Reduction Study on MgO in a Magnesium Phosphate Ore of Sedimentary Rock Type

Shi Guiming[1,2], Zhou Yichao[1] and Li Ming[1,a]

[1]College of Resources and Environment，Yuxi Normal University，Yuxi 653100，China

[2]School of Resources and Environmental Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China

[a] Corresponding author: liming@yxnu.edu.cn

Abstract. The paper described the mineralogical study and selection experiment of a magnesia phosphate ore in Sichuan province. A good results of P_2O_5 grade of 33.96%, recovery of 93.51%, MgO grade of 1.42% were obtained by one roughing and one cleaning reverse flotation process with low cost of sulfuric acid and PO-3.

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, etc), calcium-magnesium (mainly containing calcite and dolomite, etc.) 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 sedimentary magnesite phosphate rock in Sichuan province were carried out, and obtained better indexes.

2.Properties of raw ore

2.1 Chemical composition

Multiple chemistry analysis of raw ore was shown in table 1.

Table 1. Results of multiple chemistry analysis for raw ore %.

Items	P_2O_5	MgO	CaO	Fe_2O_3	Al_2O_3
Contents	25.34	6.36	44.52	0.27	0.32
Items	K_2O	Na_2O	SiO_2	CO_2	F
Contents	0.21	0.03	3.39	15.54	2.53

2.2 Material composition and structure

The mineral composition of the ore was simple, which mainly consisted of apatite and dolomite, a small amount of secondary siliceous minerals (chalcedony), a small amount of hydromite, lignite and pyrite and a small amount of biodebris (carbon). The construction of the ore was also simple, which mainly composed of micritic apatite aggregates and cementite dolomite, with a small amount of secondary siliceous (chalcedony) distributed on the dorsal base of apatite and dolomite as veins, irregular and sparse. The structure was mainly striated-strip structure, a few compact massive structure, and porous structure formed by weathering and dripping of ore.

2.3 Size screening and metal distribution of raw ore

Size screening and metal distribution of raw ore were shown in figure 1.

Figure 1 shows that when the particle size is less than 0.074 mm, the grade of P_2O_5 decrease rapidly, while the grade of MgO rise rapidly, it can be explained that the main mineral apatite containing P_2O_5 is harder than the main mineral dolomite containing MgO, and the easy grinding of dolomite may bring MgO into the fine grain grade. At the same time, the distribution rate of MgO in the -0.074 mm grain level is very high and the distribution rate of MgO may be higher after grinding, which may seriously affect the indicators of MgO in the subsequent flotation products. Therefore, the selective effect of grinding should be notice in the preparation process of slurry.

3.Experimental instruments and raw materials

Main test instruments and equipment contained: XMB-250 x 100 cone ball mill, which produced from Shanghai zhetai machinery manufacturing co., ltd. XFD type single channel flotation machine which produced form China changchun prospecting machinery plant. DHG-9623A type electro- thermostatic blast oven which produced from Shanghai jinghong test equipment co., ltd. Standard screens of 0.15 mm, 0.074 mm, 0.038 mm, 0.019 mm and 0.01 mm from no.450 plant of China aviation industry corporation.

Agent contained: phosphoric acid (H3PO4)(chemically pure) be made into 10 g/100 mL aqueous solution, PO-3 collector(industrial grade), sulfuric acid (H_2SO_4) (chemically pure) be made into 10 g/100 mL aqueous solution.

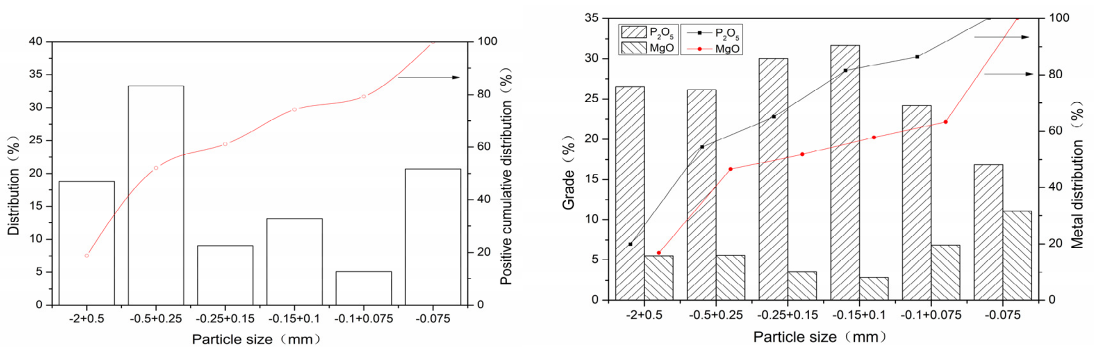


Figure 1. Size screening and metal distribution.

4.Experiments

The reverse flotation process with H_2SO_4-PO_3 composite reagent was finally determined based on the preliminary exploration of flotation reagent.

4.1 Sulfuric acid dosage test of roughing

According to the process and conditions shown in figure 2, the sulfuric acid dosage experiment of roughing was carried out, and the results were shown in figure 3.

Figures 3a, 3b and 3c shows that with the increase of sulfuric acid dosage in roughing, the tailings production rate shows an upward trend, the grade of P_2O_5 in the tailings gradually decrease, and the removal rate of MgO in the tailings gradually increase. With the amount of sulfuric acid increase to 10 kg/t, the productivity of tailings and the removal rate of MgO in tailings increase little. Therefore, the sulfuric acid dosage was determined to be 10 kg/t. The content of P_2O_5 in tailings decrease to about 4% with the amount of sulfuric acid in roughing increased to 8 kg/t. That is to say, the tailings with a yield of about 30% might be discarded after roughing selection, and the scavenging operation could be appropriately reduced. With the increase of sulphuric acid dosage from 3 kg/t to 6 kg/t, the productivity of the middlings decrease from 12.88 % to 1.65 %, and the removal rate of MgO in the middlings decrease from 30.96% to 1.91 %, indicated that the sulfuric acid dosage of the cleaning operation was so large to cause the inhibition effect strong and the separation effect poor.

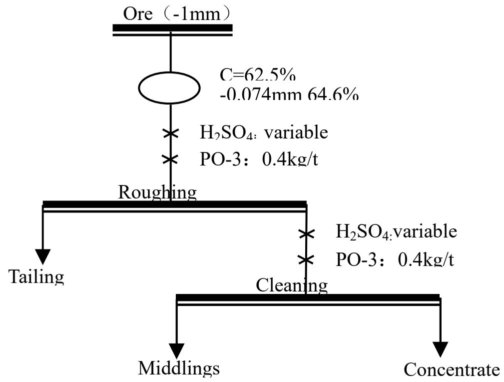
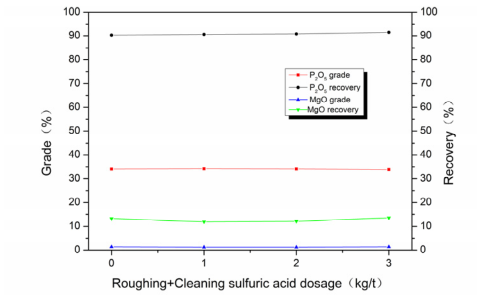
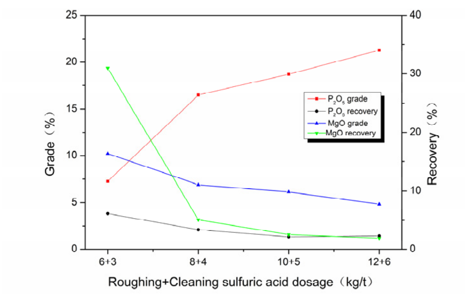


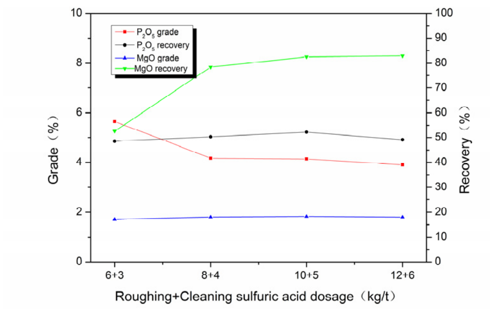
Figure 2. Flowchart of sulfuric acid dosage of roughing.



(a)Concentrate



（b）Middlings



（c）Tailings

Figure 3. Results of sulfuric acid dosage in roughing and cleaning.

4.2 Sulfuric acid dosage test of cleaing

Sulfuric acid dosage test of cleaing was carried out from the process and conditions shown in figure 4, and the test results were shown in table 2.

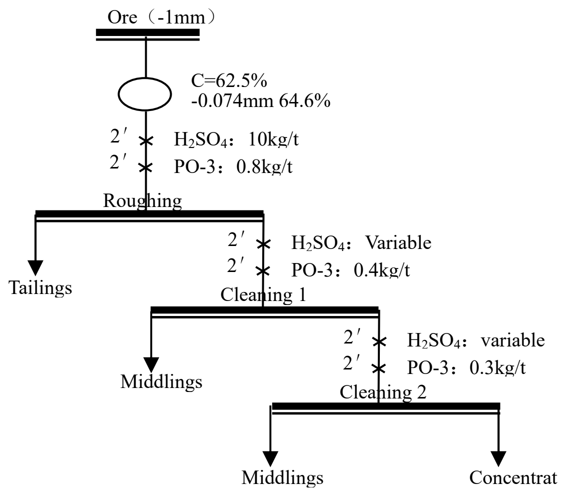


Figure 4. Flowchart of sulfuric acid dosage of cleaning.

Table 2. Results of sulfuric acid dosage of cleaning %.

Sulfuric acid dosage（kg/t）	**Items**	**Yield**	**Grade**		**Recovery**	
			P_2O_5	MgO	P_2O_5	MgO
Cleaning 1：0 Cleaning 2：0	Concentrate	62.56	34.31	1.26	87.71	11.85
	Middling 1	2.25	28.01	4.12	2.57	1.40
	Concentrate+ Middling 1	64.81	34.09	1.36	90.28	13.25
	Middling 2	3.89	22.52	7.46	3.58	4.36
	Tailing	31.30	4.80	17.50	6.14	82.39
	Raw ore	100.00	24.48	6.65	100.00	100.00

Cleaning 1：1 Cleaning 2：0.5	Concentrate	61.77	34.55	1.04	87.30	9.66
	Middling 1	2.98	26.88	5.08	3.28	2.27
	Concentrate+ Middling 1	64.75	34.20	1.22	90.58	11.93
	Middling 2	4.62	18.02	10.34	3.41	7.20
	Tailing	30.63	4.80	17.54	6.01	80.87
	Raw ore	100.00	24.45	6.64	100.00	100.00
Cleaning 1：2 Cleaning 2：1	Concentrate	63.08	34.35	1.10	88.55	10.62
	Middling 1	2.05	26.87	4.78	2.25	1.50
	Concentrate+ Middling 1	65.13	34.11	1.22	90.80	12.12
	Middling 2	3.69	19.23	9.56	2.90	5.40
	Tailing	31.18	4.94	17.28	6.30	82.48
	Raw ore	100.00	24.47	6.53	100.00	100.00

It can be seen from table 2 that the concentrate and middling 1 as a product already reach the I level of first grade of HG/T2673-95 standard. The content of MgO is less than 1.50 %, the content of P_2O_5 is 34 %, the recovery of P_2O_5 is more than 90%. Therefore, cleaning 2 could be cancelled. When the sulfuric acid dosage add, the exclusion rate MgO in middling 2 first rise and then fall. Therefore, it was determined that added 1 kg/t of sulfuric acid to the cleaning 1.

4.3 PO-3 dosage test

According to the process and operating conditions shown in figure 2, the amount of sulfuric acid was selected as 10 kg/t in roughing, the amount of sulfuric acid was selected as 1 kg/t in cleaning, and the amount of PO-3 was changed. The test results were shown in table 3.

Table 3. Results of PO-3 dosage test %.

PO-3 dosage （kg/t）	**Itmes**	**Yield**	**Grade**		**Recovery**	
			P_2O_5	MgO	P_2O_5	MgO
Roughing：0.67 Cleaning：0.33	Concentrate	67.76	34.14	1.26	91.59	13.87
	Middling	5.35	16.05	11.75	3.40	10.22
	Tailing	26.89	4.70	17.38	5.01	75.91
	Raw ore	100.00	25.26	6.16	100.00	100.00
Roughing：0.8 Cleaning：0.4	Concentrate	67.09	34.33	1.18	90.98	12.70
	Middling	5.65	15.95	11.72	3.56	10.62
	Tailing	27.26	5.07	17.60	5.46	76.68
	Raw ore	100.00	25.32	6.23	100.00	100.00

It can be seen from table 3 that with the dosage of PO-3 increase from 0.67 to 0.93 kg/t, the yield of the tailings, the loss rate of P_2O_5, the loss rate of MgO gradually increase unsignificant which proves the PO-3 dosage is less effective for the roughing. Finally, PO-3 dosage was determined to 0.8 kg/t in roughing. As the dosage of PO-3 increase from 0.33 to 0.47 kg/t in the cleaning, the yield of the middling, the recovery rate of P_2O_5, the recovery rate of MgO in the middling increase rarely which proves the PO-3 dosage is less effective for the cleaning. Finally, PO-3 dosage was determined to 0.4 kg/t in cleaning.

4.4 Grinding fineness test

According to the process and the agent conditions in figure 5, the open-circuit test was carried out with the expanded test sample which inspecting the adaptability of the determined process and conditions for the variation of grinding fineness. The test results were shown in table 4.

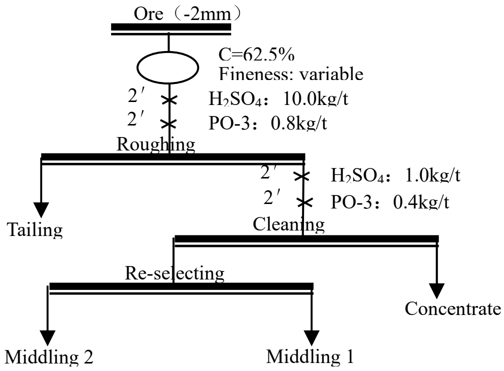


Figure 5. Flowchart of grinding fineness.

It can be seen from table 4 that with the content of -0.074 mm among from 58.5 % to 71.6 %, the phosphorus concentrate with MgO content less than 1.5 % can be obtained, and the recovery rate of P_2O_5 is greater than 92 %, which indicated the selected process and agent conditions had strong adaptability to the fineness of the mineral. With the increase of -0.074 mm content, the content of P_2O_5 is increased and the content of MgO is decreased in concentrate. Finally, the fineness of -0.074 mm was determined to around 65 % in view of the higher grinding cost.

Table 4. Results of grinding fineness %.

-0.074mm Content	**Items**	**Yield**	**Grade**		**Recovery**	
			P_2O_5	MgO	P_2O_5	MgO
58.5	Concentrate	67.70	34.24	1.26	91.31	13.69
	Middling 1	1.67	24.42	6.31	1.60	1.69
	Concentrate+Middling 1	69.37	34.00	1.38	92.91	15.38
	Middling 2	3.07	11.89	13.84	1.44	6.82
	Tailing	27.56	5.21	17.59	5.65	77.80
	Middling 2+Tailing	30.63	5.87	17.22	7.09	84.62
	Raw ore	100.00	25.39	6.23	100.00	100.00

64.6	Concentrate	67.57	34.29	1.23	91.23	13.31
	Middling 1	1.92	24.85	6.09	1.88	1.87
	Concentrate+Middling 1	69.49	34.03	1.36	93.11	15.18
	Middling 2	3.91	11.05	14.61	1.70	9.14
	Tailing	26.60	4.95	17.77	5.19	75.68
	Middling 2+Tailing	30.51	5.73	17.36	6.89	84.82
	Raw ore	100.00	25.40	6.25	100.00	100.00
71.6	Concentrate	66.54	34.38	1.19	90.04	12.41
	Middling 1	2.14	26.83	5.16	2.26	1.75
	Concentrate+Middling 1	68.68	34.14	1.30	92.30	14.16
	Middling 2	4.44	11.93	13.97	2.09	9.87
	Tailing	26.88	5.31	17.76	5.61	75.97
	Middling 2+Tailing	31.32	6.25	17.22	7.70	85.84
	Raw ore	100.00	25.41	6.28	100.00	100.00

4.5 Closed-circuit test

In conclusion, the test was carried out in accordance with one roughing and one cleaning closed-circuit process, and the test results were shown in table 5.

Table 5. Results of closed-circuit %.

Items	**Yield**	**Grade**		**Recovery**	
		P_2O_5	MgO	P_2O_5	MgO
Concentrate	70.01	33.96	1.42	93.51	15.89
Tailing	29.99	5.50	17.50	6.49	84.11
Raw ore	100.00	25.42	6.24	100.00	100.00

It can be seen from table 5 that a good index of recovery rate of concentrate containing P_2O_5 grade of 33.96 %, MgO grade of 1.42 % and P_2O_5 recovery of 93.51 % can be obtained through closed-circuit test.

5.Conclusions

(1) The mineral composition of the ore was simple, which mainly consisted of apatite and dolomite, a small amount of secondary siliceous minerals (chalcedony), a small amount of hydromite, lignite and

pyrite and a small amount of biodebris (carbon). The construction of the ore was also simple, which mainly composed of micritic apatite aggregates and cementite dolomite, with a small amount of secondary siliceous (chalcedony) distributed on the dorsal base of apatite and dolomite as veins, irregular and sparse. The structure was mainly striated-strip structure, a few compact massive structure, and porous structure formed by weathering and dripping of ore.

(2) Combination of sulfuric acid and PO-3 had strong adaptability and good beneficiation indexes for the closed-circuit flotation process. Under the grinding fineness -0.074 mm of 64.6 %, the phosphorus concentration was obtained with one roughing and one cleaning, which reached the I level a grade of the HG/T2673-95 standard. The P_2O_5 grade, MgO grade, P_2O_5 recovery rate of phosphorus concentration were more than 34 %, less than 1.5 %, and greater than 90 %.

(3) Consumptions of flotation reagent were 11.0 kg/t sulfate acid and 1.2 kg/t PO-3 that the costs of them were about 15 RMB per ton raw ore. In summary, sulfuric acid and PO-3 flotation reagents had good mineral processing indexes, simple process and low cost, which has the practical guiding significance for this kind of phosphate ore.

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