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Design and Analysis of Experimental Scheme for Preparation of Gypsum Mortar from Phosphogypsum

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Abstract. Phosphogypsum is a by-product of phosphate fertilizer and food and drug industry. The utilization rate of phosphogypsum is low all over the world. With the shortage of land resources, the pollution of atmosphere and solid waste caused by the storage of phosphogypsum mine becomes more and more acute. The main chemical composition of phosphogypsum is the same as natural gypsum, which is $CaSO_4 \cdot 2H_2O$ The impurities in phosphogypsum have a serious effect on the quality of gypsum products, by removing impurities and adjusting additives, phosphogypsum can be used as a substitute for natural gypsum to prepare gypsum mortar, which can effectively alleviate the shortage of natural gypsum supply, it can also absorb phosphogypsum and solve the environmental problems brought by phosphogypsum. The results show that the green and excellent performance phosphogypsum mortar can be prepared by the ratio of phosphogypsum 35%, cement 10%, aggregate 14%, boron mud 5%, fly ash 16%, hemp knife 5%, and the other 15%.

Keywords. Phosphogypsum, impurities, selection, mixture ratio, content

1. Introduction

Phosphogypsum is a by-product of the phosphate fertilizer and food and drug industries [1]. It has three forms: $CaSO_4 \cdot 0H_2O$, $CaSO_4 \cdot 1/2H_2O$, $CaSO_4 \cdot 2H_2O$. The main reaction methods are as follows:

$$Ca_5[PO_4]_3F + 5H_2SO_4 + 5nH_2O \rightarrow 3H_2PO_4 + CaSO_4 \cdot nH_2O + HF$$

It contains water-soluble phosphorus, fluorine and other trace elements, insoluble organic matter, radioactive substances and other impurities [2,3]. The utilization rate of phosphogypsum resources in the world is low, and most countries adopt the rough storage method of mine backfill. With the shortage of land resources, the problem of atmospheric and solid waste pollution caused by the stockpiling of phosphogypsum mines has become more and more acute. Accelerating the research and development of phosphogypsum in the agricultural, chemical, and building materials industries has gradually become an urgent issue.

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The main chemical composition of phosphogypsum is the same as that of natural gypsum, which is $CaSO_4.2H_2O$. The impurities contained in phosphogypsum have a serious impact on the quality of the prepared gypsum products [4]. By removing impurities and adjusting additives, using phosphogypsum as a substitute for natural gypsum to prepare gypsum mortar can not only effectively alleviate the shortage of natural gypsum supply, but also absorb phosphorus. Gypsum, to solve the environmental problems caused by phosphogypsum [5].

2. Experimental Protocol Design

The experiment is divided into three parts as a whole [6,7]: 1) Analysis of the chemical composition and mechanical properties of raw materials. 2) Comparison and selection of impurity treatment schemes by water washing method, flotation method, and lime neutralization method. The washing times of the water washing method are compared with the PH value of different washing times and the effect of impurity removal to determine the washing scheme, and then the impurities P, F, and organic matter of the three schemes are compared. 3) Proportion by weight: phosphogypsum 30-36%, cement 8-11%, fine aggregate 12-15%, boron mud 4-7%%, fly ash 15-18%, and hemp knife 3-6% in different proportions, designed 16 experimental groups with different dosages to compare and analyze the mechanical properties of mortar to determine the optimal combination. In four groups A, B, C, D, the adjustment of the dosage of phosphogypsum and hemp knives was used to study the effects of different dosages of phosphogypsum on the mechanical properties of mortar and the effect of hemp knives on the crack resistance; group A mainly studied the effect of different dosages of cement on the mechanical properties of mortar The influence of performance; Group B studies the influence of different dosages of fine aggregate on the mechanical properties of mortar; Group D studies the effect of different dosages of boron mud on the mechanical properties of mortar; Group D studies the effects of different dosages of fly ash on the mechanical properties of mortar; as show in table 1 for the experimental weight ratio of each group.

Table 1. Scale of weight.

Group No	Phosphogypsum %	Cement %	Fine aggregate %	Boron mud %	Fly ash %	Hemp knife %
A1	30	8	12	4	15	3
A2	30	9	12	4	15	3
A3	31	10	14	5	17	3
A4	31	11	14	5	17	3
B1	32	8	12	4	15	4
B2	32	8	13	4	15	4
B3	33	10	14	5	17	4
B4	33	10	15	5	17	4
C1	34	8	12	4	15	5
C2	34	8	12	5	15	5
C3	35	10	14	6	17	5
C4	35	10	14	7	17	5
D1	36	8	12	4	15	6
D2	36	8	12	4	16	6
D3	37	10	14	5	17	6
D4	37	10	14	5	18	6

3. Raw Material Analysis

The raw material of phosphogypsum used in this experiment was obtained from the subsidiary products produced by Yunnan Chemical Industry Research Institute Co., Ltd. The chemical composition analysis is shown in table 1, the physical properties of raw materials as show in table 2, the internal illumination index I Ra is 0.45, and the external illumination index I r is 0.48.

Table 2. Analysis of the chemical composition of the phosphogypsum plant.

Element	P_2O_5	F^-	Cl^-	Cao	MgO	SO_3	Fe_2O_3	Al_2O_3	water content	PH value
Content %	0.94	0.1	0.022	31.84	0.32	40.30	0.055	0.062	6.48	3.58

4. Raw Material Processing Plan

4.1. Water Washing Method

Phosphogypsum powder 3:1 by volume, forced to stir for 4-6 minutes to fully dissolve, then let stand for 30 minutes, discard the upper layer solution, and dry the lower layer of solid [8]. The content of impurities before and after washing as show in table 3 show in figure 1, figure 2.

Table 3. Impurity content before and after water washing.

Serial number	index	before processing	Wash times				
			1	2	3	4	5
1	P	0.94	0.85	0.74	0.52	0.50	0.48
2	F	0.10	0.06	0.02	0	0	0
3	organic matter	0.35	0.30	0.15	0	0	0
4	PH value	3.58	3.85	4.75	6.08	6.28	6.35

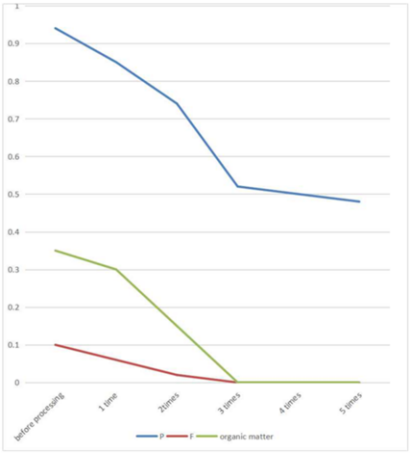

Figure 1. Impurity removal curves before and after washing and times of washing.

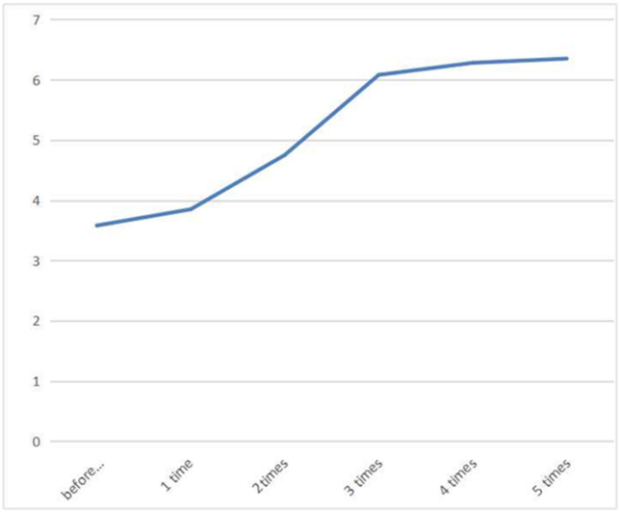

Figure 2. PH value curves before and after washing and times of washing.

4.2. Flotation Method

Use the buoyancy of water to float the impurities on the water surface, and use the scraper to skim off the impurities and organic matter on the surface. This method has a good effect on removing organic matter, but the removal effect of soluble P and F in water is general [9].

4.3. Lime Neutralization Method

This method uses the principle of chemical neutralization reaction between lime and P, F and other impurities to generate phosphoric acid and fluoride inert materials. The main reactions are as follows:

$$P_2O_5 + 3Ca(OH)_2 \rightarrow Ca(PO_4)_2 + 3H_2O$$

$$2F^- + Ca(OH)_2 \rightarrow CaF_2 + 2OH^-$$

Show in table 4, show in figure 3, figure 4 for the impurity content after three treatment schemes.

Table 4. Impurity content before and after the three treatment schemes.

Serial number	index	before processing	washing method	Flotation	neutralization law
1	P	0.94	0.52	0.92	0.51
2	F	0.10	0	0.09	0.01
3	organic matter	0.35	0	0.05	0.35
4	PH value	3.58	6.08	4.12	5.86

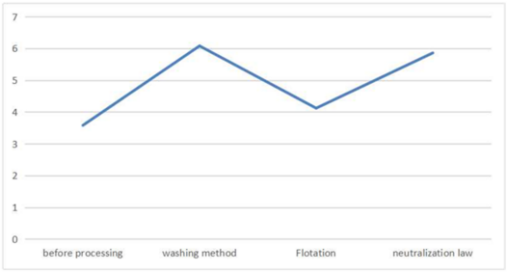

Figure 3. Impurity removal curves before treatment and different treatment methods

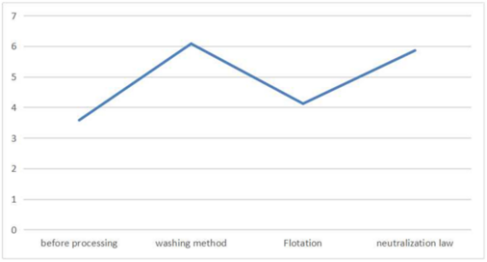

Figure 4. PH value curves before treatment and different treatment methods

5. Experimental Scheme of Mortar with Different Content

The mass ratio table is configured with pre-mixed phosphogypsum mortar, each group is configured with 30kg, and it is fully stirred and sealed in bags for later use. According to the mortar consistency of 60-80mm, add water and stir thoroughly for 3-4 minutes, and make flexural, compressive and adhesive tensile test pieces (cubes with a size of 70.7 ×70.7 ×70.7mm) and put them into the standard curing room for curing and testing respectively Setting time, 7d, 14d, 28d mechanical properties [10]. The test results as shown in table 5, figures 5,6,7,8, statistical table of experimental results of phosphogypsum groups.

Table 5. Statistics of the experimental results of each group of phosphogypsum.

Group No	Coagulation time /min		7d Strength /Mpa			14d strength /Mpa			28d strength /Mpa		
	initial setting	final set	anti-stress	Bending resistance	bonding	anti-stress	Bending resistance	bonding	anti-stress	Bending resistance	bonding
A1	55	165	2.5	1.0	0.09	8.8	2.4	0.30	10.8	3.4	0.50
A2	68	190	2.4	0.9	0.08	8.9	2.6	0.31	10.5	3.3	0.51
A3	80	205	3.0	1.1	0.10	9.3	2.9	0.32	11.0	3.5	0.52
A4	82	210	3.1	1.2	0.11	9.5	3.0	0.33	11.1	3.6	0.52
B1	57	155	2.4	0.8	0.13	8.9	2.9	0.34	10.9	3.1	0.53
B2	64	179	2.6	0.9	0.13	9.1	3.1	0.33	11.1	3.4	0.53
B3	79	199	3.2	1.2	0.15	9.4	3.2	0.35	11.3	3.4	0.53
B4	81	205	3.0	1.1	0.15	9.2	3.0	0.35	10.8	3.2	0.54
C1	53	158	2.5	0.9	0.15	9.1	2.6	0.37	10.9	3.2	0.56
C2	66	188	2.7	1.0	0.16	8.5	2.3	0.38	10.8	3.3	0.57
C3	81	195	3.3	1.3	0.15	9.7	3.1	0.39	11.1	3.7	0.55
C4	83	197	3.2	1.2	0.14	9.5	3.0	0.37	11.2	3.6	0.54
D1	54	160	2.6	0.8	0.12	9.0	2.6	0.35	11.0	3.1	0.52
D2	67	191	2.7	0.9	0.11	9.2	2.3	0.34	10.8	3.0	0.51
D3	81	198	3.4	1.2	0.10	9.4	2.9	0.33	11.1	3.5	0.50
D4	85	204	3.3	1.2	0.11	9.5	3.1	0.33	11.1	3.4	0.50

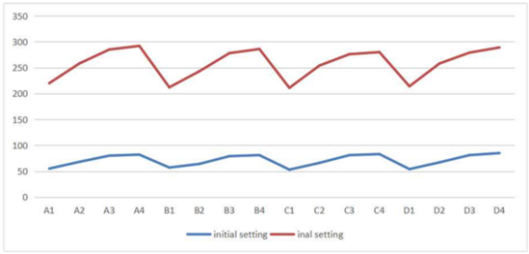

Figure 5. Curves of coagulation time for different mix ratios.

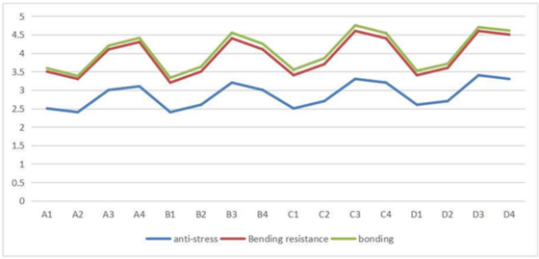

Figure 6. 7d strength curves for different mix ratios.

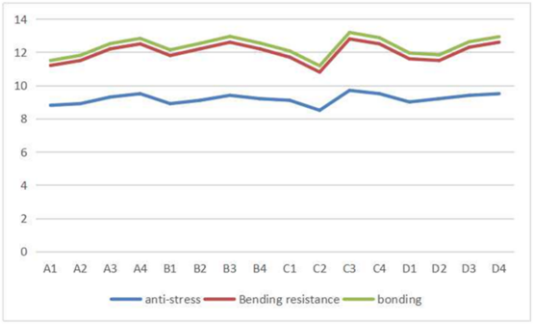

Figure 7. 14d strength curves for different mix ratios.

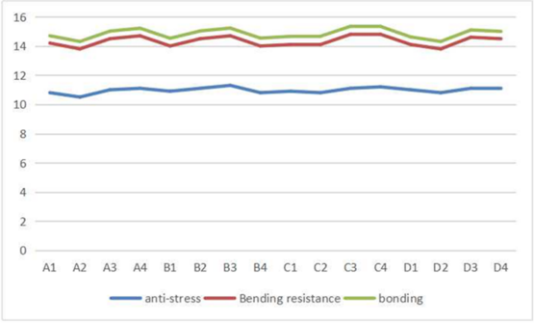

Figure 8. 28d strength curves for different mix ratios.

Fly ash can delay the setting of phosphogypsum cement mortar to a certain extent, the content changes obviously from 15% to 16%, and the values of 16%, 17% and 18% do not change much. When the content of phosphogypsum increases step by step from 30% to 37% and 3 % to 6% of hemp knife, the crack resistance of mortar shows a trend of first increasing and then decreasing. Best crack resistance. When the fine aggregate

is increased from 12% to 15% and boron mud from 4% to 6% step by step, the compressive and flexural properties of mortar show a trend of rising first and then falling. The amount of fine aggregate is 34% and boron mud is 5%. The combination of compression and flexural performance is the best. The compressive and flexural strength of the standard maintenance 7d reaches about 30% of the design strength, the 14d compressive and flexural strength reaches about 80% of the design strength, and the 28d compressive and flexural strength reaches the design strength about 100% of the design strength.

6. Conclusion

In this paper, through the comparison and selection of impurity removal schemes for phosphogypsum raw materials, it can be concluded that the 3 -pass water separation method has a good effect on removing P and F , the residual organic matter is almost 0 , and the pH is greater than 6.0 , which meets the relevant requirements for making mortar raw materials. On this basis, by adjusting the amount of phosphogypsum, cement, aggregate, boron mud, fly ash, and hemp knife, 4 groups and 16 groups were set up to conduct experiments to study the properties of phosphogypsum mortar in different combinations. According to the mass ratio of 35% phosphogypsum , 10% cement, 14% aggregate, 5% boron mud, 16% fly ash, 5% hemp knife , and other 15%, the initial setting time of the phosphogypsum mortar prepared is about 80min, and the final setting time is about 80 minutes . About 180minutes, the 28d compressive strength can reach 11Mpa, the flexural strength is about 3.5 Mpa, and the bonding strength is about 0.55 Mpa , all of which are higher than the specification requirements.

The phosphogypsum mortar produced according to this ratio is used in wall plastering projects. The product has stable performance, good construction molding, strong operability, and good economic and social benefits.

References

[1] Peng JH. Wan TZ. Tang L. Composition, shape, distribution of impurities in phosphogypsum and their effects on performance. China Building Materials Science and Technology. 2020.
[2] Gong XQ. Liu SJ. Study on the influence of lime-modified phosphogypsum on the performance of cement mortar. Journal of Wuhan University of Light Industry. 2019
[3] Gong XQ. Liu SJ. Research on the influence of basic performance of phosphogypsum cement mortar. Journal of Wuhan University of Light Industry. 2018.
[4] Wang Q, Jia R. A novel gypsum-based self-leveling mortar produced by phosphorus building gypsum(pbg)-based mortar. Therm. Anal. Calorim. 2019.
[5] Yang L, Zhang Y, Yan Y. Utilization of original phosphogypsum as raw material for the preparation of self-leveling mortar. J. Clean Prod. 2016.
[6] Xi MY. Comprehensive utilization of phosphogypsum. Environmental Science and Technology. 2001.
[7] Lu SW. Research and application of phosphogypsum-based self-leveling materials. Wuhan University of Technology. 2014.
[8] Lin RT. Development and performance research of lightweight plastering gypsum mortar. Fujian Building Materials. 2018.
[9] Ma BG. Sun ZD, Su Y. Research on setting time and strength control of phosphorus building plaster. Silicate Bulletin. 2019.
[10] Chen QH, Jiang ZW. Effect of chemical pretreatment on properties of phosphogypsum-based double-check cementitious materials. Journal of Building Materials. 2020.