Tests for the Production of Phosphoric Acid with Fosfertil Catalão Rock

Study of the Effect of Specific Volume on Filterability & Efficiency.

by
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1 – AIMS OF THE TEST

The aim of the test was to provide data on the Specific Volume (S_v) has on the Efficiency and Filterability of the Process. This data would later be used to calculate the optimum additional volume required for the optimisation of the plant at the present capacity and/or the optimum for an eventual future capacity of 600 tpd P2O5.

2 – INTRODUCTION

The principal objectives of the test were:-

1. To test the phosphate concentrate from the Fosfertil mining and beneficiation plant in Catalão with variation of the S_v. The design S_vs of 0.9, 1.35, 1.80, 2.40 & 3.00 m^3/ tpd P_2O_5 were tested all with a dosage rate of crystal habit modifier of 220 ppm /ton phosphate. The plant presently uses DBS as a crystal habit modifier at this dosage rate as filterability of the gypsum produced from this phosphate without this additive is very poor.

2. To obtain a curve of the expected process efficiency as a function of the specific volume based on the results.

3. To obtain a curve of the filterability as a function of the S_v based on the results. This filterability to be determined by leaf tests with three volumes of slurry to obtain three thicknesses of cake. The wash water used to be determined by the water balance.

4. To check the effect of the S_v on crystal growth, shape and size by checking the SSA (Specific Surface Area) and the Permeability of the gypsum produced. Confirm the correlation with the filterability results.

5. At what would be considered an adequate S_v to execute a confirmatory test without addition of DBS to confirm that volume alone cannot substitute the crystal habit modifier.

The tests lasted 20 days and the operational parameters and the results are set out below,

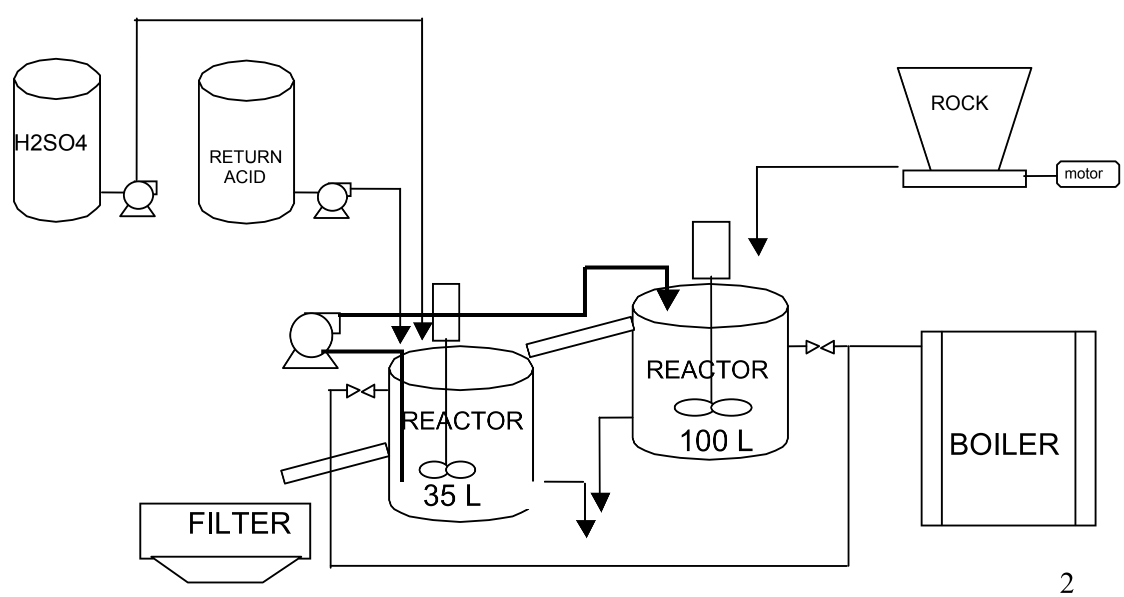
Table 2.1 – Cronogram of the test.

Test	*S_v (m^3/ tpd P_2O_5)*	*Dosage of DBS (ppm)*	*% SO_4 in Product Acid*	*% P_2O_5 in Product Acid*	*% Solids*
1	0.90	220	2.50	29.0	34.0
2	1.35	220	2.50	29.0	34.0
3	1.80	220	2.50	29.0	34.0
4	2.40	220	2.50	29.0	34.0
5	3.00	220	2.50	29.0	34.0
6	1.80 without DBS	220	2.50	29.0	34.0

3- TEST PROCEDURE

For the tests a configuration of two reactors in series was used to represent the manner that eventual increase in reaction volume might be executed in the industrial plant. These two reactors had different volumes and the reagents and additives were fed according to the following scheme.

Figure 3.1: Configuration proposed for the test.



This configuration was selected with an initial low sulphate zone where the phosphate was added flowed by a second zone where mixed sulphuric acid and return acid are added. A defined flow of slurry is recycled from the second zone to the first zone to maintain a specific sulphate gradient.

To enable this slurry recirculation a peristaltic pump was purchased for this specific test and the sizing was made so as to simulate the present flow of slurry through the flash cooler on the industrial plant.

However even though the pump was purchased for 800 l/h it could not maintain a constant flow above 500 – 600 l/h. As such modifications were made to the suction and the variable speed drive which enable 700 l/h to be achieved. Due to this limitation the circuit of the test rig had to be slightly changed so that at lower S_vs of 0.9, 1.35 e 1.80 sulphuric acid was added to the first reactor to compensate for the lack of recycle flow at a dosage rate to maintain the expected sulphate gradient. As such the rig for these tests was as follows. At higher S_vs the original circuit was possible as the pump had enough capacity to maintain the process conditions.

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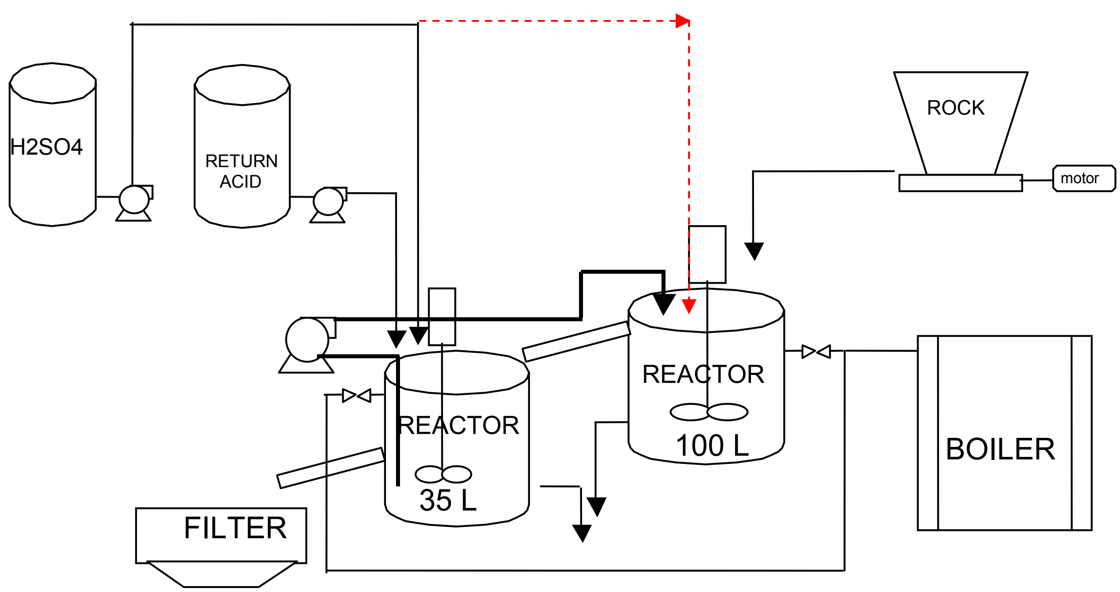


This set-up enabled the test to continue with the distribution of sulphuric acid, for the lower S_vs such that the sulphate level in the first reactor could be maintained at 18 – 22 g/l and 28 – 32 g/l in the second reactor. For S_vs above 2.4 the recirculation rate was adequate to maintain these conditions.

4- AVERAGE ANALYSIS OF THE PHOSPHATE CONCENTRATE TESTED

Table 4.1: *Average analysis of the phosphate tested*

(%)	*Phosphate from Fosfertil Catalão mine*
P_2O_5	35.88
CaO	47.96
CãO/P_2O_5	1.337
Al_2O_3	0.36
MgO	0.56
Fe_2O_3	2.55
SiO_2	2.14
F	2.20
TiO_2	0.98
BaO	0.96
SrO	0.68
> # 100	15.33
> # 200	52.92
> # 325	77.48

5- RESULTS OBTAINED

The tables below show the results of each phase of the tests.:

Table 5.1 – Reaction Efficiency & Filtration Rate as a function of S_v.

Test	*Specific Volume (m^3/ tpd P_2O_5)*	*Insoluble Losses (% P_2O_5) Dry DH Base*	Attack Efficiency (%)	*Filtration Rate (tpd P_2O_5 / m^2) Cycle 42 seconds*
1	**0.90**	1.79	92.37	13.11
2	**1.35**	1.26	94.84	16.29
3	**1.80**	1.02	95.87	15.50
4	**2.40**	0.75	96.94	15.66
5	**3.00**	0.74	96.98	16.72
6	**1.80 w/o DBS**	1.01	95.91	*7.24*

Table 5.2 – Operating parameters and analytical results

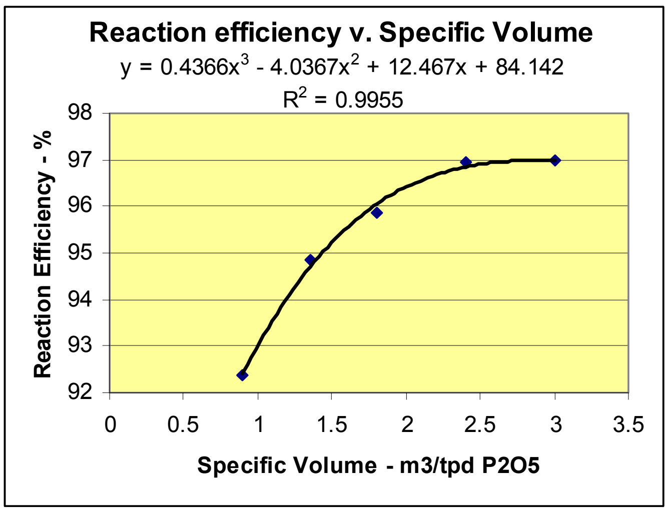
Test	***1***	**2**	**3**	**4**	**5**	**6**
S_v (m^3/tpd P_2O_5)	**0.90**	***1.35***	***1.80***	***2.40***	**3.00**	**1.80 w/o DBS**
g/l SO_4 1^0 Reator	*19.90*	*19.93*	*20.87*	*26.70*	*25.32*	***21.43***
g/l SO_4 2^0 Reator	*30.37*	*29.47*	*29.70*	*32.45*	*32.44*	***30.51***
P_2O_5 (%) Solids free basis	*28.84*	*29.39*	*29.33*	*29.16*	*28.61*	***28.92***
Sólidos % (w/w)	*33.5*	*32.9*	*35.7*	*34.4*	*34.0*	***33.90***
% P_2O_5 Unreacted	*1.09*	*0.54*	*0.34*	*0.22*	*0.22*	***0.49***
% P_2O_5 Cocrystallised	*0.70*	*0.71*	*0.68*	*0.53*	*0.52*	***0.52***
SSA (cm^2/g)	*2376*	*1495*	*1270*	*1240*	*1329*	***3780***
Slurry viscosity (cP)	*41*	*19*	*23*	*21*	*26*	***200***

6 – ANALYSIS OF THE RESULTS

6.1 – *In relation to the attack efficiency*

In analysing Table 5.1 graphically, it can be clearly seen that an increase in reaction volume has a decisive effect on the improvement up to a S_v of about 2.4, after which the curve flattens off and the gains become relatively small.:

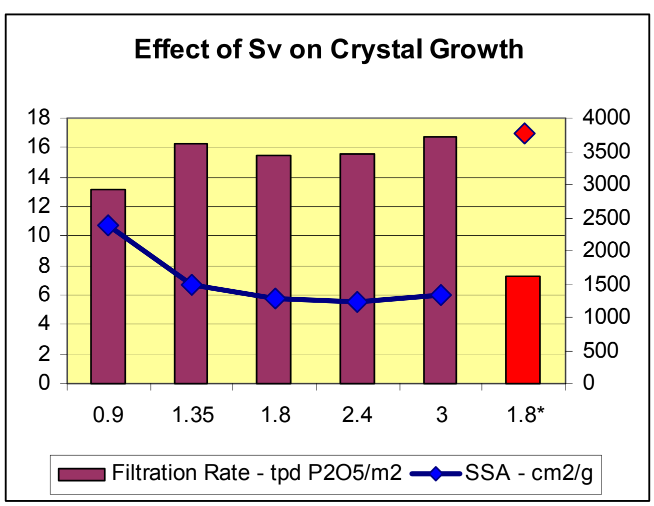
Graph 6.1.1: Reaction efficiency versus Specific Volume



6.2 – *In relation to the Filtration Rate*

Below is shown a graph representing the results with respect to crystal growth and filtration rate.

Graph 6.2.1 – Filtration rate and SSA versus Specific Volume



The graph above clearly demonstrates the inverse relationship between the SSA of the crystals and the filtration rate. The results in red are without the addition of DBS.

Analysing the results it can be seen that, surprisingly for this phosphate and with the addition of DBS, reaction volumes above the S_v value of 1.35 or even 1.8 do not seem to improve the quality of the crystal. But at a S_v of 0.9 the filtration rate is very poor.

The test made without the addition of DBS at a S_v of 1.8 produce poor crystals with a SSA of over 3000 and a filtration rate of about 7 tpd P2O5/m2 at a cycle time of 42 seconds.

7- CONCLUSIONS

- There is a progressive improvement in Reaction Efficiency with an increase in S_v up to a value of 2.40 m^3/tpd P_2O_5. At this Sv the efficiency is of the order of 97% and use of larger volumes does not seem to improve this value to any real extent.

- In the tests with the addition of DBS, above the S_v value of 1.35 there seems to be no significant improvement in the filterability with additional reaction volume.

- The elimination of the use of DBS by the use of additional volume does not seem to be economically viable.

- From these results it seems that the optimum value of S_v seems to be between 1.8 and 2.4 m3/tpd P_2O_5, and investment costs for these two volumes should be evaluated. This along with the operating cost should enable the optimum economic size of reactor for this phosphate.