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TECHNICAL AND ECONOMICAL EVALUATION OF A RAW PHOSPHATE ROCK FOR THE PRODUCTION OF FERTILIZERS

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SUMMARY

This is a description of a methodology, set up at the Fertilizer Research Centre of Montedison at Porto Marghera, for the evaluation of a phosphate rock in relation to its use in the production of wet phosphoric acid, phosphoric fertilizers and complex fertilizers by nitric attack.

The method foresees, apart from a chemical and physical-chemical characterisation of a rock, the carrying out of laboratory tests from which the consumption of raw material, the characteristics and the grades of the intermediate and final products, and the process efficiencies, for the various cycles of production, are determined.

For the execution of the various tests 200 kilograms of sample are sufficient.

The results obtained, besides defining how to use the rock tested in the various processes, are also useful for an economical evaluation of the rock; such an evaluation is expressed as a ratio of costs as compared with a rock normally used, taken as a reference, which in our case is Florida Land Pebble 73 BPL.

1) INTRODUCTION

Each time, in a plant for the production of fertilizers, one considers the possibility of using a different type of rock from that normally used; the problem arises of estabilishing if the new rock will serve its purpose, if and how the process conditions of various production cycles should be modified, and also if there will be a different cost related to the change of raw material.

These problems have become more and more important in recent years, since when price increases on the World Market, encouraged more favourable consideration towards rocks which were more economically convenients even if the quality was inferior to those used previously.

For these reasons minerals deposits have been reconsidered and exploited, which previously were thought to be of little interest for the production of fertilizers, both because of their low concentration of P_2O_5 and also because of the presence of impurities which might influence their use negatively.

Therefore the problem of classifying a rock and evaluating it in relation to its potential use in the production of fertilizers, has become more and more important.

As it is known, the best method, which offers certain results, is that of using the rock to be tested in a trial commercial production.

This method, however, requires the availability of several thousands tons of mineral, and besides this, the plant must moved away its normal production activity for a period which may become fairly lengthy.

These reasons make such a test difficult to carry out and above all very costly.

Another valid system is that of using pilot plants which reproduce, on a reduced scale, the characteristics of industrial plants.

Such a system was previously used by Montedison which,in its "Stazione Sperimentale" at Vercelli, had a pilot plant for the production of phosphoric acid and both phosphatic and complex fertilizers.

The use of this system, even if it raises less problems than tests on commercial scale, still calls for the availability of an adequate pilot plant and for a sample of rock in the order of several hundred tons.

It is known that the Gulf Design Company has recently set up a pilot plant for the production of phosphoric acid (1), with which a rock can be evaluated using a sample of "only 40 tons instead of the 30.000 or even more tons required when the test is carried out on commercial scale".

However, a large sample is still required for a test which is limited to the production of phosphoric acid alone.

Montedison in its Fertilizers Research Centre at Porto Marghera has set up a methodology on a pre-pilot scale and so with a relatively low cost and relatively simple means, which allows us to obtain a sufficient quantity of information to give a fairly reliable picture of the use of a given rock in the production of fertilizers.

This methodology consists of a physical-chemical characterisation of the mineral, including XRay analysis and of the performance of a series of laboratory tests to evaluate its grindability, its foaming tendency during the attack and, moreover, its behaviour in commercial production of acid, superphosphate and complex fertilizers by nitric attack.

The information thus collected, which can be obtained from a sample of 200 kilos, permits, apart from the technical evaluation of the rock tested, also an economical evaluation because it is possible to calculate its market value as compared with that of a reference rock for supplying final products at the same average cost.

2) CHEMICAL AND PHYSICAL CHEMICAL CHARACTERISATION

The first test performed on the rock under examination is obviously the analitical one, necessary to define the chemical composition, the mineralogical nature, the sieve analysis and certain other characteristics, the knowledge of which is useful for classifying the sample fairly, and for laying a basis for subsequent laboratory tests.

2.1) Chemical analysis. The chemical analysis which is carried out by traditional analytic methods used on phosphate rocks, includes the determination of humidity, total P_2O_5, CaO and other constituents.

The BPL grade is also calculated and the CaO/P_2O_5 ratio, detracting the CaO bound as sulphate.

The minor constituents normally determined are SO_4, CO_2, F, SiO_2 Al_2O_3, Fe_2O_3, MgO, Na_2O, K_2O, Cl, organic matters.

In particular cases, supplementary tests can also be carried out, for example for the research of sulphides etc.

As it is known, these impurities can influence the behaviour of a mineral in various processes even in a decisive way, in proportion to the percentage in which they are present.

In particular, the presence of sulphates does not modify appreciably the conditions of the treatment so long as they do not reach unusually high limits, while on the contrary, the presence of carbonates increases the acid consumption for the attack; moreover, carbonates increase the formation of foam during the attack, above all in the nitric process, necessitating a greater consumption of antifoam but, on the other hand, they improve the reactivity of the mineral, a fact which is particularly appreciated in the production of phosphate fertilizers.

The presence of fluorine causes corrosion problems, sludges, formation of slurry and atmospheric pollution; during the production of phosphoric acid, moreover, it can decrease the filtrability of the gypsums because of the formation of complex salts.

The silica, if present in notable quantities, leads to a greater wear of grinding equipments, and causes erosion on stirrers and in pumps which handle the attack slurries.

It can also influence negatively the gypsum filtration in the phosphoric acid processing and can rise problems in the production of normal and triple super-phosphates, in as much as the setting phenomenon is delayed.

A lack of silica, below the stoichiometric quantity necessary to bind the fluorine as SiF_6 ion, causes difficulty in the purification of the phosphoric acid for technical use, so that adding reactive silica during the attack is necessary.

Iron and aluminium oxides, especially the first one are particularly undesirable, since, after the dissolution of rock with acid, they form insoluble phosphates which decrease the percentage of water-soluble P_2O_5 in the final product; moreover they reduce the filtrability of the gypsums during the phosphoric acid processing, and in the end, in the production of superphosphate a product with inferior physical properties is obtained.

The presence of magnesium also cause the formation of insoluble phosphates which reduce the level of water soluble P_2O_5 in the final product; beyond a certain limit the magnesium can cause thickening during the ammoniation of relevant phosphoric acid and of the slurries from nitric attack; the sodium and potassium react with the fluorine and the silica forming fluor-silicates which can cause sludges in the equipment and their elimination can present notable problems.

The chlorine present in the rock is responsible for serious corrosion phenomena, particularly during the phosphoric acid production, and during the nitric attack; if its level rises above a certain limit it can even make a rock unusable.

The presence of organic matters increases the formation of foam during the acid attack and thus the anti-foam consumption increases; in the nitric attack the organic matters favour the development of NO_x, worsening the pollution and resulting in nitrogen loss; moreover they modify the properties of the gypsum reducing the filtrability and therefore causing loss of production in the processing of the phosphoric acid.

Finally they increase the sensivity to the phenomenon of the self-sustaining thermal decomposition in fertilizers from nitric attack.

On the basis of the results of the chemical analysis it is thus possible to have a picture of a rock not only from a qualitative aspect but also in relation to the problems which may arise during its use, with the possibility, therefore, of taking, right from the beginning, the necessary steps to avoid some troubles which can be foreseen.

2.2) XRay - Analysis. Besides the chemical analysis, XRay tests are also carried out as a rule to determine the cell parameters and the average size of the crystallites.

Via the cell parameters one can approximately define the empirical formula of the apatite, and thus its mineralogical nature while the average size of the crystallites allows together with other factors, the approximative evaluation of the reactivity of a rock.

These research techniques are based on the studies and experiments carried out by J.R. Lehr, J.P. Smith and others in 1966-67 (2.3).

2.3) Other physical determinations. Some other properties are also object of research, and these are the external specific surface, and the porosity.

As far as the external specific surface is concerned, its determination is sometimes also extended to the ground rock for the production of phosphoric acid and superphosphate, given the direct correlation that exist between external specific surface, sieve analysis and reactivity.

The specific weight, both real and apparent, and the bulk density are also determined, the latter being useful for evaluating the necessary storage space.

2.4) Sieve analysis. Finally to complete the examination of a rock from the qualitative aspect the sieve analysis is also carried out to put into evidence the possible presence of particles which are too big, and which would increase the cost of grinding, or particles which are too fine, which would cause notable troubles because of the dust during its handling

3) TECHNOLOGICAL TESTS

When the analytic investigation has been accomplished and the initial data about the phosphate rock under examination have been obtained, the laboratory test start by which the collection of data on the mineral can be completed and at the same time the optimum conditions for its use in the various commercial processes can be established.

3.1) Grindability test. The test is necessary to evaluate the grinding efficiency that can be achieved in existing plants, and the use of electric power needed to take the rock to the size required for the various production cycle.

The characteristics of the equipment and the test conditions are accurately standardized to be perfectly reproducible.

The sample to be tested is obtained by taking away from the screened raw rock, the fraction under 0.150mm.

This value has been chosen conventionally because the undersize is considered suitable for any production cycle.

Three kilograms so prepared of sample are submitted to the test of grindability.

At the end of the test the sieve analysis is repeated so as to determine the quantity of rock under 0.150mm produced during the test.

Evaluating globally the percentage of undersize at 0.150mm, as sum of the quantity present at the beginning and that formed during the test, in relation to the corresponding values obtained with the reference rock,it is possible to define the capacity of the commercial grinding plant and the relative power consumption, since the capacity obtainable with the reference rock is known.

3.2) Foaming test. This is above all important for the nitric attack, but sometimes it is also useful for the sulphuric attack for the production of w.p. phosphoric acid.

It is performed discontinuously on a sample of a 100 grams of rock, sized between 0.075 and 0.063 mm, which is attacked with nitric acid in a graded beacker, under agitation; the maximum height reached by the foam is measured.

Even in this test, standard conditions are used so that they can be completely reproduced, and the result is compared with that obtained with the reference rock.

With this test it is possible to evaluate how much antifoam is necessary to use in commercial processes.

3.3) Phosphoric acid production test. This is carried out, by applying the dihydrate process, in standard equipment, composed of three stainless steel, thermostated reactors, each with a capacity of five litres, set in a series and supplied with stirring devices.

Ground rock,sulphuric acid and recycling acid are continuosly fed by metering devices usually in the first reactor, and the slurry is recycled from the third to the first reactor by means of a pump.

The quantity of fed rock is about 1 kilogram per hour, in order to have the same residence time as in a commercial plant.

The filtration of the slurry is carried out continuously; part of the filtrate is used to prepare the recycling in the desired concentration.

Process variables normally held under observation are: size of the rock, temperature of attack, sulphuric acidity, concentration of the produced phosphoric acid, solid/liquid ratio, and possible use of agents modifying the crystalline morphology of gypsum.

The characterisation of a certain run in pre-established conditions, is carried out when the parameters have been kept costant for twenty four hours, filtering, in standard conditions, five litres of slurry, which represent the content of the third reactor and washing the gypsum panel twice with acid at decreasing concentrations, and finally with water, thus simulating a continuous four stages filter.

In such a way, it is possible to determine the filtering and washing rate of the slurry which is expressed in tons of P_2O_5 per m^2 per day.

Both phosphoric acid and gypsum are analysed to quantify the cycle.

The losses are expressed as un-attacked P_2O_5, co-crystallized P_2O_5 and water-soluble P_2O_5.

Besides this, photographs and crystallographic investigations are also carried out on the gypsum.

Usually the corrosivity of the rock towards the material of the equipment is also evaluated; for this purpose propellors in different materials are mounted on the stirrers of the reactors calculating the corrosion with the usual tests.

If the rock under examination has a notable level of iron, aluminium and magnesium, the phosphoric acid obtained, concentrated up to 42% of P_2O_5 and decantated, is processed to obtain DAP in order to evaluate the physical characteristics, the viscosity of the corresponding ammoniated slurry and the relation between water-soluble P_2O_5 and citrate-soluble P_2O_5 in the final product.

When the phosphoric acid is intended for the production of technical phosphates, it is processed to obtain sodium tripolyphosphate in order to determine the process yields and the properties of the final product.

3.4) Superphosphate production test. This is carried out discontinuously in a five litre beaker with a diameter of 16 cm and a hight of 27 centimetres, in which is placed the sulphuric acid preheated to 55° centigrades.

The ground rock (800 g) is poured into the beaker in a very short time (not more than ten seconds), while the mass is kept in agitation with a wooden spoon for about another ten seconds.

If the commercial plant where the rock under examination is to be used, is equipped for fluorine recycling,during the test the hydrofluosilicic acid necessary is poured into the beaker at the same time as the rock sample.

The reaction taking place is followed by registering the maximum height reached by the mass, the height of the panel during the setting and the following times:

- time of reaction (from the end of the introduction of the rock to the beginning of the process of solidification),
- setting time (from the beginning of the solidification until when the product ceases being plastic and turns to be soft),
- discharging time (this is evaluated when the product has attained such physical properties as softness, sponginess, to be easily handled).

To carry out these evaluations correctly it is necessary to have acquired a good laboratory experience.

The maximum temperature of reaction is determined by means of a thermometer inserted right from the beginning in the reacting mass.

The process variables to be observed and optimised are: size of rock, concentration and amount of sulphuric acid and possibly of hydrofluosilicic acid and possible use of surface-active agent.

The aim is to mantain the reaction, setting and discharging times within determined limits which are comparable to those of the reference rock.

The superphosphate obtained is put into a suitable thermostated container and kept at temperature of 45° centigrades; it is analysed after twenty four hours, and after 3,5, 10 and 15 days of curing to determine the yield of solubilisation of P_2O_5 and thus the curing time necessary to bring such a yield above 98%.

With the same method a test for the production of triple superphosphate can be carried out.

3.5) Test for the production of fertilizers by nitric attack. Usually this test concerns the production of a ternary complex fertilizer like 12-12-12, and is composed of the following six fundamental operations:

a) attack of the rock with a mixture of nitric, sulphuric and phosphoric acid in a twelve litre reactor, equipped with a 400 watt stirrer using a sample amount such as to obtain 10 kilograms of final product,
b) ammoniation of the attack slurry up to neutralisation grade of soluble P_2O_5 corresponding to about a molal ratio NH_3/H_3PO_4 = 1,
c) addition of potassium chloride,
d) concentration of the slurry,
e) granulation with final ammoniation,
f) drying, classification and treatment with anticaking.

Every operation is performed, trying to stay as near as possible to the real operating conditions of the commercial plant, particularly as far as temperature, residence times in the liquid phase, and degree of neutralisation are concerned.

The parameters are, however, optimised, with the aim of resolving the various problems connected with the characteristics of the intermediate phases, such as foaming tendency of the slurry,viscosity of the ammoniated slurries and their granulability.

The final product is thus submitted to analytic tests, and to technological tests (measuring of the caking tendency and of the horizontal speed of self-sustaining thermal decomposition).

On the basis of the results obtained, it is possible to define the criteria to adopt for the formulation, and the operating conditions for producing other complex fertilizers by nitric attack.

4) EXAMPLE OF CHARACTERISATION

To give a more complete idea, and to illustrate better the methodology described above,a practical example of the evaluation of a raw phosphate rock follows.

4.1) Chemical and physical-chemical characterisation. In tables 1, 2 and 3 the results of the analyses and the investigations carried out on a rock under examination are given as compared with the values that are typical for the reference rock.

From the examination of the results in tables 1 and 2, apart from their difference in the P_2O_5 content and therefore of their BPL grades,it appears to be a matter of two carbofluoroapatites with a somewhat different empirical formula.

The rock under examination contains an appreciable quantity of calcium sulphate which can have a negative action on the setting phenomenon of the superphosphate.

Moreover, the dolomite and limestone are also present in appreciable quantities: this characteristic, together with the evaluation of the crystallites size and the external specific surface, appears to indicate that the rock under examination has a greater reactivity than the reference rock.

Other aspects to be taken into consideration are low iron and aluminium content, high magnesium content and above all,a notable presence of organic matters which, very probably, will cause problems due to the foam and to NO_x formation during the nitric attack.

As far as the sieve size is concerned (see table 3) the rock under examination proves to be finer than the reference one, but the amount under 0,075 mm is contained within acceptable limits and so dusting problems are not expected during its handling.

4.2) Technological tests.

4.2.1) Grindability test. The test is performed in a cylindrical ceramic ball mill with diameter = length of 27 centimeters;460 balls of various diameters are used with a total weight of 6.65 kilograms, corresponding to a ball/rock weight ratio of 0,451.

The mill turns at 64 rpm; the test lasts two hours.

In table 4, a picture of the values, before and after the test, is given, from which it appears that for the rock under examination the undersize 0,150 mm has increased from 60.9% to 89.6%, while for reference rock it has reached 62.5%.

Consequently the rock under examination allows for an increase in the capacity of grinding of about 40%. This corresponds to a decrease of about 30% in the electric power cost and to a decrease of about 20% in the maintenance cost.

4.2.2) Foaming test. For this test a 100 grams sample of rock is needed, with a granulometric range between 0.075 and 0.063 mm .

HNO_3 is put into a graded five litre beaker, 140 mm diameter and 325 mm height, equipped with a 300 rpm stirrer.

The used HNO_3 is at 53% by weight and preheated to 60°C, in such a quantity as to have, in relation to the rock, an equivalent attack ratio 1,2 mol ($2HNO_3$)/mol CaO.

The rock is added quickly, under agitation to the acid, and then the agitation is continued for another ten seconds, measuring the maximum height reached by the foam in the graded beaker.

The test is repeated using antifoam to determine the amount of that agent necessary to reduce to zero the formation of foam.

In table 5 the results obtained in this test are given; it is apparent that the rock under examination needs twice the amount of antifoam agent as the reference rock.

4.2.3) Phosphoric acid production test. This was performed according to the method indicated in paragraph 3.3.

With a series of preliminary tests an optimum run condition set was found from which the significant data are given in table 6, while in table 7 the characteristics of the phosphoric acid and the gypsum produced are shown, together with the yield of P_2O_5.

In figures 1 and 2 the photographs of the gypsum obtained are given.

The increased use of surfactans has allowed a considerable improvement of the morphology of the crystals, besides having an antifoaming action.

Moreover, the filtration rate that can be achieved with the rock under examination proves to be practically the same as with the reference rock; therefore a similar productive capacity is predictable in the commercial plant.

The P_2O_5 yield is inferior by 2.5 points.

The values of the corrosion found on the stirrer propellors, although proving superior as compared with the reference rock are acceptable.

4.2.4) Superphosphate production test. The test was performed according to the technique described in paragraph 3.4).

On the basis of preliminary tests the most favourable operating conditions were defined, which are given in table 8, while in table 9 the analysis of the obtained superphosphate is given.

The use of surfactans has allowed to achieve setting times near those obtained with the reference rock and therefore the productive capacity of the plants should not suffer any variation. Without the use of surfactans the greater setting-times indicate a 25% reduction in the productive capacity.

The good reactivity of the rock under examination, enhanced by the particular kind of the surfactans employed, is well shown by the analysis in table 9.

This analysis demonstrates that the yield of solubilisation exceeds 98% after 5 days curing, while with the reference rock about double period is required.

4.2.5) Test for the production of fertilizers by nitric attack. The complex fertilizer 12.12.12 was produced according to the methodology indicated in paragraph 3.5).

On the basis of a preliminary test, the formulation was defined in relation to the rock analysis.

In table 10 the final amounts of raw materials and the test conditions are given, while in table 11 the characteristics of the product obtained are shown.

With the rock under examination the slurry appears slightly more viscous and so the neutralization grade in the liquid phase was reduced.

The analysis conferms the validity of formulation criteria adopted.

The intermediates by phospho-nitric and sulpho-nitric attack were found to have 14,7-24,7-0 and 12,9-12,9-0 grades respectively, instead of

14,9-24,4-0 and 14,3-14,4-0 which are obtained with the reference rock.

4.3) Technical evaluation. From the examination of the data collected with the chemical and physical-chemical characterisation, and from the performance of the technological tests, a technical evaluation of the examined rock can be given.

The mineral of the example given is a rock which proves to be suitable for the production of phosphoric acid, superphosphate and complex fertilizer from nitric attack, with the productive cycles used by Montedison.

The operating conditions to be adopted in the various cases can be deduced from the examination of the results given previously.

4.4) Economical evaluation. The information gained during the characterisation and the performance of the laboratory tests, makes an economical evaluation of the examined rock possible.

One can establish how much the examined rock should cost as compared with the reference rock in order to get the same final products at the same average cost.

To do this, it is supposed that the rock under examination replaces the reference rock in every process in a factory producing fertilizers.

In the case of this example it was assumed that the typical factory consumes 350,000 metric tons per year of reference rock, subdivided as follows:

- 135,000 mtons per year for the production of phosphoric acid;
- 115,000 mtons per year for the production of superphosphate;
- 110,000 mtons per year for the production of complex fertilizers by nitric attack.

The reference rock and sulphuric acid are valued respectively at 38.6 and 42 doll/mton, and the H_3PO_4, that has to be puchased to maintain constant the grades of finished products,is valued at 245 dollars per ton P_2O_5.

In table 12 the calculation concerning the tested rock is described in detail; it has been done on the basis of data presented in the preceeding tables.

The following formula is used to determine the cost ratio between tested and reference rocks:

$$K = \frac{(350{,}000\ t/y \times 38.6\ doll/t) - X}{350{,}000\ t/y \times 38.6\ doll/t}$$

where K is the cost ratio and X is the total increase of cost due to the use of the rock under examination.

The amounts of new rock needed for the various processes become approximately:

- 159,000 tons per year for the production of phosphoric acid
- 113,000 tons per year for the production of superphosphate
- 97,000 tons per year for the production of complex fertilizers.

From table 12 it seems obvious that the rock under examination must cost 0.78 times the reference rock to obtain final products at the same average cost.

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TABLE N° 1

Chemical and chemical-physical characterization:

a) Chemical analysis

	Units	VALUES			
		Tested rock		Reference rock	
		run of pile	dry basis	run of pile	dry basis
Moisture	% wgt	1.2		1.5	
P_2O_5		28.8	29.15	33.1	33.6
CaO		48.4	49	48.5	49.24
SO_4		3.5	3.54	1.2	1.22
CO_2		7.5	7.59	3.6	3.65
F		3.4	3.44	3.9	3.96
SiO_2		1.9	1.92	3.4	3.45
Al_2O_3		0.48	0.49	1.4	1.42
Fe_2O_3		0.38	0.38	0.7	0.71
MgO		1.66	1.68	0.3	0.30
Na_2O		1.31	1.33	0.6	0.61
K_2O		0.13	0.13	0.1	0.1
Cl		0.02	0.02	0.01	0.01
Org. Matter (expressed as C) ..		0.39	0.39	0.24	0.24
CaO (°)/P_2O_5	g/g	1.61	1.61	1.444	1.444
$Ca_3(PO_4)_2$ corresponding to P_2O_5 = BPL	%	62.9	63.7	72.3	73.4

(°) Detracted the CaO bound stoichiometrically to SO_4

TABLE N° 2

b) X Ray analysis and physical determinations

			VALUES	
			Tested rock	Reference rock
X-Ray Analisis	Cell parameter: a	Å	9,320	9,328
	c	"	6,890	6,884
	Crystallite size:			
	- along the X axis	"	360	450
	- " " Z "	"	570	550
	Empirical formula		$Ca_{10}(PO_4)_{5.2}(CO_3)_{1.3}F_{1.8}$	$Ca_{10}(PO_4)_{5.1}(CO_3)_{1.1}F_{2.5}$
Physical determinations	External specific surface	cm^2/g	732	165.5
	Porosity (75,000÷38 A) ..	cm^3/g	0.042	0.0648
	Real specific weight	g/cm^3	2.916	2.979
	Apparent specific weight.	"	2.681	2.511
	Bulk density	"	1.515	1.642

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TABLE N° 3

Chemical and chemical-physical characterization:

c) Sieve analysis

	Units	VALUES			
		Tested rock		Reference rock	
		Partial	Integral	Partial	Integral
Net opening of the sieves:					
+ 1.000 mm	% wgt	0.2	0.2	0.6	0.6
1.000÷0.500 mm	" "	9.3	9.5	8.1	8.7
0.500÷0.425 mm	" "	3.8	13.3	6.4	15.1
0.425÷0.300 mm	" "	4.2	17.5	29.2	44.3
0.300÷0.212 mm	" "	5.6	23.1	36.1	80.4
0.212÷0.150 mm	" "	16.0	39.1	14.2	94.6
0.150÷0.100 mm	" "	52.4	91.5	3.5	98.1
0.100÷0.075 mm	" "	6.2	97.7	0.5	98.6
- 0.075 mm	" "	2.3	2.3	1.4	1.4

TABLE N° 4

Technological tests:

a) Grindability test

	Units	Tested rock				Reference rock			
		A	B	C	D	A	B	C	D
Net opening of the sieves:									
+ 1.000 mm	% wgt	0.2	0.2	0.0	0.0	0.6	0.6	0.2	0.2
1.000÷0.150 mm	" "	38.9	38.9	10.4	10.4	94.0	94.0	37.3	37.3
- 0.150 mm	" "	60.9		28.7	89.6	5.4		57.1	62.5
		100.0	39.1	39.1	100	100	94.6	94.6	100

Note: A) Sample of raw rock
B) Sample A, detracted the undersize below 0,150 mm
C) Sample at the end of the grindability test
D) Sample at the end of the test, readded with the undersize 0,150

TABLE N° 5

b) Foaming test

	Units	VALUES	
		Tested rock	Reference rock
Height reached by the foam ...	mm	200	70
Antifoam ROLSOL 4015R(°) necessary to destroy the foam	g/100 grams of rock	0.2	0.1

(°) Produced by ROL-Montedison - Italy

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TABLE N° 6

Technological tests

c) Continuons test for production of H_3PO_4 - Run data

	Units	VALUES	
		Tested rock	Reference rock
a) Raw materials:			
- ground rock	g/h	1,000	1,000
- oversize 0.150 mm	%	30	24
- H_2SO_4 at 75%	g/h	1,081	1,120
- surfactans Montaline SPCV^{R}(°) ..	"	0,180	0,130
- recycle: flow rate	"	2,993	2,596
density at 20°C	g/cm^3	1.245	1.210
P_2O_5	%	21.2	19.1
- washing water for gypsum	g/h	1,460	1,400
b) Attack temperature	°C	78	82
c) Stirrer speed	rpm	500	500
d) Slurry characteristics:			
- apparent specific weight	g/cm^3	1.510	1.520
- solid/liquid ratio	ml/ml	400/600	450/550
e) Filtration rate on Meraklon cloth type 1805/TS by Testori	tP_2O_5/m^2 d	13.8	14
f) Corrosion of stirrer propellors:			
- AISI 316 L propellor	g/m^2 d	1.5	0.5
- URANUS B6 propellor	"	0.45	0.1
- HASTELLOY C propellor	"	0.05	0.0

(°) Produced by SEPPIC MONTANOIR (FRANCE)

TABLE N° 7

d) Continuous test for production of H_3PO_4 - Acid and gypsum characteristics P_2O_5 yields

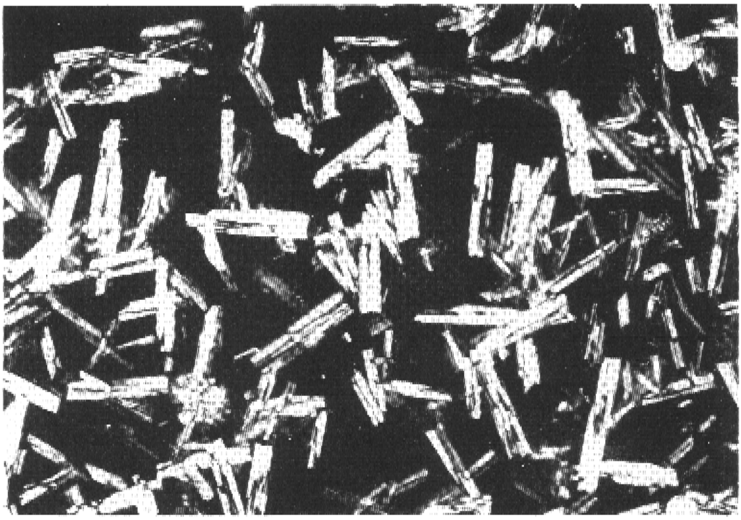
	Units	Tested rock		Reference rock	
		H_3PO_4	Gypsum	H_3PO_4	Gypsum
Specific weight at 20°C	g/cm^3	1.310		1.315	
Moisture	%		31		28
P_2O_5 water soluble	"		0.13		0.12
P_2O_5 cocrystallized (citrate-soluble)	"		0.13		0.26
P_2O_5 insoluble	"		0.54		0.18
P_2O_5 total	"	28	0.8	28	0.56
Non phosphoric acidity (as H_2SO_4)....	"	2.5		2.6	
SO_4 total	g/l	18.0		21.3	
CaO total	"	4.0		3.8	
H_2SO_4 free	%	1.37		1.6	
$CaSO_4$. $2H_2O$	"		96.1		98
$CaSO_4$. ½H_2O	"		3.9		2
P_2O_5 yields: P_2O_5 water-soluble		99		99.2	
P_2O_5 cocrystallised		99		98.4	
P_2O_5 insoluble		96		98.9	
P_2O_5 total		94		96.5	

TECHNICAL AND ECONOMICAL EVALUATION OF A RAW PHOSPHATE ROCK FOR THE PRODUCTION OF FERTILISERS

FIGURA N° 1

Technological Tests:

c) Continuous test for production of H_3PO_4 - Photographs of Gypsum produced (100 x) (see table 7)



Tested rock

Reference rock

TECHNICAL AND ECONOMICAL EVALUATION OF A RAW PHOSPHATE ROCK FOR THE PRODUCTION OF FERTILISERS

TABLE N° 8

Technological tests

d) Test for the production of superphosphate - Operating conditions

	Units	VALUES	
		Tested rock	Reference rock
a) Raw materials:			
- rock- sieve analysis:			
+ 0.150	% wgt	5	5
0.150÷0.075	" "	40	35
- 0.075	" "	55	60
- H_2SO_4 concentration (as H_2SO_4 100%)	" "	71.5	75.3
- H_2SiF_6 concentration	" "	23.0	23.3
b) Consumption per ton of superphosphate :			
- rock run-of-pile	kg	562	558
- H_2SO_4 100%	kg	348	350
- H_2SiF_6 100%		15.3	18.6
- surface active agent Montaline SPCVR(°)		0.2	
c) Temperatures:			
- of acid	°C	45	50
- maximum of reaction	°C	112	100
d) Max height of reacting mass	cm	19	15
Height of panel	cm	11	10
e) Times:			
- of reaction	sec	100	90
- of setting	"	200	180
- of discharge	min	9.5	9

(°) Produced by SEPPIC MONTANOIR (FRANCE)

TECHNICAL AND ECONOMICAL EVALUATION OF A RAW PHOSPHATE ROCK FOR THE PRODUCTION OF FERTILISERS

TABLE N° 9

Technological tests:

d) Superphosphate production test -Analysis of the superphosphate obtained with the tested rock

	Units	After 1 day curing	After 3 days curing	After 5 days curing
Moisture	% wgt	13.0	12.6	12.5
P_2O_5 free (volumetric method) .	" "	6.0	4.5	3.8
P_2O_5 water soluble	" "	14.3	15.0	15.2
P_2O_5 water and citrate-soluble	" "	14.9	15.7	16.0
P_2O_5 insoluble	" "	1.2	0.5	0.2
P_2O_5 total	" "	16.1	16.2	16.2
P_2O_5 water soluble/P_2O_5 water and citrate-soluble	" "	96.0	95.5	95.0
SO_4	" "	35.4		
F	" "	1.8		
Solubilisation yield	" "	92.5	96.9	98.8

With the reference rock,the grade of the obtained superphosphate is 18,2% water and citrate soluble P_2O_5 after curing. The same grade is obtained with the tested rock only by adding phosphoric acid to sulphuric acid (see table 12) during the attack reaction.

TABLE N° 10

e) Test for the production of fertilisers - Ternary 12.12.12 - formulation and operating conditions

	Units	VALUES	
		Tested rock	Reference rock
a) Raw material:			
- rock	kg/t	306	316
- H_2SO_4 100%	"	86.6	101.0
- HNO_3 100%	"	291	291
- NH_3 100%	"	79	79
- P_2O_5 in phosph. ac.	"	40.3	23.3
- KCl 60%	"	206	206
- Antifoaming ROLSOL 4015^{R}(°).....	"	0.6	0.3
b) Rock sieve analysis:			
+ 0,150 mm	"	35	30
0,150÷0,075 mm	"	43	45
- 0,075 mm	"	22	25
c) Times:			
- of attack	hours	1	1
- of ammoniation	"	3	3
d) Temperatures:			
- of attack	°C	60	60
- of ammoniation (max)	"	110	110
e) Degree of neutralisation:			
- pre-ammoniated slurry	molN/	0.5	0.6
- ammoniated slurry	mol P	0.96	0.98
f) Viscosity at 110°C:			
- pre-ammoniated slurry	cp	35	20
- ammoniated slurry	"	180	150

(°) Produced by ROL by Montedison group - Italy

TECHNICAL AND ECONOMICAL EVALUATION OF A RAW PHOSPHATE ROCK FOR THE PRODUCTION OF FERTILISERS

TABLE N° 11

Technological tests

f) Test for the production of fertiliser - Ternary 12.12.12 - Characteristic of the product obtained

	Units	VALUES	
		Tested rock	Reference rock
Moisture	% wgt	1.4	1.6
Ammoniacal nitrogen	" "	6.2	6.2
Nitric nitrogen	" "	5.9	6.0
Total nitrogen	" "	12.1	12.2
P_2O_5 water soluble	" "	4.0	4.2
P_2O_5 water and citrate-soluble....	" "	12.1	12.1
P_2O_5 insoluble	" "	0.1	0.2
P_2O_5 total	" "	12.2	12.3
K_2O	" "	12.2	12.3
P_2O_5 water-soluble/P_2O_5 water and citrate soluble	" "	33	35
Caking tendency (°)		free flowing	free flowing
Horizontal self-sustaining decomposition rate (on ISMA basket)	cm/h	12	10

(°) Product treated with an anti-caking agent

TABLE N° 12

Comparative Economic Evaluation

	Units	VALUES	
1 - Grinding:			
a) Reduction in electric power consumption .	10^3\$/y	- 22.7	
b) Reduction in maintenance cost	"	- 28.3	
			- 51
2 - Production of phosphoric acid:			
a) Increased consumption of sulphuric acid .	"	665	
b) Increased consumption of surfactans	"	11	
			676
3 - Production of superphosphate:			
a) Reduced consumption of sulphuric acid ...	"	- 256	
b) Consumption of H_3PO_4 for enrichment	"	1,309	
c) Consumption of surface-active agent	"	47	
			1,100
4 - Production of complex fertilisers			
a) Reduced consumption of sulphuric acid ...		- 192	
b) Increased consumption of H_3PO_4 for enrichment		1,315	
c) Increased consumption of antifoam		87	
d) Increased loss of nitric nitrogen		42	
			1,252
TOTAL	10^3\$/y		2,977

TABLE N° 12 (continued)

Evaluation of cost ratio between tested and reference rock corresponding to the same average cost of the products:

$$K = \frac{350,000 \times 38.6 - 2,977,000}{350,000 \times 38.6} = 0.78$$