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IGNEOUS AND SEDIMENTARY PHOSPHATE ROCK EVALUATION AT FOSKOR'S PHOSPHORIC ACID PILOT-PLANT FACILITY

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Le groupe Foskor de sociétés, cad Foskor Phalaborwa Ltd, et Indian Ocean Fertilizers Ltd. (IOF, Richards Bay) connaîtront le début d'expansions majeures de ses installations en l'an 2000. Vers le milieu de l'an 2000 IOF débuteront l'agrandissement de leur unité d'acide phosphorique de 330 000 tonnes P2O5 à un total de 770 000 tonnes par an. A Phalaborwa l'agrandissement de l'atelier de phosphate brut est presque achevé et commencera sa production complète avant la fin de l'an 2000 (accroissement de 700 000 à 3,5 millions de tonnes au total de phosphate par an).

Ces expansions impliquent l'évaluation approfondie de la faisabilité techno-économique, et, pour ce faire, Foskor a modernisé et utilisé à la fois ses installations- pilotes de flottation et d'acide phosphorique. Bien que l'atelier pilote d'acide phosphorique soit relativement petite, environ 5,7 litres elle offre les possibilités suivantes :

- Analyse d'échantillons spécifiques de phosphate brut et comparaison entre différents types et origines de phosphate.
- Détermination des paramètres de performance, filtration, et rendement.
- Détermination de la qualité de l'acide et sa concentration jusqu'à 54% P2O5
- Production d'échantillons d'acide et de gypse pour analyse en aval.
- Evaluation de la corrosion et analyse de différents aciers inoxydables.

C'est pourquoi cet exposé examine (avec quelques études de cas) le rôle de l'atelier pilote d'acide phosphorique dans la fourniture d informations adhoc pour l'évaluation du phosphate brut comme convenant à la production d'acide phosphorique.

SUMMARY

The Foskor group of companies, that is, Foskor Phalaborwa Ltd. and Indian Ocean Fertilizers Ltd. (IOF-Richards Bay), will see the initiation of major expansions at its facilities during 2000. Towards the middle of the year, IOF will start expanding their phosphoric acid plant by 330 000 tons P_2O_5 to a total of 770 000 tons per annum. At Phalaborwa, an extension to the phosphate rock plant is nearing completion and will begin full-scale production before the end of 2000 (an increase of 700 000 tons to a total of 3.5 million tons of rock per annum).

These expansions require extensive evaluation of the techno-economic feasibility, and in order to do this, Foskor updated and utilised both its flotation and phosphoric acid pilot-plant facilities. Although the phosphoric acid pilot-plant is relatively small, about 5.7 litres, it has the following capabilities:

• Testing of specific rock phosphate samples and comparison between various rock phosphate types and origins.

- Determination of acid quality and acid concentration to 54 percent P₂O₅.
- Production of acid and gypsum samples for down stream testing.
- Evaluation of corrosion and testing of various stainless steels.

This paper therefore discusses (together with some actual case studies) the role of the acid pilot-plant facility in providing relevant information for the evaluation of rock phosphate in terms of its suitability for phosphoric acid production.

RESUMÉ

1. INTRODUCTION

1.1 Location

As is depicted in Figure 1, Foskor's phosphate rock mine and concentrate plant is situated in Phalaborwa, in the Northern Province of South Africa. The mine is connected to its phosphoric acid and fertiliser plant (IOF), which is located in Richards Bay on the East Coast of South Africa, by some 800 km of rail.



Figure 1. Location of the Foskor group - the phosphate mine in Phalaborwa and phosphoric acid plant (IOF) in Richards Bay.

1.2 Ore bodies and derived rock phosphate products

As with most igneous deposits, the ore body is quite diversified and consists of 95% Pyroxenite, 3% Foskorite and 2% Carbonatite (Figure 2). The complex extends vertically into the earth to an unknown depth.



Figure 2. The geology of the ore body(s) from which Foskor's rock phosphate products are derived.

1.2.1 Foskor mine and rock phosphate concentrate plant

The phosphate mine has a current output capacity of 2.8 million tons of rock phosphate concentrate. Production is derived from 16 million tons of ore per annum extracted by Foskor's own mining activity and 10 million tons in tailings from Palabora Mining Company's (PMC – a copper producing mine) operation. Three rock phosphate products are produced by a milling and flotation operation, namely 88P (from the pyroxenite mine), 88S (from Foskorite and PMC tailings), and 80M (mixture of the two).

In order to meet future market expectations, expansions of both the mine (2.8 to 3.5 million tons of rock) and IOF acid plant was deemed necessary. It is envisaged that the expansion at Foskor's mine will be complete and commissioned by June 2000.

1.2.2 IOF phosphoric acid and fertilizer plant

IOF currently receives 80M from the mine, which is processed either alone or in blends with rock from Togo (a sedimentary rock phosphate). The processing technology used at present is dihydrate Prayon with Prayon-Rupel tilting pan filtration. For the expansion, two technologies were considered: Prayon and the Raytheon-Badger process. For reasons of familiarity, proven technology and economic reasons, IOF management are to implement Prayon technology for the expansion.

IOF's capacity at this stage is approximately 440 000 tons of P_2O_5 per annum. IOF also produce granulated products in a facility of about 400 000 tons capacity. Commissioning of the new phosphoric acid (770 000 tons P_2O_5 per annum total capacity) facility at IOF will take place in April 2001. IOF's sulphuric acid production is also to be upgraded to meet the demand internally.

1.3 Strategy

The evaluation of strategic options to facilitate the expansions within the Foskor group of companies, is by no means a trivial process. This is because the ultimate performance of any phosphoric acid plant is critically dependent on the type and quality of rock phosphate used. In the decision making process, a complex matrix of interrelated issues requires appraisement:

- What parts of the ore body should be processed and what would be the efficiency and therefore the cost of producing concentrate from such ore bodies (bearing in mind that Foskor utilises a milling and flotation process to produce rock phosphate concentrate)?
- What grade of rock (BPL content) should be produced and what would its resultant quality (in terms of impurities) be?
- What would be the quality of the beneficiated phosphoric acid, what processing efficiencies would be obtained and at what expense considering the type and degree of post-precipitation that might form, the extent of corrosion, the requirements for down stream granulation and other effects in a phosphoric acid plant.
- What process engineering technology would be best?
- How acceptable would these products (phosphate rock and phosphoric acid) be to Foskor's clients?

The element of risk involved in each option has to be quantified in both technical and monetary terms. To augment this complex decision process, technical evaluation of possible rock phosphate products on a pilot-plant scale was necessary to provide clear techno-economic information.

This paper discusses the role of the phosphoric acid pilot-plant in providing sufficient meaningful data on which to base such strategic decisions.

2. METHODOLOGY

2.1 Pilot plant configuration and operation

The design, of which a schematic diagram appears in Figure 3, is intended to simulate a full-scale, phosphoric acid (dihydrate) plant.

Dry rock phosphate is fed via a screw feeder into reactor 1, one of two jacket - heated reactors. Heating of the reactors and maturation tank is required to maintain desired temperatures. The total volume of the reactors is 4680 cm³. Concentrated sulphuric acid and recycled phosphoric acids are added in a mixing vessel in reactor 2. The gypsum - phosphoric acid slurry produced is recycled between the two reactors at approximately 27 litres per hour. Gypsum slurry is held in a maturation vessel (1000cm³ capacity) before being

filtered. The throughput of rock, sulphuric acid, recycle acid and slurry produced are monitored by weight loss/gain on weighing balances.

Filtration is by simple buchner funnel and flask method, however a filtration rate (filter leaf) test is conducted every 6 to 8 hours in a separate apparatus which is designed to yield an accurate estimation of the rate which would be obtained in a full scale filtration operation.

Both reactors are open to atmosphere and mixing of slurry is maintained by single overhead impeller agitation.

The plant is operated in shifts of 8 hours each. When required, the plant can be operated continuously for 24 hours per day (3 shift system) using 1 operator per shift. The plant can also be run semi-continuously for 10 to 16 hours per day. The day to day practicalities (such as preparing rock samples and analysis) of the operation is overseen by a foremantechnologist.



Figure 3.

Schematic diagram of pilot-plant configuration

2.2 Parameters monitored

The pilot-plant is designed to simulate a full-scale operation as closely as possible. There are some fundamental differences however:

- i) Mixing/agitation in the pilot-plant is considered almost ideal, unlike full-scale operations. The implications of this are that pilot-plant campaigns will always yield proportionally better efficiencies and filtration rates.
- ii) Whereas full-scale operations require removal of excess heat energy via flash cooling, the heat loss in the above-described pilot-plant is such that external heating is required. Nevertheless, temperatures, solids content, mother liquor and recycle acid densities are maintained as close to full scale as possible.
- iii) Due to the size of the pilot-plant, filtration and filtration testing, is done batch-wise.

The *modus operandi* of a dihydrate acid plant, whether a pilot or full scale plant, is basically the same. The efficiency is critically dependent on the careful control of reagent input parameters, termed independent variables. These variables have direct impact on the parameters from which efficiency or productivity of the plant is measured (termed dependent variables). The interrelation between the different variables provides the intrinsic complexity associated with WPA plants. As an illustration, the rock feed rate, together with the sulphuric acid dosage and rock quality will influence the free sulphate, which in turn affects the filtration rate and ultimately the overall production rate.

In pilot-plant campaigns, the following variables (Table 1.) are continuously monitored:

Table 1.	Variables monitored and controlled during pilot-plant campaigns.
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Independent Variables	Dependent Variables
H ₂ SO ₄ feed rate (g/hour)	H ₂ SO ₄ to rock consumption ratio
Rock feed rate (g/hour)	"Free" SO₄ in mother liquor (% m/m)
Crystal habit modifier (CHM) / antifoam dosage (g/ton rock)	Rock to P_2O_5 production ratio
Amount of recirculating water (via recycle acid density)	Filtration rate (tons $P_2O_5 / m^2 / 24$ hours)
Reaction temperature (°C)	Overall, co-crystallised and un-reacted
	efficiencies (% based on dry gypsum analysis)
Reaction retention time (hours)	Slurry solids content (% m/m)
Rock phosphate quality	Filter acid strength and amount (% m/m,
	g/hour)

2.3 Preparation of rock phosphate samples

All samples of rock are homogenised, dried to absolute dryness, and characterised in terms of particle size and chemical composition prior to pilot campaigns. All rock phosphate samples utilised in this study were produced in Foskor's metallurgical pilot-plant facility (in the case of "88P" and "83M") or taken from the production plant ("80M").

It is also important to keep historic data about all product compositions and qualities and its related processing performance for comparison purposes.

2.4 Impurities, post-precipitation and scaling

The fundamental effects of rock quality (the role of impurities) on the phosphoric acid processing efficiency and degree, type and mechanism of post-precipitation is the subject of ongoing studies conducted at the University of Pretoria^{1,2}. These studies are integral to rock

quality, and plant campaigns therefore form part of the overall evaluation of rock phosphate suitability. The amount, type of post-precipitate, and scaling are also monitored during each campaign.

2.5 Corrosion testing

Two methods of corrosion measurement in a WPA environment are used, namely, galvanic corrosion and mass loss corrosion.

2.5.1 <u>Galvanic or electrochemical corrosion</u>

Test coupons, comprising the stainless steel under examination, are placed in a holding cell (the cell holding the electrodes and test coupon), in compartment number two of the pilot-plant reactor during phosphoric acid production. The experiment is conducted while simultaneously operating the pilot-plant. The stainless steel being tested is thus subjected to precisely the same conditions (temperature, solids density, etc) as would be found in full scale operating conditions.

Measurements of all potentials and currents are made under steady state conditions, that is, when no further variations in current and potential are observed. All considerations in the corrosion experiments are adhered as closely to corrosion ASTM standards as possible^{3,4}.

2.5.2 Mass loss corrosion

Mass loss corrosion measurements are made simply by accurately pre-weighing and placing the test coupons in suitable holders in compartment 2 of the phosphoric acid reactor and subjecting to the acid-slurry environment for an extended period of time (1 to 2 weeks), whereupon the mass is again accurately determined. The corrosion rate is determined from the mass loss according to a calculation as defined in ASTM standard G31 -72^4 .

3. CASE STUDIES

Three case studies were selected to demonstrate the capability of the pilot-plant and the type and validity of results that can be obtained during digestion of phosphate rock and initial conversion to weak phosphoric acid. Two planned and proposed products, 88P (from Loesche novel milling technology) and 83M were prepared by conventional means in Foskor's metallurgical pilot facilities and compared with an existing product, 80M (produced for local phosphoric acid consumption). The studies are discussed collectively in this paper for the reason that the individual campaigns are similar in practice and that they may be compared.

The compositions of the samples are depicted in Table 2.

Case	Rock	P₂O₅ %	CaO %	MgO %	Al ₂ O ₃ %	Fe ₂ O ₃	SiO₂ %	React SiO ₂	F %	CO ₂	SrO	Na ₂ O	K₂O	CI	CER [#]
No.	Туре	70	70	70	70	70	70	70	70	%	ppm	%	%	ppm	
1	"83M "	37.5 0	52.9 1	1.00	0.33	0.38	0.86	0.83	2.18	3.3	8125	0.22	0.17	628	1.3
2	"88P"	39.7 3	53.8 9	0.20	0.05	0.10	1.77	1.48	2.20	0.60	8829	0.45	0.01	517	1.1
3	"80M "	38.0 0	54.3 4	1.11	0.09	0.32	0.88	0.78	2.27	3.1	7700	0.18	0.07	995	1.2

Table 2. Composition of rock phosphate samples prepared for phosphoric acid evaluation campaigns*.

#All analysis given as per cent (m/m) unless otherwise stated

^{*}CER is the corrosion equivalent ratio, which is an expression of the molar ratio of the amounts of reactive SiO₂, Al_2O_3 , and the MgO in the rock as compared to fluoride.

As a validation of data obtained, results of campaigns are also compared with Pierre Becker's "Fosprice" model, which predicts, among other things, the overall efficiency based on rock analysis and production capacity. The results of this model comparison appear in Appendix A of this article.

3.1 Campaign Results

The parameters attained at steady state, together with production efficiencies such as filtration rates, and P_2O_5 to rock and sulphuric acid ratios, appear in Table 3a and 3b. Acid quality and corrosion rates on 904L stainless steel are shown in Table 4.

Two sets of results are reflected per case in Tables 3a and 3b, that compare the conversion efficiency with or without the aid of crystal habit modifier. Table 3a displays the reagent input and reactor digestion conditions and 3b shows the efficiency parameters obtained (post filtration).

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Filter ac Produce a/hr	872	882	814	1052	881	908
Filter acid P ₂ O ₅ % (m/m)	27.1	27.1	28.4	29.5	26.4	27.0
Filter acid Density ɑ/cm³	1.289	1.289	1.291	1.298	1.290	1.292
Slurry Solids % (m/m)	29.1	28.9	29.8	28.4	29.1	29.8
Slurry Produced a/hr	2303	2274	2273	2169	2349	2267
Slurry Density ɑ/cm³	1.481	1.479	1.487	1.484	1.481	1.489
Filtration Rate TP₂O₅/m²/d	4.5	5.2	4.6	8.2	4.1	6.3
Retention Time hrs	3.02	3.05	30.6	3.21	2:95	3.07
H ₂ SO4 Consump. q/hr	415	428	425	417	422	416
Rock Consump. a/hr	437	454	440	439	677	445
C.H.M. g/ton Rock	0	293	0	206	0	142
Rock Type	83M		88P		80M	
Case No.	-		2		3	

Summary of operating conditions and data obtained from pilot-plant case study campaigns. Table 3a.

Summary of operating conditions and data obtained from pilot-plant case study campaigns (continued). Table 3b.

P ₂ O ₅ Accoun t. %	103.1	101.8	100.3	100.7	100.9	100.4
-1₂SO₄/P₂O₅ Ratio	2.47	2.43	2.40	2.32	2.60	2.52
tock/P₂O₅ Ratio	2.54	2.60	2.56	2.56	2.70	2.70
l₂SO₄/Rock Ratio	0.97	0.93	0.97	0.95	0.96	0.94
Total fficiency %	96.4	97.2	97.3	95.6	96.1	97.2
Recycle acid P ₂ O ₅ % (m/m)	15.2	15.5	16.3	16.3	14.7	16.0
Recycle acid Density g/cm³	1.16	1.16	1.16	1.16	1.16	1.16
Recycle acid Pumped g/hr	1798	1870	1740	1743	1859	1837
Gypsum Inreacted P ₂ O ₅ % (m/m)	0.41	0.40	0.37	0.91	0.61	0.44
Gypsum Co-cryst. P ₂ O ₅ % (m/m)	0.12	0.13	60.0	0.11	0.10	0.12
Gypsum otal P ₂ O ₅ % (m/m)	0.83	0.63	0.63	1.05	0.89	0.64
Gypsum ¹ roduced g/hr	656	657	677	617	677	675
Rock Fype	83M		88P		80M	
Case No.	-		2		ю	

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Case No.	Rock Type	P ₂ O ₅ %	CaO %	MgO %	Al ₂ O ₃ %	Fe ₂ O ₃ %	SiO ₂ %	F %	SrO %	Na₂O ppm	K ₂ O %	CI ppm	SO₄ %	Mass loss corrosion (mm/year)
1	"83M"	27.1	0.15	0.78	0.05	0.33	0.97	1.37	0.01	543	0.03	628	2.1	0.031
2	"88P"	28.4	0.11	0.25	0.01	0.11	0.90	1.20	0.01	618	0.02	332	2.2	0.155
3	"80M"	27.0	0.19	0.65	0.04	0.23	0.79	1.33	0.01	605	0.03	995	2.4	0.042

 Table 4.
 Typical composition and quality of filter phosphoric acid*.

* All analysis given as per cent (m/m) unless otherwise stated

3.2 Discussion of results

The results of the pilot-plant campaign are reasonably accurate and demonstrates even small variations in parameters are critical to the outcome of a decision to utilise a certain type and quality of rock. For example, small variations in rock P_2O_5 and impurity content result in considerable differences in the processing efficiency (filtration rate, rock and sulphuric acid to P_2O_5 ratio) of the rock. Interestingly, the same trend is shown by the "Fosprice" model.

Other effects, such as the marked improvement in filtration rate upon addition of crystal habit modifier, which can not be predicted by the model, are also depicted in the results of the campaigns. The addition of organic based modifier is not a necessity, particularly in the case of "88P" (and other sedimentary rocks), but certainly makes control of the operation simpler and more efficient.

The model would also not be able to predict the degree of post-precipitation and overall recovery efficiency upon concentration of the weak product phosphoric acid to 54 per cent. This can be specifically determined by simple laboratory concentration tests conducted in a vacuum rotary evaporator in which the amount and type of post-precipitate can be analysed. This, however, would not account for the effects of contaminating ions in recirculating process water in a full-scale situation. Samples of such water would have to be obtained from the client and run as make-up water in the pilot-rig to determine its full effect.

In all instances, it is interesting that the sulphuric acid and rock to P_2O_5 ratios as obtained in the campaign results are always better than that of the model. It is important to note that the pilot-campaign results would also be better (higher filtration rates lower rock and acid ratios) than full-scale operations, due to the ideal and better-controlled conditions under which small scale campaigns are run. With due consideration and comparison to historic data, such as obtained by the product on both pilot and full scale plant (which is the case with "80M", which yielded results very close to that of the model), the predictability of any other product (including sedimentary origin rocks) can be obtained with a high degree of confidence.

Corrosion results show some unexpected trends in that while the corrosion equivalence ratio (CER) can give a good indication of the rate due to uncomplexed fluoride, it nevertheless can not predict the overall corrosion rate. This can be seen when comparing case 1,2 and 3 where case 2 had the lowest CER value (1.1) but a relatively

high corrosion rate (0.155mm/year) was obtained. It is important therefore, to look at the values of chloride, and to obtain actual corrosion data before predicting what the rate of corrosion would be.

4. CONCLUSIONS

- Although the Foskor pilot-plant is relatively small in scale, accurate and meaningful results can be obtained.
- Utilisation of the data obtained from the pilot-plant substantially reduced the financial and technical risk and provided relevant information crucial to the decision making process.
- Together with other tools such as corrosion measurement and acid concentration tests, and used in conjunction with a modelling program such as Pierre Becker's "Fosprice" the performance of any product, igneous or sedimentary, can be predicted with high degree of certainty.
- Foskor is therefore confident to provide this service free of charge to its clients.

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6. REFERENCES

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APPENDIXES A

"Fosprice" Modelling Results



Computer software by Pierre Becker / Duetag-France

13



Computer software by Pierre Becker / Duetag-France



Computer software by Pierre Becker / Duetag-France

APPENDIX B Figures 4 and 5



Figure 4. Latest expansion at Foskor's metallurgical facility, showing new milling technology.



Figure 5. Foskor's phosphoric acid pilot-plant facility showing feed pumps, reactors, rock screw feeder and maturation tank.