.

### B - EXPERIENCE IN REVAMPING

#### I - INTRODUCTION

The starting of Plant "A" of S.I.A.P.E. in 1952 and that of Plant "B" (formerly N.P.K.) in 1964 and their up-keep and satisfactory operation since commissioning has led to the acquisition by TUNISIA of an experience in the "revamping" of phosphoric acid and T.S.P. units. In fact, the nominal capacities of both these Plants have almost doubled since their beginning, because of the improvements and the upkeep and renewal undertakings.

See Appendix: TABLE OF PRODUCTIONS OF PLANTS "A" and "B".

In addition to the local experience in the field of renovation, operations have been undertaken on an international scale.

### THE CASE OF S.I.A.P.E. PLANT B IS DESCRIBED BELOW

After the Tunisification, a group of TUNISIAN experts carried out a study of this plant and put forward some recommendations to increase the production capacity of this Plant.

# 1) - INITIAL CONDITION OF THE UNITS BEFORE RENOVATION:

The maximum production which could be achieved during the first 10 years of this plant is:

- a) H<sub>2</sub>SO<sub>4</sub> UNIT...PRODUCTION 167,598 T/YEAR
- b) P205 UNIT....PRODUCTION 60,579 T/YEAR
- c) T.S.P. UNIT.....PRODUCTION 169,119 T/YEAR
- d) UTILITIES: TWO UNITS OF 2,400 KW/h EACH.
- 2) SOLUTIONS RECOMMENDED FOR IMPROVING PRO-

### a) - Hosou Plant

increasing the capacity of the recovery boiler;

increasing the capacity of the blower; acquisition of a new acid distributor in the towers;

installation of droplet separator for the drying and absorption towers; installation of a tubular type condenser

installation of a tubular type condenser system;

increasing the capacity of the sulphur oven; increasing the quantity of the catalytic mass.

### b) - PHOSPHORIC ACID PLANT

installation of diluters for sulphuric acid from 98% to 75% enabling a certain amount of heat to be removed and reducing the load on the reactor degassing fan; acquisition of two cumulative weight meters in order to improve phosphate feeding into

the unit; change of the slurry and the gypsum pumps with a type of modified pumps; overhauling of the reactors, the filters and accessories.

### c) - T.S.P. PLANT

installation of a sea water and phosphoric acid gas scrubbing station enabling the acid concentration to be increased; acquisition of a new type of sieving unit.

### d) - UTILITIES

installation of a third turbine of capacity 5 MW/h acquisition of a new auxiliary boiler of capacity 18 T/h.

### 3) - RESULTS OBTAINED

As a result of the renovation of the different units, the following capacities were achieved:

a - H<sub>2</sub>SO<sub>4</sub> UNIT: 251,255 TONNES/YEAR

b - PoOs UNIT: 82.917 TONNES/YEAR

c - T.S.P. UNIT: 232,015 TONNES/YEAR

Besides increasing the capacity, these investments enabled environmental pollution to be reduced by trapping dusts and harmful gases such as fluorine and  $SO_2/SO_3$ .

In fact, the renovations of the S.I.A.P.E. Plants "A" and "B" enabled the objectives fixed to be achieved, the production has been regular during the last five years and has been maintained at its maximum.

In order to succeed in such an undertaking, several requirements should be met:

a long experience in the field of fertilizer manufacture;

availability of a well-established and efficient
know-how;

availability of a planning department with experience in the field;

availability of well-experienced staff for managing the various installations and the various processes.

It should also be noted that other firms have also asked for the assistance of S.I.A.P.E. for the renovation of their Plants. This enabled us to make use of our experience and our technology in order to develop international cooperation.

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YEAR	77	78	79	80	81	82	83	84	, <b>8</b> 5
P <sub>2</sub> 05 %	28,91	28,66	28,75	28,67	29,14	29,18	29,05	28,98	28,47
CaO %	49,75	48,92	49,33	48,97	49,54	49,86	49,87	49,56	48,63
MgO %	0,84	1,03	1,05	0,89	0,91	0,52	0,72	0,54	0,78
so4 %	4,54	4,69	4,68	5,06	4,76	4,77	4,75	4,44	4,38
S102 %	3,58	4,12	3,25	3,57	2,92	2,92	2,98	3,31	3,67
Fe 203 %	0,30	0,33	0,28	0,30	0,20	0,22	0,26	0,34	0,36
e 203 %	0,59	0,55	0,60	0,47	0,13	0,25	0,27	0,54	0,88
ro2 %	6,10	6,18	6,23	7,02	6,48	5,97	6,45	6,29	6,43
7 7	0,102	0,09	0,08	0,09	0,04	0,05	0,06	0,05	·
F %	3,66	3,67	3,76	3,66	3,75	-	3,88	3,84	3,49
1.0 %	1,32	1,42	1,05	1,17	1,04	0,95	1,12	1,11	1,11
P. 1000 %	10,43	10,44	10,47	10,98	10,66	11,00	11,15	11,31	11,78

YEAR	1978	1979	1980	1981	1982	1983	1984	1985 ′
P205 %	28,84	28,63	28,42	28,56	28,38	27,98	27,71	27,59
Ca0 -	48,88	48,82	48,42	48,46	48,44	48,08	47,90	47,42
Mg0 -	1,67	1,34	1,53	1,04	1,19	1,15	0,88	0,91
s04 -	3,77	3,72	3,68	3,78	3,60	3,60	3,91	-
Si02 -	3,85	3,41	4,08	3,75	3,91	3,98	4,75	4,73
Fe203-	0,37	0,34	0,28	0,28	0,28	0,28	0,44	0,49
A1203 -	0,75	0,94	0,37	0,37	0,20	0,38	0,81	1,17
C@2 -	6,21	6,24	7,03	6,59	6,25	6,95	6,47	6,69
c1 -	0,02	0,01	0,01	0,01	0,01	0,01	0,04	
F -	3,56	3,56	3,66	3,65	_	3,86	3,74	3,49
М.О	2,74	2,06	2,23	2,13	2,43	2,51	2,32	2,26
P. 1000°	10,74	11,24	11,63	11,17	11,80	11,64	9,79	-

5 - 18

Year Oversize (openings in	77 jum.)	78	79	80	81	82	83	84	85
2 1 0,5 0,3 0,2 0,125 Passe	2,7 6,0 11,0 17,6 32,7 79,1 20,9	4,4 9,4 16,0 23,0 40,0 85,0	3,0 6,8 12,7 18,5 36,6 75,9 24,1	2,1 5,2 11,7 18,5 34,3 69,1 30,9	2,1 4,8 10,2 15,2 31,9 75,8 24,2	2,0 4,7 10,2 17,0 32,4 71,6 28,4	3,8 7,4 12,8 21,1 41,0 86,1	4,6 8,7 14,2 22,0 42,1 89,3 10,7	5,2 10,4 16,9 25,4 44,7 87,6

Year Oversize (openings in	78 mm.)	79	80	81	82	83	84	85
2	0,4	0,2	0,3	0,5	0,3	1,1	2,0	1,8
İ	2,0	2,2	2,1	2,6	2,0	3,7	6,6	6,2
0,5	8,9	8,7	10,1	10,6	9,t	11,3	15,9	15,9
0,3	18,5	17,1	20,6	19,8	20,0	23,9	28,7	31,2
0,2	37,4	37,1	37,2	38,6	36,3	42,6	47,3	51,6
0,125	69,0	63,5	61,2	69,8	61,6	72,2	80,3	80,5
Passe	31,0	36,5	38,8	30,2	38,4	27,8	19,7	19,5
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TABLE 5

# CHEMICAL COMPOSITION OF LOW GRADE PHOSPHATE ROCK HIGH IN IMPURITIES

P205	*	 24.8
Ca0	8	 43.34
S03	ŧ	 1.13
C1	*	 0.02
Fe203	*	 2.81
A1203	*	 0.88
МдО	*	 1.57
SiO2	8	 18.34
CO2	*	 3.34
ORGANIC MATTER	*	 _
MOISTURE	8	 0.21

### ANNEX

SOCIETES DU GROUPE CHIMIQUE TUNISIEN COMPANIES OF THE TUNISIAN CHEMICAL GROUP AND TYPES OF PERTILIZER PRODUCED IN TUNISIA:

- S.I.A.P.E.

: 570,000 T/YR TSP (47% P205)

- I.C.M.

: 430,000 T/YR H3PO4 (54% P2O5)

100,000 T/YR TSP (39% P205)

60,000 T/YR DCP (DICALCIUM PHOSPHATE)

- S.A.E.P.A.

: 330,000 T/YR H3PO4 (54% P2O5)

330,000 T/YR DAP

330,000 T/YR AMMONIUM NITRATE

- I.C.G.

: 400,000 T/YR TSP (47% P205)

- S.T.E.C.

: 30,000 T/YR SINGLE SUPERPHOSPHATE 10,000 T/YR COMPOUND FERTILIZERS NPK (6-8-8 & 10-10-20)

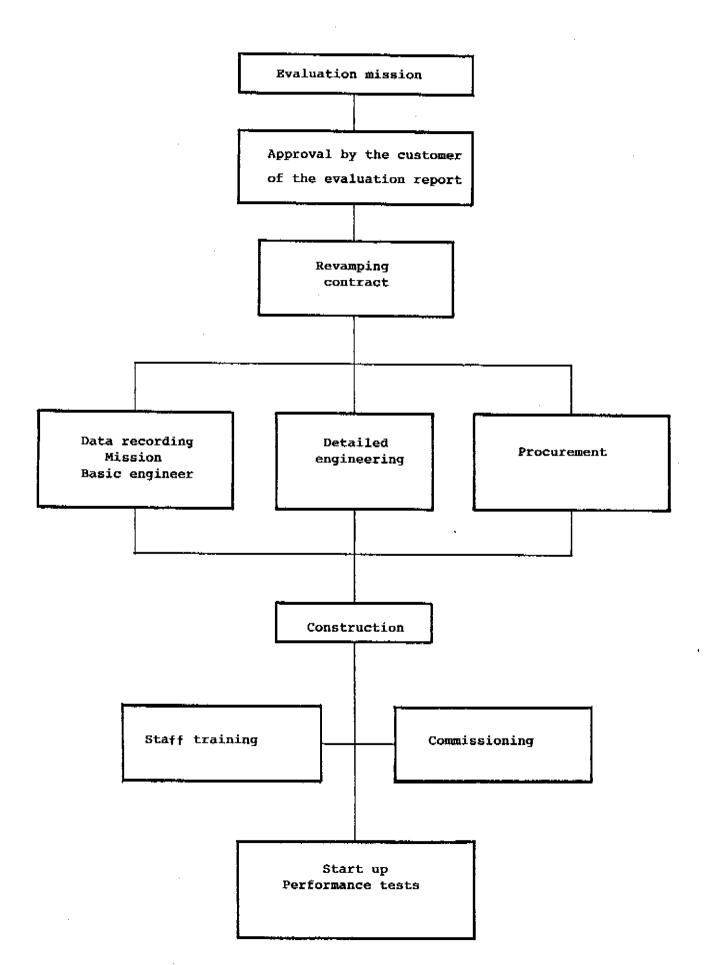
- E.G.

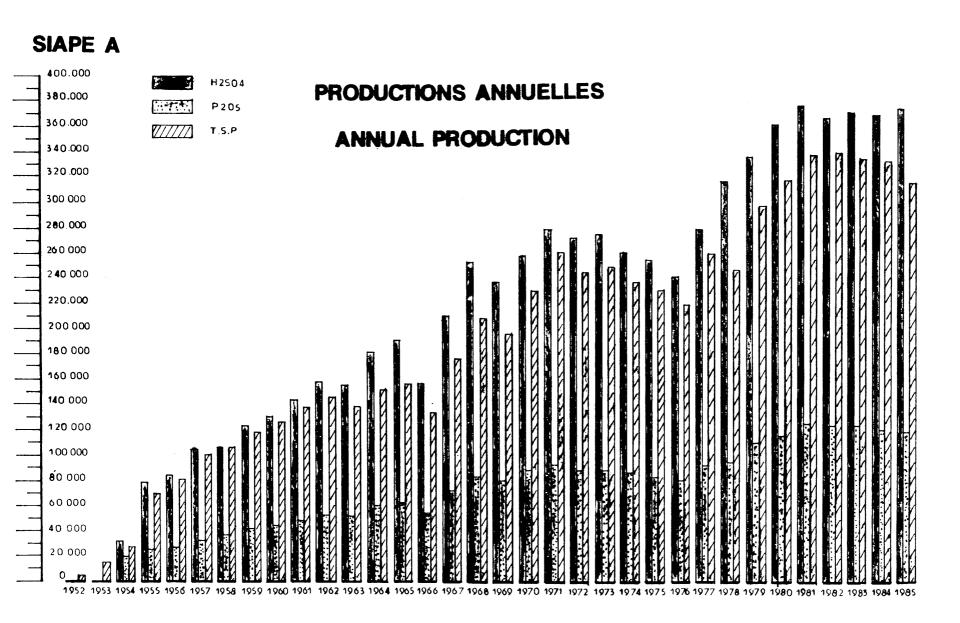
: 400,000 T/YR DAP OR 500,000 T/YR NPK (17-17-17 ; 24-24-0 ; 12-24-12 ; etc...)

- EK KIMIA

: 25,000 T/YR STPP (SODIUM TRIPOLYPHOSPHATE

### REVAMPING PROCEDURE





H2S04

P2 05

SIAPE B

V#///##/

0000 **80 00**0

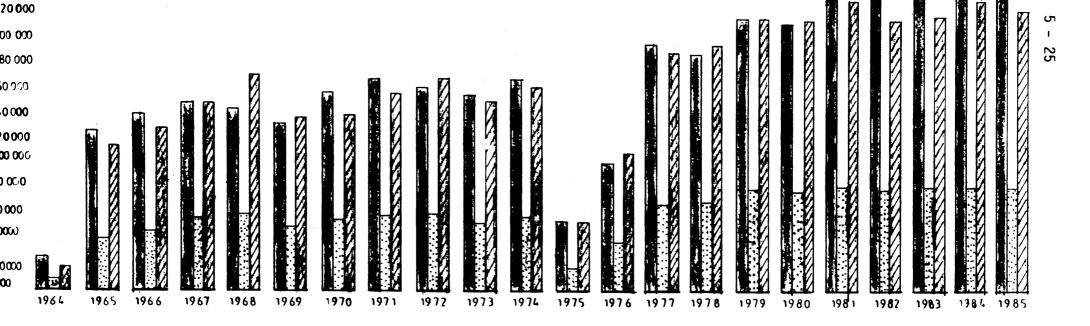
60000 40000

**0**0

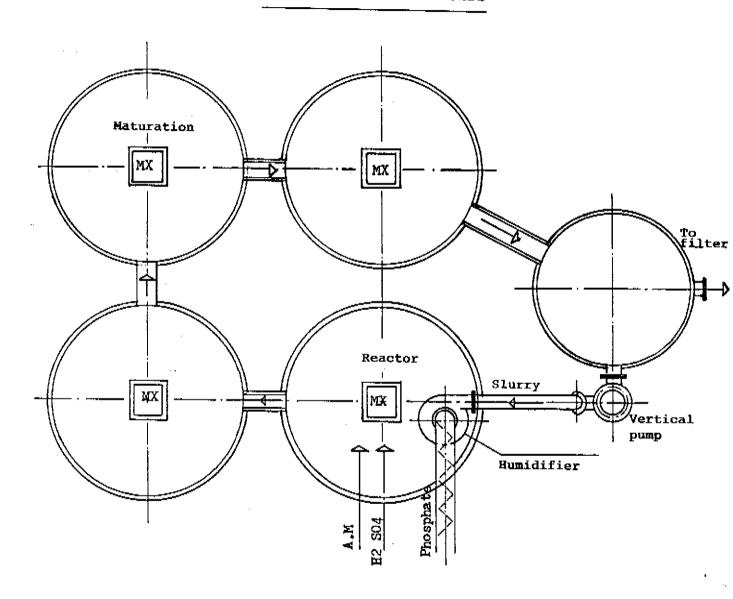
3: 1:

TSP

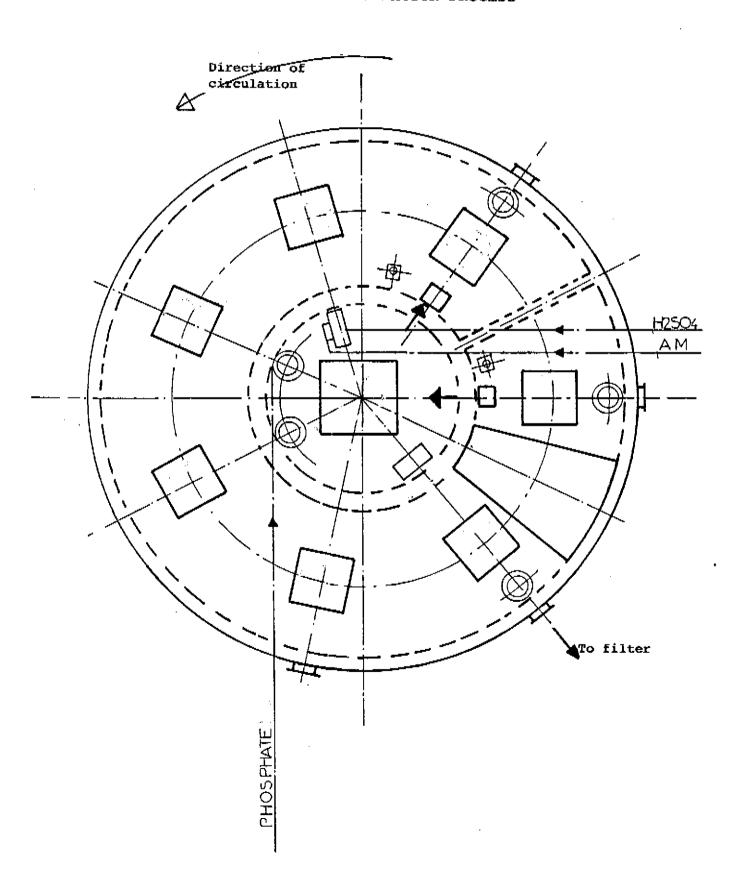
# PRODUCTIONS ANNUELLES ANNUAL PRODUCTION

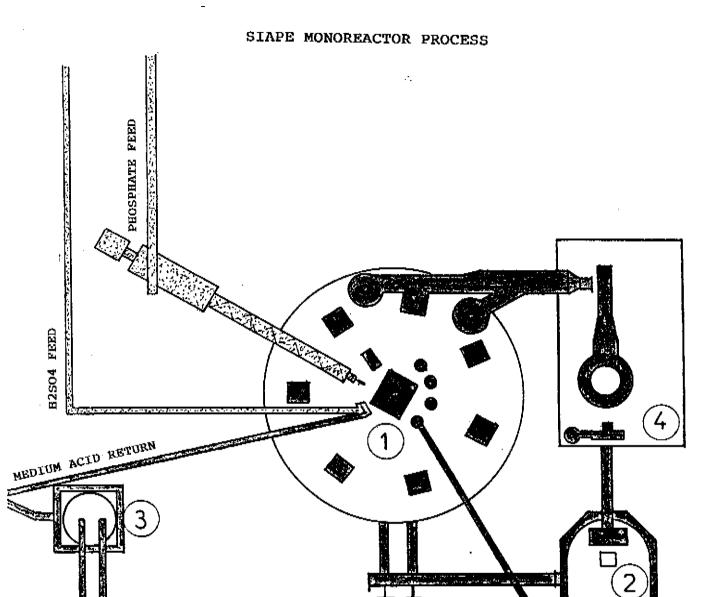


# SIAPE MULTITANK PROCESS



### SIAPE MONOREACTOR PROCESS





- 4 Deaeration station
- 3 Filter feed tank
- 2 Digestion tank
- 1 Reactor

- TA/86/5 Thirty years of the SIAPE process for the production of phosphoric acid by the wet process and experience in the revamping of phosphoric acid by E. Essaba & Y. Louizi, SIAPE, Tunisia
- DISCUSSION: (Rapporteurs: Messrs M. Barloy, SCPA, France & A. HAMDI, SAEPA, Tunisia)
- Q Mr. M. BARLOY, SCPA, France
  - a) When stating that the concentration of free sulphuric acid is kept between 0.8 and 1.2%, do these values include gypsum dissolved in the acid?
  - b) In the P205 losses, can you indicate the distribution between unreacted P205, cocrystallized P205 and soluble P205?
  - c) In the rock phosphate, can you indicate the particle size of the phosphate used and the size which you consider as optimum?
  - d) Is the solid content characteristic of the phosphate or the process?
- A a) The total free sulphuric acid concentration of the slurry is indeed between 0.8 and 1.2%.
  - b) The total P205 losses can be split as follows:
    - unreacted P205 : 1-2 g/kg dry gypsum
    - cocrystallized P2O5 : 6-7 g/kg dry gypsum
    - soluble P205 : 2 g/l gypsum solution corresponding to a filtration recovery of 98-98.5%.

These figures depend upon the quality of the phosphate processed and on its P205 content.

- c) Particle size of the rock phosphate used:
  - 4 to 10% above 1 mm
  - 70-90% higher than μ125
- q) The solid content is not a constraint of the process; for Gafsa phosphate, the content is about 30%. For other phosphates it can vary; the reactor can accommodate solid contents of up to 40%.
- 4 Mr. J.D. CRERAR, Norsk Hydro Fertilizers Ltd, United Kingdom

The improved SIAPE process showed the benefit of an additional compartment in the reactor system (digestion tank). What further advantages are possible by more compartments?

A - No, we do not intend to come back the multi-compartment system we described in the first part of our paper. The addition of a digestion tank, which first increased production, improved the overall efficiency of the phosphoric acid plant (improvement of dihydrate gypsum crystals confirmed experimentally).

Indeed, the recycling of a cooled slurry from the additional tank to the central compartment have a favourable effect on the filtration efficiency que to better crystallization. The digestion tank was built and tested satisfactorily in our plant "A". It was adopted in the I.G.G. plant which started in 1985 and in SIAPE II under construction.

For further information see the paper we presented at 1980 IFA Technical Conference and to the journal "Aiche issue of Chemical Engineering Progress".

Q - Mr. N. HUMMADI, JPMC, Jordan

It was mentioned that moisture content in the rock used in one of SIAPE plant is 35% H2O.

- a) What effect does this moisture content have on the overall P205 recovery?
- b) Did you experience any problem in the control of SO4 in the reactor due to above mentioned moisture content?
- A a) The humidification of phosphate rock up to 35% moisture has no detrimental effect on the overall P205 recovery. Indeed we noticed that, by doing away with the grinding, we avoided the fine fractions responsible for cocrystallized losses. By moistening the unground rock to 35%, the overall recovery is kept constant compared to a rock supplied in dry and ground form.
  - b) As a result of phosphate humidification, we did not find problems of sulphate control in the reactor. We always have the same values as with dry phosphate. Indeed the water balance introduced in the reactor remained constant since the water used for moistening came from the filter.
- Q Mr. P. BECKER, COFAZ, France

Influence of the humidification of unground rock on the coating process (during phosphoric reaction)?

A - We do not notice any coating phenomenon. The process water balance at the reactor inlet remained altogether constant.