International Fertilizer Industry Association

ISMA* Technical Conference

Vienna, Austria

11-13 November 1980

In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).

IMPROVEMENT OF SIAPE PROCESS FOR THE MANUFACTURE OF PHOSPHORIC ACID IN ORDER TO PROCESS LOW GRADE PHOSPHATES WITH HIGH IMPURITY CONTENT

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INTRODUCTION

Phosphoric acid producers are increasingly confronted with the continuous and regular fall in P_2O_5 grade of phosphate rock and at the same time with the increase of their impurities content.

This is a very acute situation for the local processing industry which is exclusively supplied with phosphate extracted on the spot.

The gradual loss of quality is due, on the one hand, to the intensification of extraction which necessitates the exploitation of less rich layers and, on the other hand, to the fact that the most elaborate merchant qualities are preferably destined for exports.

Thus, the search for new solutions becomes necessary in order to adapt the existing facilities to the developments of quality of the basic material which is phosphate rock.

It was the duty of SIAPE, which owns the process of phosphoric acid with dihydrate, a process exclusively used in Tunisia, to undertake researches and to build the first improved facility which will be able to process the available rock phosphate.

The use of a lower grade phosphate than that provided for in the master data according to which phosphoric acid units were built, usually means an important decrease:

- In P_2O_5 extraction ratio from the phosphate rock.
- In P_2O_5 concentration of the manufactured phosphoric acid.

The mono-reactor of SIAPE's plant "A" which was brought into service in June 1973, has a nominal capacity of 300 tons P_2O_5/day on the basis of Gafsa rock processed by washing or by ventilation, with 65 BPL grade, that is to say approximately 29.8% P_2O_5.

However, the grade of this quality rock has been decreasing in the recent years dropping down to 27.5% P_2O_5 with an increase of CO_2 contents rising from 5.5 to an average of 6.5 of silica, which rose above 3.5% and of MgO which ranges between 0.7 and 1%, bringing about a fall in output which may reach 2% and a dilution of the produced phosphoric acid ranging from 1 to 2%.

PRINCIPLE

The difficulties arising from the processing of such phosphates increased with the fall in P_2O_5 grade and with the increase of the impurities content.

Attack ratio has been decreasing and the filtration capacity which was of 3 t P_2O_5/m^2/day dropped to 2.5 t P_2O_5/m^2/per day.

The numerous tests carried out in SIAPE plant allowed us to ascertain that we can process low grade phosphates containing embarrassing impurities by lowering the grade of produced acid and by increasing the slurry temperature when it reaches the filter. It is for this reason that the slurry temperature when it reaches the filter ranges from 78 to 80° C.

However, this high temperature would have been incompatible with the formation of crystals of high quality filtration which normally necessitates a temperature gradient between the attack point and the slurry on the filter.

Therefore, it was interesting to try to perfect a combination of two very important factors for the filtration, namely:

- Reaching a temperature gradient between the temperature of the recycled slurry and the slurry in the attack compartment..
- Maintaining a sufficiently high temperature on the filter to improve filtration.

Our researches led us to argue from analogy: when we realized that the drop of the grade could lessen the influence of the diminishing phosphate quality we thought that the lowering of the slurry temperature could have a similar result, since grade and temperature do condition the stability of dihydrate crystals.

For the swelling of gypsum crystals produced in the monoreactor on the basis of this quality phosphate, we realized that an external slurry supply with a well-crystallized and stable dihydrate gypsum was necessary.

Therefore, the question is to cool a part of the slurry in circulation with a temperature gradient of a minimum 5° C in order to obtain a completely demulsified slurry containing very stable dihydrate crystals which have the most adapted dimensions to the filtration that we reset in the attack compartment, thus bringing highly developed germs.

Thus, we have been led to build a supplementary tank that we will call "Digestion tank", although the term is not very appropriate; this "Digestion tank" is designed to improve the trituration and to cool a part of the slurry in circulation in the principal reactor.

The slurry is taken at the end of the monoreactor peripheric compartment at the temperature of 78-80° C and returns in the attack compartment with a much lower temperature of 72-73° C. The slurry filter is always, in this case, supplied by a hot slurry of 78-80° C taken from the same place than before.

This entails the use of two rates of recirculation:

- One is internal, the temperature of which is close to that of the slurry reaction.

- The other one is more modest and by an external means with a rather high temperature gradient.

This method enables us to improve gypsum crystallization while keeping the temperature at its usual level in the monoreactor and without having recourse to dilution. The obtained outputs by this system are equivalent to those obtained by the processing of the usual phosphate in the principal reactor which functions by itself.

Production capacity of the facility increased proportionally to the increase of the volume brought by this new digestion tank.

This confirms the fact that the increase of the reactive volume is not itself responsible for the improvement of the outputs. The temperature gradient undergone by the slurry extracted from the monoreactor is the cause of this improvement.

The reactive specific volume remains the same, that is to say 1.5 $m^3/t/P_2O_5/$ day.

The regular fall of the grade of phosphates used and the increase of impurities are put under control by the improvement of SIAPE process. This method will be recommended to our licenced phosphoric acid producers likely to process new qualities of phosphate.

This solution calls for a supplementary investment, but it is compensated by the corresponding gain in production.

For an existing facility, the gain of 1% in the output allows by itself the recuperation in one year of the whole amount invested for the realization of this complementary facility.

DIMENSIONING OF EQUIPMENT

The test undertaken by SIAPE led us to give a dimension of the new tank on the basis of the reactive volume increase of 20% which means a production of 60 t/P_2O_5/day.

RECKONING

- Nominal capacity of the monoreactor= 300 t/P_2O_5/day
- Reactor volume: 450 m^3 useful
- Specific volume: 1.5 $m^3/t/P_2O_5$/day
- 20% increase of the nominal capacity corresponding to 60 t P_2O_5/day
- Necessary reactive volume for this increase: 60 X 1.5= 90 m^3
- Supplementary calories to be evacuated for a phosphate of 60 BPL: 220 thermies/t of used phosphate

- 9.5 t/h of phosphate= 2.090 thermies/h
  $2.09.10^6$ Kcal/h

The output rate of the chosen recirculation pump is 450 m^3/h.

The specific heat of the slurry if 0.6 Kcal/°Ckg

$2.09.10^6 = 4.50\ 10^3 \times 1.5 \times 0.6\ X$

$2.09.10^3 = 405\ X$

$X = T. = \frac{2.09.10^6}{405.10^3} \neq 5$

The slurry which returns to the compartment must have a temperature of 73-75° C..

We must consider the instantaneous rate of the facility by taking into account the time needed for washing and maintenance equivalent to 0.8% of the needed time - 360 x 1.08= 388.8 t P_2O_5/day.

The temperature gradient is 5° C. The experience we have from cooling by air sweeping enables us to fix on the necessary air flow, that is 25 000 Nm^3/h.

For the measuring of air flow, we have to take into account the ratio surface/volume, the stirring power, temperature and humidity of the air and and a depression of 50 mm of water.

The additional reactor is supplied by a system of communicating vessels starting from the principal reactor and at the end of the ring at a point where the slurry is ripe.

After being stirred up and cooled, the slurry is recycled in the central compartment or attack compartment by a centrifugal pump with a very low manometric height and a very big flow.

The characteristics of the additional tanks manufactured in SIAPE plant are the following:

DESCRIPTION:

The necessary useful volume for the additional tank is theoretically of 90 m^3; the volume which is practically used has been of 110 m^3 and has a shape of a tank with an oval section; we chose this shape to avoid the dead zones: it can be easily adapted to an agitation composed of two movers whose interaction allows the creation of a strong turbulence likely to favour an optimum stirring.

The agitation system contains two agitators of a SIAPE type with two rows of blades turning 110 times per minute and each one consumes a power of 31 kw, that is 0.56 kw for 1 m^3 of slurry.

The superior blades are adjusted on the height to the surface of the slurry in order to beat the foams and to favour the thermic exchange between the slurry and the sweeping air.

A ventilator with a rate of 25 000 Nm^3/h and of a total pressure of 120 mm CE allows cooling of the slurry.

The tank is supplied by an overflow from the principal reactor at the end of the ring with the help of a system of communicating vessels. The inlet in the digestion tank is performed in the lower part.

Once stirred up and cooled, the slurry is pumped by a horizontal centrifugal pump with a low manometric height to be recycled in the central compartment of the monoreactor where the attack of phosphate by the mixture sulphuric acid-phosphoric acid is performed.

PRACTICAL RESULTS

The system was brought into operation in April 1979 and allows the attainment of the expected results.

The effective temperature gradient is from 7 to 8 degrees.

Although difficult, the observation of crystals enabled us to note the absence of an important quantity of small crystals which usually hinder the filtration. This ascertainment is confirmed by the fall of the ratio of solids in the produced acid.

COMPARATIVE TABLE FOR 28% P_2O_5 PHOSPHATE ROCK:

a) Operating rate - Acid grade - Output - Filtrability

	Monoreactor only	Monoreactor with digestion tank
Operating rate	320 t P_2O_5/day	400 t P_2O_5/day
Acid grade in P_2O_5	26.5 to 27%	27.5 to 28.5%
Output	93.5 to 94.2%	98.8 to 95.2%
Filtrability	2.7 t/m^2	3.3 t/m^2

SLURRY CHARACTERISTICS:

b) Reactor running at nominal capacity without digestion tank (320 t P_2O_5/day)

SLURRY DENSITY ON THE FILTER: 1.370 - 1.390

c) Reactor running with digestion tank (rate of 400 t P_2O_5/day)

	Supply of Digestion tank	Digestion tank Output	Supply of filter
Slurry density	1.26 - 1.33	1.42 - 1.47	1.38 - 1.43

d) Solid content of the produced acid:

Without digestion tank: Average of 1.5%

With digestion tank: Average of 1%

The digestion tank can also ensure a working stability whatever the quality of the used phosphate: it avoids the temporary misadjustments due to a misadjustment of titration.

Moreover, the possible interventions on this tank do not hamper the working of the principal reactor and it can be stopped at any moment without hindering the normal operation of the principal reactor after reducing its rate.

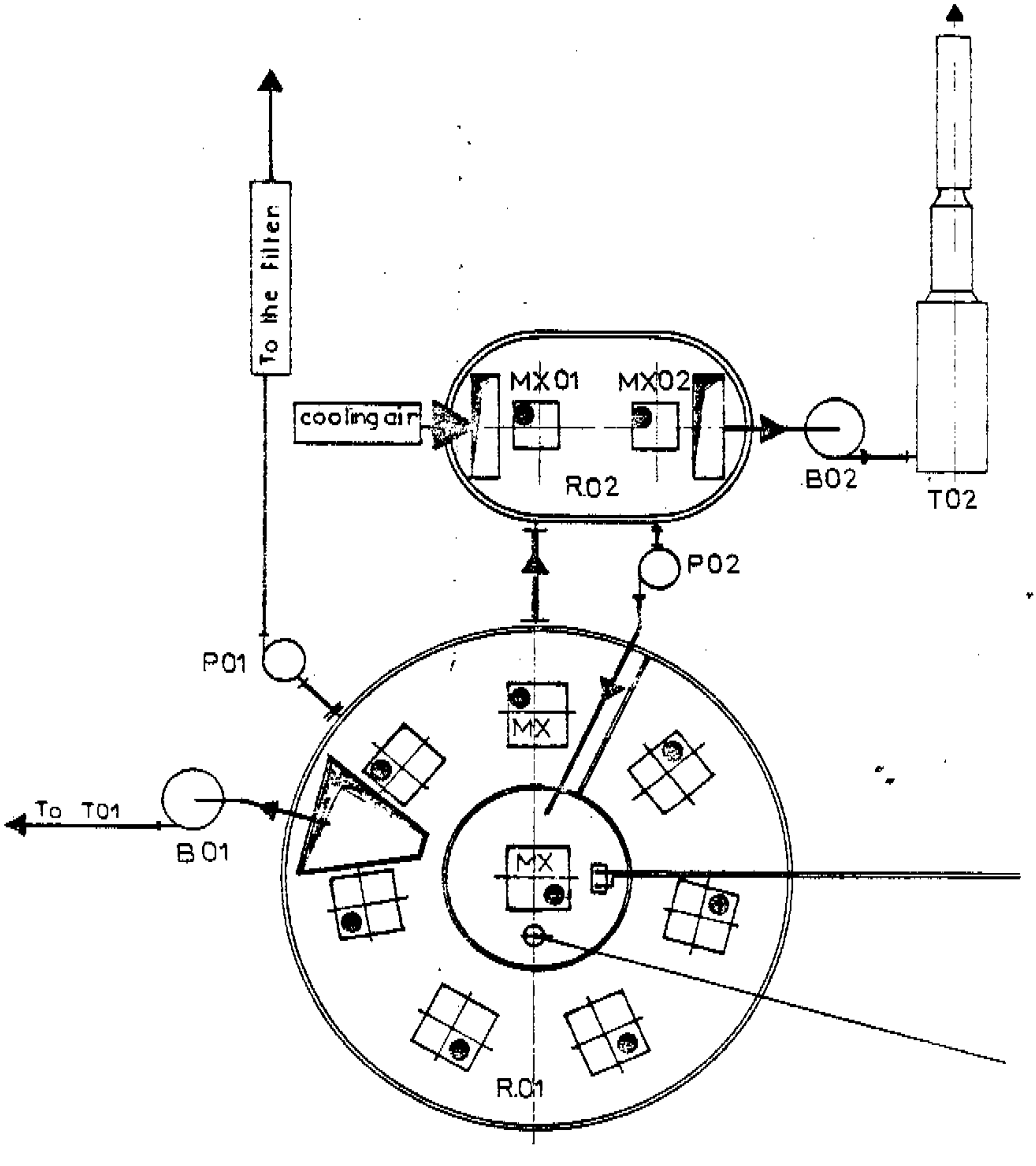
A tank of this type can also be devised to solve the different problems encountered during the exploitation of the phosphoric acid dihydrate unit.

For high-quality phosphates over 65 BPL, the "digestion tank" can be considered as a simple means to increase the daily output from 20 to 30% without intervening in the main equipments, in this case the efficiency output of P_2O_5 is maintained above 96% in most cases.

Some qualities of phosphate rock have high grades in P_2O_5 but are little reactive. In this case, the tank is an easy means to increase the volume of reaction to the required values offering an advantage for a better control of the reaction.

Also for the units which use anti-foaming agents, a tank of this type can be built in order to destroy mechanically the foams and to avoid high expenses arising from exploitation.

SIAPE	Phosphoric Acid Unit	N°
SFAX TUNISIE	Additional tank	



R01 _ Principal Reactor
R02 _ Additional tank
MX01/02 _ Agitators
B02 _ Fan
T02 _ Wasber
P02 _ Pump

REV											
DATE	26.6.80										

TA/80/5 Improvement of SIAPE process for the manufacture of phosphoric acid in order to process low grade phosphates with high impurity content by SIAPE, Tunisia

DISCUSSION : (Rapporteur F. Thirion, Soc. de Prayon, Belgium)

Questions

Mr. P. BECKER, COFAZ, France

1) What is the corresponding investment for a medium size plant?

2) When building a new plant, is it possible to include this integrated improvement in a single tank?

Mr. P. MORAILLON, Génerale des Engrais, France

1) Is the slurry recycled between the central compartment and the peripheric compartment of the main reactor? How is this reactor cooled?

2) As a counter-test, did you try to feed the filter from a secondary cooling reactor?

Mr. G. de la CROIX, Coppée-Rust, Belgium

Did you establish a reliable measured correlation between the power used by the agitation and the efficiency of the reaction and/or of the filtration? If so, is this correlation not more dependent on the type of agitation than of its power?

Mr. R.F. BARUT, Heurtey Industries, France

1) What do you think of the use of a vacuum cooler as secondary reactor?

2) Can you indicate the impurity content in the two cases: with and without a secondary reactor?

Mr. M. BARLOY, CERMI, France

1) You state that the addition of a tank improves the efficiency of crystallization. Do you mean in terms of soluble, co-crystallized, unattacked P_2O_5?

2) If in an existing plant we only look for increased efficiency, is it not more simple to add a flash cooler?

3) In a new plant, would you systematically add a second tank?

Mr. T.J. PEARCE, Estech Chem. Corp. United States

On table, page 5-5, Output - Monoreactor with digestion tank data given 98.8 to 95.2%. Is this correct or should it be 93.8 to 95.2%

Mr. N. ROBINSON, Fisons Ltd, United Kingdom

One of the advantages of the multi-reactor system is that it allows the gypsum crystals to stabilize after reaction before the slurry is recycled to the rock addition point. This usually reduces the particle size distribution and improves gypsum filtrability.
The paper gives no details on the shape and size of the gypsum crystals.

1) What changes in crystals occur as a result of using the two recycle slurry flows?

2) How critical is the 5° temperate drop? What happens if this drop is 10°?

Mr. F. THIRION (Société Industrielle de Prayon, Belgium)

In your plant, do you use additives for the reaction or the filtration? If so, what are they?

Summary of the answers

The output of the monoreactor is improved both in the reaction and in the filtration and the filtrability of the slurry is increased from 2.7 to 3.3 t P_2O_5/m^2/day.

These improvements result from a double recycling of the slurry: the first, very important, from the central to the peripheric compartment; the second, less important (450 m^3/h), from the digestion tank to the central tank.

The main difference in the characteristics of these two recyclings result from the fact that the temperature differential between the central and the peripheric compartments is t = 0.5 to 1° C and that the temperature differential between the digestion tank and the central compartment is t = 8° C.

Although it is difficult to realize it in the microscope, the improvement is remarkable:
- on the one hand, the filtrability of the slurry went up from 2.7 to 3.3 t P_2O_5/m^2/day,
- on the other hand, the solid content of the acid produced went down from 1.5 to 1%, which results in a decrease of the number of small crystals which usually hinder the filtration.

In the reaction stage of the SIAPE process no additive has ever been used. Regarding the additives to the filtration, they have been reduced by 70% since the start-up of the digestion tank.

The cooling system used in the SIAPE process is an air cooling through forced ventilation combined with an adjusted agitation. We believe that this system is easier to operate than a pump evaporator.

The filter is fed from the main reactor. Initially, the idea was to feed the filter from the digestion tank, but the temperature of the slurry, 72-73° C is a handicap, in view of the viscosity of the slurry, due to organic matter.

No correlation was established between the power used by the agitation and the output of the reactor or the filter.

Concerning the solubilization of ferral, the analysis does not show any noticeable variation in the acid produced (filtered).

	Actual monoreactor	Monoreactor with disgestion tank
Fe_2O_3	0.22 - 0.23%	0.2 - 0.21%
Al_2O_3	0.46 - 0.53%	0.44- 0.50%
Acid grade	27% (average)	28% (average)

Ferral is not an important impurity in Tunisian phosphate.

The plant built in 1978, associated with a 300 t P_2O_5/day plant, cost US Dollars 400-450,000. For a 500 t P_2O_5/day plant, it would amount to US Dollars 550-600,000.

For a new plant, we consider integrating this improvement with a monoreactor whenever the phosphate rock has an irregular quality, a low grade or a high impurity content.