

# Study of Reverse Flotation for a Collophanite with Sulfuric Acid

Shi Guiming<sup>1,2</sup>, Zhou Yichao<sup>1</sup> and Li Ming<sup>1,a</sup>

<sup>1</sup>College of Chemistry Biology and Environment, Yuxi Normal University, Yuxi 653100, China

<sup>2</sup>School of Resources and Environmental Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China

<sup>a</sup> Corresponding author: liming@yxnu.edu.cn

**Abstract:** Mineralogy and flotation test of a collophanite were carried out. Under the grinding fineness of -0.074 mm 78%, the phosphorus concentrate contained yield of 53.83%, grade of 35.49%, recovery of 88.12%, MgO grade of 1.22% was obtained through a closed-circuit processes of one roughing, two cleaning and one scavenging with the sulfuric acid, TF-64. The quality of phosphorus concentrate had reached the II grade of HG/T2673-95 standard.

## 1. Preface

Phosphate rock is an important strategic resource and a non-renewable resource, which provides an important source of fertilizer for modern agriculture and is also the material basis of fine phosphorus chemical industry [1-3]. According to the composition of its impurity minerals, it can be divided into siliceous (mainly containing quartz, chalcedony, silicate, et al.), calcium-magnesium (mainly containing calcite and dolomite, et al.) and silicon-calcium -magnesium mixed phosphate ore [4-6]. The reserves of phosphate mineral resources of Sichuan province are large amount, but their phosphate mineral resources are mainly sedimentary phosphate rock deposits, which are very difficult to beneficiation and high cost of beneficiation. Enterprises have low economic benefits and some even deficiency [7-8].

In this paper, the mineralogy and beneficiation of a phosphate rock in China were carried out, and obtained better indexes.

## 2. Ore properties

### 2.1 Chemistry multiple analysis results of raw ore

Chemistry multiple analysis results of raw ore were shown in table 1.

Table 1. Chemistry multiple analysis results of raw ore (%).

Items	P <sub>2</sub> O <sub>5</sub>	MgO	CaO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O
Contents	21.58	9.45	42.54	0.320	0.096	0.048

Items	Na <sub>2</sub> O	SiO <sub>2</sub>	CO <sub>2</sub>	F	Others	-
Contents	0.150	1.44	17.75	2.01	4.616	-

## 2.2 Composition of minerals

The ore is identified as phosphorus block rock ore through identification that the mineral composition is relatively simple. The main components in the ore are colloidal apatite, dolomite and a small amount of quartz (chalcedony). The dolomite phosphorite is mottled structure, dense striated structure and granular structure. The rock is a granular structure, which supported by matrix and basal cemented by basement.

## 3. Experimental results and discussion

### 3.1 Grinding fineness test

The grinding fineness test process and the agent conditions were shown in figure 1, and the test results were shown in table 2.

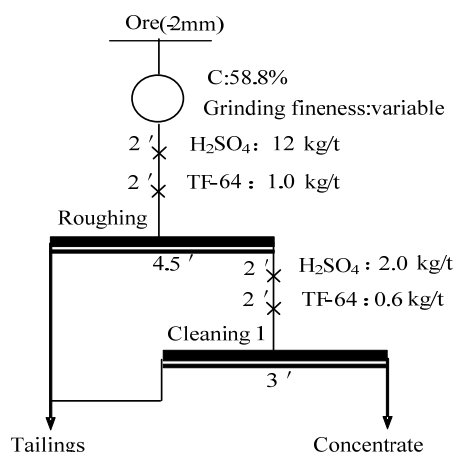


Figure 1. Flowchart of grinding fineness test.

Table 2. Results of grinding fineness -0.074 mm test (%).

Grinding fineness	Items	Yield	Grade		Recovery	
			P <sub>2</sub> O <sub>5</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	MgO
72.2	Concentrate	52.13	34.13	1.78	82.11	9.94
	Tailings	47.87	8.10	17.57	17.89	90.06
	Raw ore	100.00	21.67	9.34	100.00	100.00
78.6	Concentrate	49.34	35.70	1.14	81.24	6.01
	Tailings	50.66	8.03	17.35	18.76	93.99
	Raw ore	100.00	21.68	9.35	100.00	100.00
88.4	Concentrate	48.46	35.72	1.02	79.84	5.28
	Tailings	51.54	8.48	17.18	20.16	94.72
	Raw ore	100.00	21.68	9.35	100.00	100.00

It can be seen from table 2 that with the increase of grinding fineness (-0.074 mm), the yield of concentrate gradually decrease, the grade of  $P_2O_5$  increase, the grade of MgO decrease, and the recovery rate of  $P_2O_5$  decrease. When the grinding fineness -0.074 mm increase to 78.6%,  $P_2O_5$  grade in concentrate is 35.70%, MgO content is 1.14%,  $P_2O_5$  recovery rate is 81.24%, and MgO exclusion rate is 93.99%. The grade of  $P_2O_5$  in concentrate change little, and the recovery rate decrease slightly as the grinding fineness increase gradually. The grinding fineness of -0.074 mm was determined as 78.6% after comprehensive consideration.

### 3.2 Dosage test of sulfuric acid as roughing inhibitor

Figure 2 shows the test process of dosage test of sulfuric acid as roughing inhibitor. Grinding fineness was 78.60% of -0.074 mm and TF-64 dosage was 1.4 kg/t. The test results were shown in table 3.

It can be seen from table 3 that with the increase of the sulfuric acid dosage, the yield of concentrate gradually decrease, the grade of  $P_2O_5$  in concentrate gradually increase, the grade of MgO gradually decrease, and the recovery rate of  $P_2O_5$  gradually decrease, while the yield of tailings gradually increase, and the grade of  $P_2O_5$  in tailings decrease first and then increase. When the amounts of sulfuric acid exceeds 14 kg/t, the grade of  $P_2O_5$  in tailings increases slightly. Therefore, the dosage of sulfuric acid as roughing inhibitor was determined as 14 kg/t.

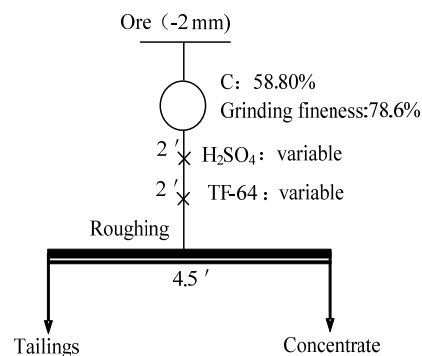


Figure 2. Flowchart of roughing.

Table 3. Dosage of sulfuric acid as roughing inhibitor (%).

Sulfuric acid Dosage	Items	Yield	Grade		Recovery	
			$P_2O_5$	MgO	$P_2O_5$	MgO
12 kg/t	Concentrate	64.67	30.08	4.84	89.71	33.48
	Tailings	35.33	6.31	17.60	10.29	66.52
	Raw ore	100.00	21.69	9.35	100.00	100.00
14 kg/t	Concentrate	60.79	31.67	3.94	88.76	25.65
	Tailings	39.21	6.22	17.71	11.24	74.35
	Raw ore	100.00	21.69	9.34	100.00	100.00
16 kg/t	Concentrate	53.79	34.61	2.29	85.87	13.18
	Tailings	46.21	6.63	17.57	14.13	86.82
	Raw ore	100.00	21.68	9.35	100.00	100.00

### 3.3 Dosage test of TF-64 as roughing collector

Figure 2 shows the test process of dosage test of TF-64 as roughing collector. Grinding fineness was 78.60% of -0.074 mm and sulfuric acid dosage was 14 kg/t. The test results were shown in table 4.

Table 4. Dosage results of TF-64 as roughing collector (%).

TF-64 Dosage	Items	Yield	Grade		Recovery	
			P <sub>2</sub> O <sub>5</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	MgO
1.0 kg/t	Concentrate	60.55	31.88	3.79	88.99	24.57
	Tailings	39.45	6.05	17.86	11.01	75.43
	Raw ore	100.00	21.69	9.34	100.00	100.00
1.2 kg/t	Concentrate	57.46	32.78	2.98	86.87	17.78
	Tailings	42.54	6.69	17.37	13.13	82.22
	Raw ore	100.00	21.69	9.35	100.00	100.00
1.4 kg/t	Concentrate	54.76	33.48	2.95	84.52	17.30
	Tailings	45.24	7.42	17.07	15.48	82.70
	Raw ore	100.00	21.69	9.34	100.00	100.00

It can be seen from table 4 that with the increase of the TF-64 dosage, the yield of concentrate gradually decrease, the grade of P<sub>2</sub>O<sub>5</sub> in concentrate gradually increase, the grade of MgO gradually decrease, and the recovery rate of P<sub>2</sub>O<sub>5</sub> gradually decrease, while the MgO exclusion rate of tailings gradually increase. When the TF-64 dosage exceed 1.2 kg/t, the grade of P<sub>2</sub>O<sub>5</sub> in tailing increase slightly, the grade of MgO change slightly, while the recovery of P<sub>2</sub>O<sub>5</sub> decrease obviously. Therefore, the dosage of TF-64 as roughing collector was determined as 1.2 kg/t.

### 3.4 Dosage test of sulfuric acid in cleaning 1

Figure 3 showed the test process of sulfuric acid dosage test and conditions in cleaning 1. The TF-64 dosage was 0.6 kg/t. The test results were shown in table 5.

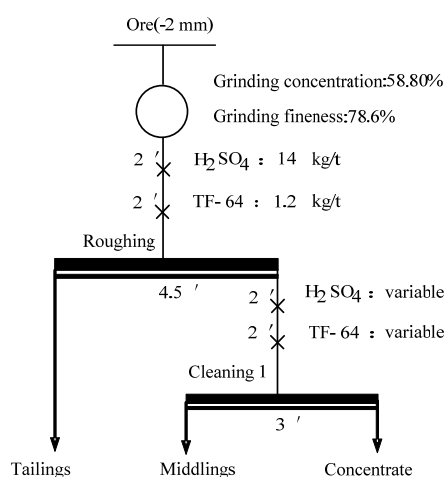


Figure 3. Flowchart of sulfuric acid dosage test in cleaning 1.

Table 5. Results of sulfuric acid dosage in cleaning 1 (%).

Sulfuric acid dosage	Items	Yield	Grade		Recovery (%)	
			P <sub>2</sub> O <sub>5</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	MgO
0.5 kg/t	Concentrate	48.86	35.42	1.33	79.85	6.94
	Middlings	8.74	15.65	11.33	6.31	10.58
	Tailings	42.40	7.07	18.21	13.84	82.48

	Raw ore	100.00	21.67	9.36	100.00	100.00
1.0 kg/t	Concentrate	48.79	35.26	1.26	79.37	6.58
	Middlings	8.34	16.45	12.61	6.33	11.25
	Tailings	42.87	7.23	17.92	14.30	82.17
	Raw ore	100.00	21.68	9.35	100.00	100.00
1.5 kg/t	Concentrate	48.45	35.30	1.27	78.84	6.58
	Middlings	8.86	16.72	12.34	6.83	11.70
	Tailings	42.69	7.28	17.88	14.33	81.72
	Raw ore	100.00	21.69	9.34	100.00	100.00

It can be seen from table 5 that the grade of  $P_2O_5$  in the middling is lower, the recovery rate is higher, indicated that it is necessary to add sulfuric acid in the cleaning 1. When the sulfuric acid dosage in cleaning 1 gradually increase, the concentrate yield,  $P_2O_5$  and MgO grade change insignificantly, while the exclusion rate of MgO gradually increase. The MgO exclusion rate has reached 63.10% with sulfuric acid dosage is 1.0 kg/t, while the MgO exclusion rate increase by only 0.90% with sulfuric acid increase to 1.5 kg/t. Therefore, the sulfuric acid dosage in cleaning 1 was determined as 1.0 kg/t.

### 3.5 Dosage test of TF-64 in cleaning 1

Figure 3 shows the test process of TF-64 dosage test and conditions in cleaning 1. The sulfuric acid dosage was 1.0 kg/t. The test results were shown in table 6.

Table 6. Results of TF-64 dosage in cleaning 1 (%).

TF-64 Dosage	Items	Yield	Grade		Recovery	
			$P_2O_5$	MgO	$P_2O_5$	MgO
0.4 kg/t	Concentrate	51.09	34.57	1.70	81.43	9.30
	Middlings	6.07	14.84	12.78	4.15	8.31
	Tailings	42.84	7.30	17.97	14.42	82.39
	Raw ore	100.00	21.69	9.34	100.00	100.00
0.6 kg/t	Concentrate	48.64	35.49	1.22	79.62	6.34
	Middlings	8.59	15.33	12.27	6.08	11.27
	Tailings	42.77	7.25	18.01	14.30	82.39
	Raw ore	100.00	21.68	9.35	100.00	100.00
0.8 kg/t	Concentrate	47.49	35.52	1.23	77.84	6.24
	Middlings	9.54	17.27	11.42	7.60	11.63
	Tailings	42.97	7.34	17.89	14.56	82.13
	Raw ore	100.00	21.67	9.36	100.00	100.00

It can be seen from table 6 that the recovery of  $P_2O_5$  decrease gradually and the exclusion rate of MgO increase with the TF-64 dosage increase in cleaning 1. When the TF-64 dosage in cleaning 1 increase to 0.6 kg/t, the partly recovery of  $P_2O_5$  is 92.91% and the exclusion rate of MgO is 64.00%. Therefore, the TF-64 dosage in cleaning 1 was determined as 0.6 kg/t.

### 3.6 Open-circuit test

After the above series of condition tests, the reagent system of roughing, scavenging and cleaning 1 were determined. The test process and operating conditions were shown in figure 4, and the test results were shown in table 7.

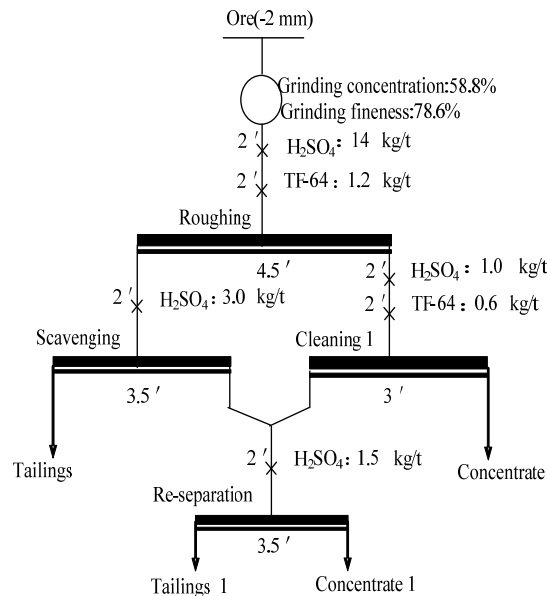


Figure 4. Flowchart of open-circuit.

Table 7. Results of open-circuit (%).

Items	Yield	Grade		Recovery	
		P <sub>2</sub> O <sub>5</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	MgO
Concentrate	48.59	35.42	1.21	79.39	6.29
Concentrate 1	4.31	32.78	2.35	6.52	1.08
Tailings 1	4.96	10.93	12.10	2.50	6.42
Tailings	42.14	5.96	19.13	11.59	86.21
Concentrate + Concentrate 1	52.90	35.21	1.30	85.91	7.37
Tailings 1 + Tailings	47.10	6.48	18.39	14.09	92.63
Raw ore	100.00	21.68	9.35	100.00	100.00

Table 7 shows that the concentrate 1 with a grade of 32.78% is obtained after re-separation for middlings. The concentrate and concentrate 1 are combined as the final concentrate, which P<sub>2</sub>O<sub>5</sub> grade is 35.21% and MgO grade is 1.30%, and P<sub>2</sub>O<sub>5</sub> recovery rate is 85.91%.

### 3.7 Closed-circuit test

In order to ensure the concentrate quality of the closed-circuit test, we added a cleaning during the closed-circuit test. The reagents had been properly adjusted because there were some reagents brought back in the middlings. The closed-circuit test process and reagent conditions were shown in figure 5, the test results were shown in table 8.

Table 8 shows that the yield, P<sub>2</sub>O<sub>5</sub> grade, MgO grade, P<sub>2</sub>O<sub>5</sub> recovery rate of concentrate are 53.83%, 35.49%, 1.22% and 88.12%. The yield, P<sub>2</sub>O<sub>5</sub> grade, MgO grade, P<sub>2</sub>O<sub>5</sub> loss rate of tailings are 46.17%, 5.58%, 18.83%, 11.88% and 92.97%. Phosphorus concentrate quality had reached the II grade of HG/T2673-95 standard.

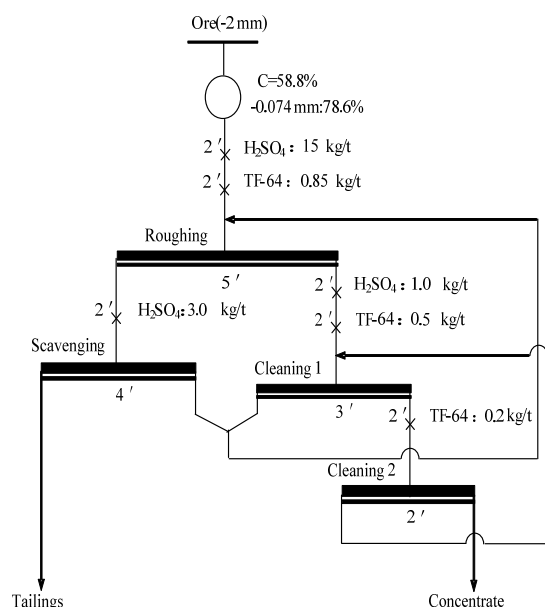


Figure 5. Flowchart of closed-circuit.

Table 8. Results of closed-circuit (%).

Items	Yield	Grade		Recovery	
		P <sub>2</sub> O <sub>5</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	MgO
Concentrate 1	13.46	35.48	1.22	22.03	1.76
Concentrate 2	13.54	35.42	1.19	22.12	1.73
Concentrate 3	13.43	35.61	1.25	22.06	1.80
Concentrate 4	13.40	35.45	1.21	21.91	1.74
Subtotal	53.83	35.49	1.22	88.12	7.03
Tailings 1	11.54	5.58	18.83	2.97	23.24
Tailings 2	11.48	5.46	18.95	2.89	23.27
Tailings 3	11.60	5.64	18.79	3.02	23.31
Tailings 4	11.55	5.63	18.74	3.00	23.15
Subtotal	46.17	5.58	18.83	11.88	92.97
Total	100.00	21.68	9.35	100.00	100.00

#### 4. Conclusions

(1) The phosphate ore has good washability, and the grinding fineness should be around -0.074mm of 78%.

(2) The phosphate concentrate with yield of 53.83%, P<sub>2</sub>O<sub>5</sub> grade of 35.49%, P<sub>2</sub>O<sub>5</sub> recovery of 88.12%, MgO grade of 1.22% was obtained by a closed-circuit processes of one roughing, two cleaning and one scavenging that used sulfuric acid and TF-64 as combination agents. The phosphorus concentrate quality had reached the II grade of HG/T2673-95 standard.

#### Acknowledgements

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