Research Article

Effect of Soluble P_2O_5 Form on the Hydration and Hardening of Hemihydrate Phosphogypsum

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The preparation of building gypsum from phosphogypsum is one of the ways to realize its resource utilization. However, the precondition is first to reveal the influence mechanism of the impurities in phosphogypsum on the properties of plaster. Thus, the influence of the form of soluble P_2O_5 (H_3PO_4 , $H_2PO_4^-$, and HPO_4^{2-}) that is considered the most significant impurity on the hydration and hardening of hemihydrate phosphogypsum was studied in this paper, which included the setting time, mechanical properties, hydration rate, crystal structure, and micromorphology of hydration products. The results showed that both H_3PO_4 and $H_2PO_4^-$ can accelerate the early hydration of hemihydrate phosphogypsum, shorten the setting time of paste, and improve the mechanical properties of hardened paste. In addition, H_3PO_4 had a more significant effect than $H_2PO_4^-$. Different from H_3PO_4 and $H_2PO_4^-$, HPO_4^{2-} significantly delayed the early hydration reaction of hemihydrate phosphogypsum, significantly prolonged the setting time of the paste, and significantly reduced the mechanical properties of the hardened body. The test data of XRD, IR, and SEM showed that two negative effects can be caused when soluble P_2O_5 existed in the form of HPO_4^{2-} . On the one hand, HPO_4^{2-} can lead to the formation of $Ca_3(PO_4)_2$ insoluble substance in the hydration reaction of hemihydrate phosphogypsum, which significantly hindered the hydration reaction and prolonged the setting time of the paste. On the other hand, HPO_4^{2-} can change the crystallization habit of the hydration product of $CaSO_4 \cdot 2H_2O$ and make it change from needle-columnar to semiflake and irregular shape, resulting in a significant reduction in the mechanical properties of hardened paste.

1. Introduction

Phosphogypsum is an industrial by-product produced in the process of wet production of phosphoric acid [1, 2]. According to statistics, 4–6 tons of phosphogypsum can be produced for each ton of phosphoric acid [3]. Currently, global emissions of phosphogypsum have exceeded 6 billion tons and are growing at about 150 million tons per year, according to relevant data. The annual output of phosphogypsum in China's phosphoric acid industry has exceeded 70 million tons, but the utilization ratio is only 40% according to China Phosphate and Compound Fertilizer Industry Association. The stock of phosphogypsum stockpiles has reached 5 hundred million tons in China at present, which not only occupies a lot of land resources but also causes serious environmental pollution to air, groundwater, and soil.

Similar to natural gypsum, the main chemical component of phosphogypsum is $CaSO_4 \cdot 2H_2O$, which is usually over 90 wt. %. The production of building gypsum is one of the main ways of resource utilization of phosphogypsum [4–7]. However, the application performance of phosphogypsum is often significantly inferior to that of natural gypsum due to harmful impurities such as P, F, and organic matter [8–13]. Therefore, to explore the influence of harmful impurities in phosphogypsum in order to implement effective impurity removal and purification is always a hot topic in the field of phosphogypsum research but is also one of the key issues in the efficient utilization of phosphogypsum resources.

Many studies have showed that phosphorus impurities have the greatest impact on the properties of hemihydrate phosphogypsum, among many types of impurities [14–16]. Phosphorus in phosphogypsum mainly exists in three forms:

soluble P_2O_5 , eutectic P_2O_5 , and insoluble P_2O_5 , among which soluble P_2O_5 has the most significant effect. Soluble P_2O_5 exists in four forms: H_3PO_4 , HPO_4^{2-} , $H_2PO_4^-$, and PO_4^{3-} , which are produced by the ionization of phosphoric acid in an aqueous solution. Due to the high concentration of Ca^{2+} in gypsum and the low solubility of $Ca_3(PO_4)_2$ as an insoluble salt, the concentration of PO_4^{3-} in the system is low. Therefore, soluble P_2O_5 in phosphogypsum mainly exists in three forms: H_3PO_4 , HPO_4^{2-} , and $H_2PO_4^-$. Many studies have shown that the presence of soluble P_2O_5 can significantly hinder the hydration, condensation, and hardening process of hemihydrate phosphogypsum and reduce the strength of its hardened body [17–20]. The influence mechanism of soluble P_2O_5 is that it can combine with calcium ions in a liquid phase to produce insoluble calcium phosphate and cover the surface of gypsum particles, which hinders the hydration reaction of gypsum [21]. However, previous studies on the influence of soluble P_2O_5 on phosphogypsum performance often do not distinguish the form of soluble P_2O_5 since a single form of soluble P_2O_5 is difficult to separate from phosphogypsum, which is obviously not enough to help people fully understand the internal influence mechanism of soluble P_2O_5 . In addition, most studies on the influence of soluble P_2O_5 on building phosphogypsum mainly focus on macroscopic properties, while there are still few studies on the influence mechanism of soluble P_2O_5 on the hydration rate of hemihydrate phosphogypsum and the crystal structure of hydration products.

Based on the above research deficiencies, this paper intended to study the influence of a soluble P_2O_5 form on the hydration reaction rate, crystal structure, and micromorphology characteristics of hemihydrate phosphogypsum by introducing different forms of soluble P_2O_5 , establish the relationship between the hydration hardening characteristics of hemihydrate phosphogypsum and its physical and mechanical properties under the influence of soluble P_2O_5 form, and reveal the internal influence mechanism of soluble P_2O_5 .

2. Materials and Test Methods

2.1. Materials. Phosphogypsum was purchased from Henan Qiangnai New Material Co., Ltd. Under laboratory conditions, phosphogypsum was washed with deionized water until neutrality to remove soluble impurities. The chemical composition of phosphogypsum before and after water washing is shown in Table 1. It can be seen that phosphogypsum contained harmful impurities such as phosphorus and fluorine. After full water washing, the total P_2O_5 content in phosphogypsum decreased from 0.78% before water washing to 0.64%, indicating that the soluble P_2O_5 content in phosphogypsum was 0.14%. Phosphoric acid was bought from Yantai Shuangshuang Chemical Co., Ltd, which had a H_3PO_4 concentration of 85 wt. %. The analytical pure reagents of NaH_2PO_4 and Na_2HPO_4 for the experiment were purchased from Tianjin Hengxing Chemical Reagent Co., Ltd. The polycarboxylic acid water reducer (PCE) was purchased from Shandong bock Chemical Co., Ltd. The deionized water was always used in the experiment.

2.2. Test Methods

2.2.1. Preparation of Hemihydrate Phosphogypsum. First, the washed phosphogypsum was dried at 40°C and then calcined at 150°C for 12 h. Finally, the experimental hemihydrate phosphogypsum can be obtained after 7 d aging treatment of the calcined powder. According to the Chinese Standard GB/T9776-2008, the water consumption for a standard consistency of the hemihydrate phosphogypsum powder and its change with the content of PCE were determined, as shown in Table 2. The standard consistency of the experimental phosphogypsum is up to 97% water. The addition of PCE can effectively reduce the standard consistency of hemihydrate phosphogypsum. The water consumption for standard consistency of phosphogypsum is significantly reduced to 71% when the optimum PCE content is 0.8%.

2.2.2. Setting Time and Mechanical Property Test. First, in order to investigate the influence of the soluble P_2O_5 form on the setting time and mechanical properties of hemihydrate phosphogypsum, the content of H_3PO_4 , NaH_2PO_4 , and Na_2HPO_4 was determined to be 0.23%, 0.24%, and 0.28%, respectively, according to the content of soluble P_2O_5 in phosphogypsum before and after washing. Second, each form of the soluble P_2O_5 reagent was weighed and pre-dissolved to the mixing water measured by 71 wt. % of the standard water consumption for consistency at the optimal dosage of PCE. Then, the hemihydrate phosphogypsum was added to the mixing water and stirred to make a uniform paste. The initial setting time and final setting time of paste were measured by the vicar apparatus. The prepared paste was molded into blocks of $40\text{ mm} \times 40\text{ mm} \times 160\text{ mm}$. After 1 d age, the wet flexural strength and wet compressive strength of each hardened paste were tested. At the same time, parts of the formed samples were dried in an oven at 40°C until they were completely dried, which was used for the test of dry flexural and dry compressive strength of the hardened paste. The samples with H_3PO_4 , NaH_2PO_4 , and Na_2HPO_4 were named P- H_3PO_4 , P- $H_2PO_4^-$, and P- HPO_4^{2-} , respectively. The sample without the soluble P_2O_5 reagent was marked as control.

2.2.3. Hydration Degree Test. According to the method described in Section 2.2.2, each sample pastes were prepared. After reaching the specified age, the hydration was stopped immediately with anhydrous ethanol for 24 h, and then the samples were dried in an oven at 40°C . Finally, the dried samples were placed in an oven at 250°C for low-temperature heat treatment to constant weight. According to the mass loss of the sample before and after heat treatment, the content of crystal water was calculated to characterize the hydration degree of the sample.

2.2.4. XRD, IR, and SEM Tests. After the dry strength test, the fractured sample was used for XRD, IR, and SEM characterization. The phase composition and structure of

TABLE 1: Chemical composition of phosphogypsum before and after water washing.

Condition	CaO	Fe ₂ O ₃	Al ₂ O ₃	F	SO ₃	SiO ₂	P ₂ O ₅	R ₂ O	MgO
Before washing	38.28	0.58	1.50	0.35	48.25	8.75	0.78	0.95	0.19
After washing	39.49	0.23	1.36	0.345	49.12	7.51	0.64	0.84	0.14

TABLE 2: Test results of water consumption for standard consistency.

PCE dosage (%)	0	0.2	0.4	0.6	0.8	1.0
Water for standard consistency (wt%)	97	82	74	72	71	71

hydration product crystals of each sample were tested by a Bruker D8 Advance X-ray diffractometer. The test range was 5–50° at a scanning rate of 0.02°/min. The infrared spectrum data of each hydration sample were collected by the Fourier infrared absorption spectrometer with the model of Nicolet SDX CFTIR. After the fractured samples were coated with gold, the SEM images were captured on a Quanta-450 SEM at 15 kV.

3. Results and Discussion

3.1. Setting Time. Figure 1 shows the effect of the soluble P₂O₅ form on the setting time of building phosphogypsum. For the control sample, the initial setting time and final setting time were 11 min and 17 min, respectively. The addition of H₃PO₄ can significantly accelerate the setting and hardening rate of the paste, and the initial setting time and final setting time were shortened to 7 min and 12 min, respectively. Similar to H₃PO₄, the addition of H₂PO₄[−] can also promote the setting and hardening of the paste, but its effect is less than that of H₃PO₄. Different from the effect of soluble P₂O₅ in the form of H₃PO₄ and H₂PO₄[−], the addition of HPO₄^{2−} significantly delayed the setting and hardening rate of paste. The test data showed that the initial setting time and final setting time of P – HPO₄^{2−} sample were significantly prolonged to 21 min and 27 min, respectively, compared with the control sample.

3.2. Mechanical Properties. Figure 2 shows the effect of different forms of soluble P₂O₅ on the compressive strength of hemihydrate phosphogypsum. For the control sample, the wet compressive strength and dry compressive strength of hardened pastes were 3.8 MPa and 6.8 MPa, respectively. After the addition of H₃PO₄, the wet compressive strength and dry compressive strength of hardened paste increased to 5.3 MPa and 9.5 MPa, respectively, which increased by 39.5% and 39.7% compared with the control sample, respectively. The addition of H₂PO₄[−] also can improve the compressive strength of hemihydrate phosphogypsum, but the effect was not as significant as H₃PO₄. The wet compressive strength and dry compressive strength of the P – H₂PO₄[−] sample increased by 28.9% and 25%, respectively, compared with the control sample. Different from H₃PO₄ and H₂PO₄[−], the addition of HPO₄^{2−} significantly reduced the strength of hemihydrate phosphogypsum. The data showed that the wet compressive strength and dry compressive strength of the

P – HPO₄^{2−} sample were only 3.6 MPa and 3.1 MPa, respectively, which were reduced by 5.3% and 20.5%, respectively, compared with the control sample. Figure 3 shows the flexural strength test results. It can be seen that the variation law of flexural strength and compressive strength was similar. Similarly, the addition of H₃PO₄ and H₂PO₄[−] can improve the wet and dry flexural strength of hemihydrate phosphogypsum, and the effect of H₃PO₄ is more significant than H₂PO₄[−]. The addition of HPO₄^{2−} can significantly reduce the flexural strength of the hardened paste.

3.3. Hydration Degree. The effect of different forms of P₂O₅ on the crystalline water content of hemihydrate phosphogypsum is shown in Figure 4. It can be seen that, in the initial hydration stage, the form soluble P₂O₅ of H₃PO₄ and H₂PO₄[−] can effectively accelerate the hydration reaction of hemihydrate phosphogypsum, and the role of H₃PO₄ in promoting hydration is significantly higher than that of H₂PO₄[−]. Different from H₃PO₄ and H₂PO₄[−], soluble P₂O₅ in the form of HPO₄^{2−} can significantly delay the early hydration reaction of the hemihydrate phosphogypsum. For example, after hydration for 30 min, the content of crystal water in P – H₃PO₄ and P – H₂PO₄[−] samples was 18.1% and 16.4%, respectively, which was significantly higher than of the control sample. However, the crystalline water content of P – HPO₄^{2−} sample was only 12.3% at this time, which was significantly lower than that of the control sample. After 24 h hydration, the crystal water content of each sample was similar, which was close to the theoretical crystal water content of dihydrate gypsum crystals, indicating that the hemihydrate gypsum in each sample has been completely hydrated. By comparing the data in Figures 2 and 5, it can be seen that the effect of soluble P₂O₅ form on the hydration rate of hemihydrate phosphogypsum was consistent with its effect on the setting time.

3.4. XRD Analysis. Figure 5 shows the XRD spectrum of each hydration sample. It can be seen that the hydration products of each 9 are calcium sulfate dihydrate crystals. Moreover, the peak shape of the diffraction peak in each sample was very sharp, which indicated that the crystal crystallization degree of the hydration products of dihydrate gypsum in each sample was high. The difference was that the peak intensity of the three strong peaks of dihydrate gypsum crystal in the P – H₃PO₄ sample was significantly lower than that of the control, P – H₂PO₄[−], and P – HPO₄^{2−} samples. Generally speaking, the intensity of the crystal diffraction peak was directly proportional to the number of crystals and crystallinity. According to the test results of the hydration rate, the amount of the dihydrate gypsum crystal in each

FIGURE 1: Influence of soluble P_2O_5 form on setting time of hemihydrate phosphogypsum.

FIGURE 2: Influence of soluble P_2O_5 form on the compressive strength of hemihydrate phosphogypsum.

sample was the same after 1 d hydration. Therefore, it can be inferred that the existence of soluble P_2O_5 may affect the hydration crystallization process of hemihydrate phosphogypsum and then significantly change the crystallization degree of the crystal surface corresponding to the three strong peaks in dihydrate gypsum crystals.

In order to further explain the influence of soluble P_2O_5 , the (020) crystal plane diffraction peak was taken as the research object for in-depth analysis and description, as shown in Figure 6. It can be seen that the diffraction angles of the crystal plane diffraction peaks of each sample (020) were different. Through the peak seeking function of jade analysis software, the diffraction angles of (020) crystal plane of the control sample, P-H₃PO₄, P-H₂PO₄⁻, and P-HPO₄²⁻ can be determined to be 11.63°, 11.585°, 11.643°, and 11.679°, respectively. This phenomenon may be caused by the

formation of CaHPO₄·2H₂O solid solution in a dihydrate gypsum crystal due to the existence of soluble P_2O_5 . Many studies showed that CaHPO₄·2H₂O and CaSO₄·2H₂O belong to a monoclinic system and have relatively similar lattice constants. Therefore, CaHPO₄·2H₂O can enter the CaSO₄·2H₂O lattice to form a solid solution under certain conditions. In order to facilitate the analysis, the diffraction angles of the diffraction peaks of the (020) crystal plane of pure CaSO₄·2H₂O and pure CaHPO₄·2H₂O are calibrated in Figure 6, which were 11.588° and 11.68°, respectively. For the control sample, the diffraction angle of (020) crystal plane was between CaSO₄·2H₂O and CaHPO₄·2H₂O crystals. It can be seen that there was a solid solution of CaHPO₄·2H₂O in the hydration process of the control sample. The source of CaHPO₄·2H₂O in the control sample was mainly related to its eutectic phosphorus. Relevant research studies showed that the eutectic P_2O_5 still existed when phosphogypsum was calcined at 150°C. Therefore, when hemihydrate phosphogypsum reacted with water, part of HPO₄²⁻ dissolved from its lattice may reparticipate in the crystallization process of dihydrate gypsum. For the P-H₃PO₄ sample, the diffraction angle of the (020) crystal plane was very close to that of pure CaSO₄·2H₂O. This may be due to the fact that H₃PO₄ was difficult to deeply ionize and produce more HPO₄²⁻ ions in the reaction liquid phase as a weak electrolyte, which resulted in the crystal surface diffraction peak site of P-H₃PO₄ being closer to pure CaSO₄·2H₂O. For P-H₂PO₄⁻, the shift of diffraction peak position to the right may also be related to the significant ionization of H₂PO₄⁻ ions. For P-HPO₄²⁻, the directly doped HPO₄²⁻ ions can significantly participate in the formation of calcium sulfate dihydrate, resulting in the crystal diffraction peak position more inclined to the direction of CaHPO₄·2H₂O.

Accordingly, the eutectic phenomenon in the control sample, P-H₂PO₄⁻, and P-HPO₄²⁻ significantly inhibited the growth of gypsum crystals along the c-axis and relatively increased the growth rate and crystallinity of (020) crystal planes, which resulted in the diffraction peak intensity of (020) crystal planes in these three groups being significantly higher than that of P-H₃PO₄.

3.5. IR Analysis. In order to further analyze the influence mechanism of the form of soluble P_2O_5 , the infrared spectrum data of each hydration sample were tested, as shown in Figure 7. The stretching vibration peaks of crystalline water hydroxyl in calcium sulfate dihydrate were detected near 3547 cm⁻¹ and 3045 cm⁻¹. The peak near 1685 cm⁻¹ and 1621 cm⁻¹ was the bending vibration peak of S-O in CaSO₄. For the sample of control, P-H₂PO₄⁻, and P-HPO₄²⁻, the characteristic absorption peak of eutectic phosphorus was also detected near 845 cm⁻¹, which was consistent with the analysis results of XRD. In addition, the characteristic absorption peak of PO₄³⁻ was also detected near 1036 cm⁻¹ in control and P-HPO₄²⁻ sample, indicating that insoluble Ca₃(PO₄)₂ was generated in the hydration process of these two groups of samples. The reason why the control sample and P-HPO₄²⁻ sample had a longer setting time than other group may be related to this phenomenon.

FIGURE 3: Influence of soluble P_2O_5 form on the flexural strength of hemihydrate phosphogypsum.

FIGURE 4: Influence of soluble P_2O_5 form on the content of water of crystallization of hemihydrate phosphogypsum at different hydration times.

Many studies have proved that the formation of $Ca_3(PO_4)_2$ can effectively cover the surface of hemihydrate gypsum crystal and delay its hydration process [21, 22].

3.6. Scanning Electron Microscopy. Figure 8 shows the microtopography photograph of each sample. For the control sample, the hydrated dihydrate gypsum crystal mainly showed a coarse columnar structure, and a part of the form of the product was irregular plate. In addition, there was an obvious parallel superposition between the columnar

crystal products. Obviously different from the control sample, the dihydrate gypsum crystal formed in the P-H₃PO₄ sample was significantly needle columnar, and each crystal showed a high degree of overlapping and interleaving, which reflected relatively higher strength of the hardened body. For the P-H₂PO₄⁻ sample, the morphology of hydration products was mainly a needle column and an irregular shape. The crystal morphology of hydration products in the P-HPO₄²⁻ sample was mostly plate-like or irregular, and only a few crystals were columnar. These morphological characteristics significantly affect the crystal

FIGURE 5: XRD spectra of each sample.

FIGURE 6: X-ray diffraction peak of the (020) crystal plane of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in each sample.

FIGURE 7: Infrared spectra of each sample.

FIGURE 8: Microscopic morphologies of each sample: (a) control; (b) P- H_3PO_4 ; (c) P- H_2PO_4^- ; (d) P- HPO_4^{2-} .

skeleton effect of dihydrate gypsum in the P- HPO_4^{2-} sample, resulting in significantly poor mechanical properties of its hardened body.

4. Conclusions

This paper mainly studies the influence of soluble P in three forms of H_3PO_4 , H_2PO_4^- , and HPO_4^{2-} on the hydration and hardening of hemihydrate phosphogypsum. The main conclusions were as follows:

- (1) Soluble P_2O_5 in the form of H_3PO_4 and H_2PO_4^- can effectively accelerate the early hydration reaction of hemihydrate phosphogypsum, and the influence of H_3PO_4 was greater than that of H_2PO_4^- . Accordingly, the setting time of paste with soluble P_2O_5 in the form of H_3PO_4 and H_2PO_4^- was significantly shortened, and similarly, the effect of H_3PO_4 was more significant than that of H_2PO_4^- . Different from H_3PO_4 and H_2PO_4^- , HPO_4^{2-} significantly hindered the early hydration reaction and prolonged the setting time of hemihydrate phosphogypsum.
- (2) The test results of mechanical properties showed that when the soluble P_2O_5 in phosphogypsum mainly existed in the form of H_3PO_4 and H_2PO_4^- , the mechanical properties of building phosphogypsum can

be improved to a certain extent, and the enhancement effect of H_3PO_4 on the mechanical properties of hardened paste was higher than that of H_2PO_4^- . On the contrary, soluble P_2O_5 with HPO_4^{2-} as the main form had a significant negative effect on the mechanical properties of hemihydrate phosphogypsum.

- (3) The test results of XRD, IR, and SEM comprehensively reflected that two negative effects can be caused when the soluble P_2O_5 in phosphogypsum exists in the form of HPO_4^{2-} . On the one hand, HPO_4^{2-} can lead to the formation of $\text{Ca}_3(\text{PO}_4)_2$ insoluble substance in the hydration reaction of hemihydrate phosphogypsum, which significantly hindered the hydration reaction and prolonged the setting time of the paste. On the other hand, HPO_4^{2-} can change the crystallization habit of the hydration product of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and make it change from needle-columnar to semiflake and irregular shape, resulting in a significant reduction in the mechanical properties of the hardened paste.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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