

## Research Article

# Effect of Ball Milling Time on the Performance of Phosphorous Building Gypsum

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The use of phosphogypsum to prepare phosphorus building gypsum (PBG) is of great value to the resource utilization of phosphogypsum. In this study, PBG was ball-milled to obtain phosphorus building gypsum with good performance, which can meet the requirements of the Chinese standards for first-class building gypsum. Meanwhile, the changes of net slurry physical properties, mechanical properties, and particle size parameters of PBG under different treatment times were analyzed. With the increase of ball milling time, the particle size of PBG decreased rapidly and then stabilized, and the specific surface area gradually increased and then started to rise back. Ball milling can significantly reduce the standard consistency water requirement of phosphogypsum, resulting in a shorter setting time and higher strength of phosphogypsum. In the fixed water consumption test, the effect of ball milling time on the performance of phosphogypsum was small. Compared with sieving, washing, aging, and other means of PBG treatment, ball milling has the advantages of simplicity, environmental protection, and low cost, and it has some practical significance in production.

## 1. Introduction

Phosphogypsum is a byproduct of the industrial production of phosphoric acid, which is mainly composed of calcium sulfate dihydrate and contains phosphorus, fluorine, organic matter, and other impurities [1]. Resourcefulness of phosphogypsum poses a global environmental problem [2]. There are considerable stocks of phosphogypsum in China, and its production increases every year. In 2020, ~76 million tons of phosphogypsum were generated, but only ~40% were utilized [3]. The production of PBG from phosphogypsum is one of the primary ways to consume phosphogypsum [4, 5]. The performance of PBG is much worse than natural building gypsum because the composition of PBG is more complex [6]. Moreover, the particle morphology, particle size distribution, and crystal morphology of PBG are different from those of natural building gypsum [7, 8].

At present, certain pretreatment methods are required for making building gypsum through phosphogypsum, such

as chemical methods [9], water washing methods [10], flotation methods [11], sieve methods [12], and thermal treatment methods [13, 14]. As a common powder pretreatment method, the ball grinding method has been widely applied in the preparation or modification of cement [15–17]. However, chemical ball milling is a secondary method when chemical and water washing methods are used for phosphogypsum or building gypsum. Li [18] studied the impact of ball milling time on the specific area/particle size distribution/water consumption of normal consistency/strength at 2h and strength at dry condition of building gypsum. The results showed that the best performance of building gypsum was obtained when the ball milling time was 3 min. However, if milling time continues to increase, the performance of the building gypsum cannot be improved. Xiong [19] obtained building gypsum by roasting and aging milled phosphogypsum. The experiment results showed that building gypsum had the best performance when ball milling time was 20 min and the particle size was

25.5  $\mu\text{m}$ , but a long ball milling time was detrimental for the performance of building gypsum.

Previous studies have focused on the performance variation of building gypsum in normal consistency tests. In this study, the performance of building gypsum by milling method was investigated using the theory of mechanical force chemistry [20, 21]. The focus of this study was to compare the effects of milling time on the performance of building gypsum, the consistency of physical and mechanical properties regarding water consumption of normal consistency, and the effect of fixed water consumption. Meanwhile, the effect of ball milling on the physical and mechanical properties of building gypsum was investigated. Finally, according to the Chinese Standard GB/T 9775-2008, the first-class quality of building gypsum was achieved in this study.

## 2. Materials and Methods

**2.1. Materials.** Phosphogypsum was from the phosphogypsum storage yard of Yunnan Yuntianhua Co., Ltd. It was a light-yellow powder, and its chemical composition is shown in Table 1. Dehydration of phosphogypsum was performed in a constant-temperature blast oven at 160°C for 4 h to prepare PBG.

**2.2. Test Procedures.** The prepared PBG was milled using a small horizontal ball mill. Two kilograms of the material were loaded into the ball mill each time. The ratio of PBG to balls was 1 : 1, and the samples were then grouped according to the duration of the ball milling process (10, 20, 30, 40, 50, 60, and 70 min), as shown in Table 2.

Firstly, changes in the powder state of samples in each group were analyzed, including the distribution of particle size, specific surface area, and particle micromorphology. Secondly, the water consumption of normal consistency of each group of samples was tested, as well as the setting time and the flexural and compressive strength at 2 h at the water consumption of normal consistency. Thirdly, suitable water consumption was selected. The slurry flowability, setting time, and flexural and compressive strength at 2 h were tested for each group of samples at fixed water consumption. The test procedures are depicted in Figure 1.

### 2.3. Testing Methods

Physical properties of paste: refer to the Chinese national standard “Gypsum plasters—Determination of physical properties of pure paste” (GB/T 17669.4-1999).

Mechanical properties: refer to the Chinese national standard “Determination of Mechanical Properties of Building Plaster” (GB/T 17669.3-1999).

Particle size distribution and specific surface area: the particle size of the samples was analyzed using Jinan Winner 2005 wet laser particle size analyzer with anhydrous ethanol as the dispersant.

Microscopic morphology of gypsum particles: observation was conducted by NOVA NANO SEM450 field

emission using a scanning electron microscope. The powder samples were gold-sprayed.

## 3. Results and Discussion

**3.1. Effect of Ball Milling Time on the Properties of PBG at Water Consumption of Normal Consistency.** In this paper, the test was first conducted at a fixed flow rate (180 mm) to test the water consumption of normal consistency of samples. The setting time and the flexural and compressive strength at 2 h under the water consumption of normal consistency were then measured.

**3.1.1. Water Consumption of Normal Consistency.** Figure 2 shows the water consumption of normal consistency tests on eight samples of PBG.

As the ball milling time increases, the water consumption of normal consistency decreases to a minimum value and then increases. When the ball milling time is <50 min, the water consumption of normal consistency decreases with the increased ball milling time (from 76.4% to 60.07%). The water consumption of standard consistency reaches a minimum value of 60.07%, which is 21.5% lower than PBG without the ball milling. When the ball milling time exceeds 50 min, the water consumption of normal consistency increases with an additional increase in ball milling time.

**3.1.2. Setting Time.** Figure 3 shows the setting time test results for the eight PBG samples at water consumption of normal consistency.

With the increase of ball milling time, the initial setting time of the samples decreases and then increases. When the ball milling time is <30 min, the initial setting time significantly reduces with the increased ball milling time. When the ball milling time ranges from 30 to 50 min, the initial setting time is almost unchanged. When the time of ball-milling treatment was >50 min, the initial setting time increases with the increase of the ball-milling treatment time. The initial setting time of the PBG reaches a minimum value of 266 s at a ball milling time of 40 min, and it then increases with a further increase in the ball milling time. The minimum value (266 s) represents a decrease of ~34% compared with the 404 s before the ball milling.

With an increase in the ball milling time, the final setting time of PBG continuously decreases. When the ball milling time is <20 min, the final setting time remarkably decreases with an increase in milling time, reaching an overall reduction of 66.5%. When the ball milling time is >20 min, a slower drop is recorded, reaching a minimum value of 472 s at the ball milling time of 70 min. The minimum value is ~47% lower than the value (894 s) before the ball-milling treatment.

**3.1.3. Mechanical Strength.** Figure 4 shows the 2 h strength at water consumption of normal consistency for eight samples of PBG.

TABLE 1: Chemical composition of phosphogypsum.

Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	Organism
Content (%)	14.52	1.66	0.15	0.005	0.17	31.94	0.22	0.94	45.38	0.25

TABLE 2: Experimental design.

No.	P-0	P-1	P-2	P-3	P-4	P-5	P-6	P-7
Milling time (min)	0	10	20	30	40	50	60	70

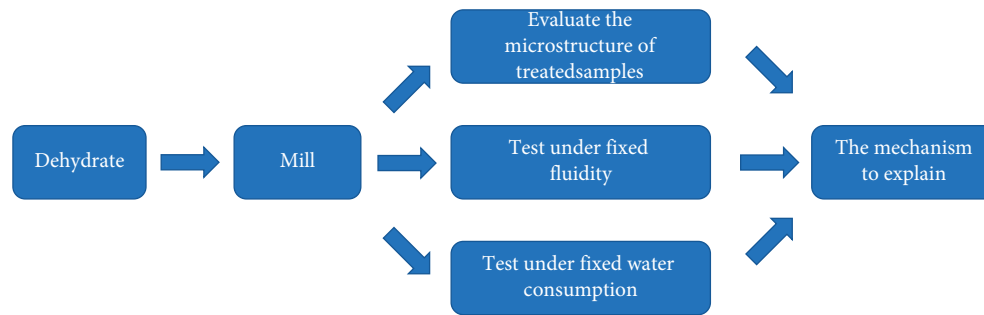


FIGURE 1: Test procedures.

With an increase in ball milling time, the 2 h flexural strength of phosphate gypsum first increases and then decreases (Figure 4(a)). When the ball milling time is <20 min, the 2 h flexural strength significantly increases with the increase of ball milling time. At the ball milling time between 20 and 50 min, the 2 h flexural strength slowly increases. At the ball milling time of 50 min, the strength reaches a maximum value of 3.41 MPa, representing a 49.6% increase compared with gypsum of 2.28 MPa before the ball-milling treatment and a subsequent rapid decrease when the ball milling time is >50 min.

The 2 h compressive strength of PBG is improved and then decreases as the ball milling time increases (Figure 4(b)). The strength first increases gradually when the ball milling time is <40 min. When the ball-milling treatment time is >50 min, the strength rapidly decreases with increased ball milling time. The strength increases by 81% at the ball-milling treatment time of 50 min, and it reached a maximum value of 7.78 MPa, representing an 81.4% increase compared with the value of 4.29 MPa before the ball milling.

**3.2. Effect of Ball-Milling Treatment Time on the Properties of PBG at Fixed Water Consumption.** According to the normal consistency test results, with changes in ball milling time, the setting time and 2 h strength of PBG significantly changed. However, under the water consumption of normal consistency, the water consumption of each test samples widely varied. In order to analyze the effect of the ball-milling treatment, the water consumption of normal consistency (60%) of the test samples milled for 50 min was selected as the fixed water consumption. The test was conducted under fixed water consumption to compare the slurry flowability, setting time, and 2 h strength of each group of samples under fixed water consumption.

**3.2.1. Slurry Flowability.** Figure 5 shows the slurry flowability tests on eight groups of PBG at a fixed water consumption (60%).

The slurry flowability of the PBG increases first and then decreases as the ball milling time increases; it increases from 60 to 180.4 mm as the ball milling time increases to 50 min, representing a 200% increase. When the ball milling is >50 min, the slurry flowability decreases with a further increase in the ball milling time.

**3.2.2. Setting Time.** Figure 6 shows the variations of the setting time with the ball milling time for the eight samples of PBG at a fixed water consumption (60%).

With the increase in the ball milling time, the initial setting time of the samples increases first and then decreases (Figure 6(a)). When the ball milling time is <20 min, the initial setting time of PBG significantly increases with the increased ball milling time. When the ball milling time is >20 min, the initial setting time reaches a maximum value of 366 s when milled for 20 min, representing a 28.9% increase compared with the value of 284 s before ball milling. When the ball milling time is increased above 20 min, the initial setting time decreases with a further increase in ball milling time. With the ball milling time of 70 min, the initial setting time decreases to the minimum value of 248 s, a 32% decrease compared with the maximum value of 366 s.

With an increase in the ball milling time, the final setting time of the PBG increases and then decreases (Figure 6(b)). When the ball milling time is <20 min, the final setting time significantly increases with an increase in ball milling time. The maximum value of 618 s was reached at the milling time of 20 min, an increase of 21.7% compared with the value of 508 s before ball milling. With ball milling times greater than 20 min, the initial setting time decreases with a further

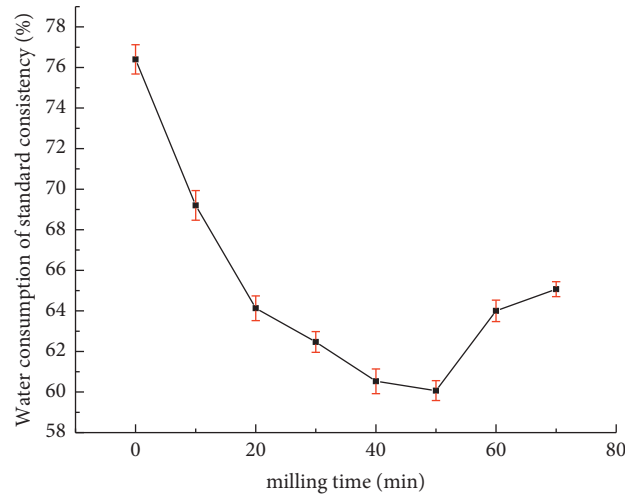


FIGURE 2: Trend of the water consumption of normal consistency.

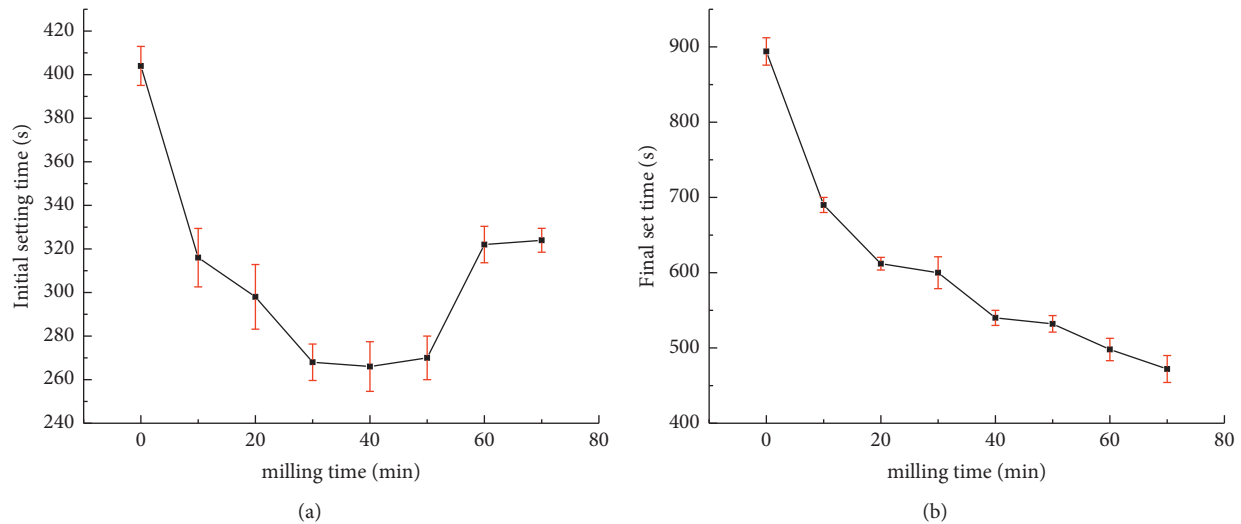


FIGURE 3: Variation of the setting time PBG with ball milling time. (a) Initial setting time. (b) Final setting time.

increase in the ball milling, reaching a minimum value of 480 s when milled for 70 min, representing a 22.3% decrease compared with the maximum value of 618 s.

**3.2.3. Mechanical Strength.** Figure 7 shows the flexural and compressive strength of the eight samples of PBG at a fixed water consumption rate (60%).

The 2 h flexural strength of the PBG fluctuated between 3.17 and 3.52 MPa as the increased ball milling time (Figure 7(a)). The compressive strength at 2 h increases initially and then decreases subsequently (Figure 7(b)). When the ball milling time is <50 min, the compressive strength gradually increases with the increase of ball milling time. The maximum value of 7.78 MPa is at 50 min mill, representing a 26.5% increase compared with 6.15 MPa before ball milling. With an additional increase in the ball milling time, the compressive strength at 2 h rapidly decreases.

### 3.3. Effect of Ball Milling Time on the PBG Powder State

**3.3.1. Distribution of Particle Size.** Figure 8 shows the particle size distribution of the PBG at different ball milling durations.

The particle size distribution of the PBG samples shows a bimodal feature with absolute peaks at 10–15 and 40–80  $\mu\text{m}$ . As the duration of ball milling increases, the peak close to the 40–80  $\mu\text{m}$  particle size shifts to the left, which indicates that the coarse end particles are broken. Moreover, the particle size gradually becomes smaller, the height of the peak close to the particle size of 10–15  $\mu\text{m}$  increases, and the particles at the fine end of the surface gradually increase; however, the particles at the fine end remain unchanged.

Based on the statistics of the particle size distribution data for the eight groups of PBG obtained from the laser particle sizer, four particle size parameters, namely, fine end size (D10), intermediate size (D50), coarse end size (D90),

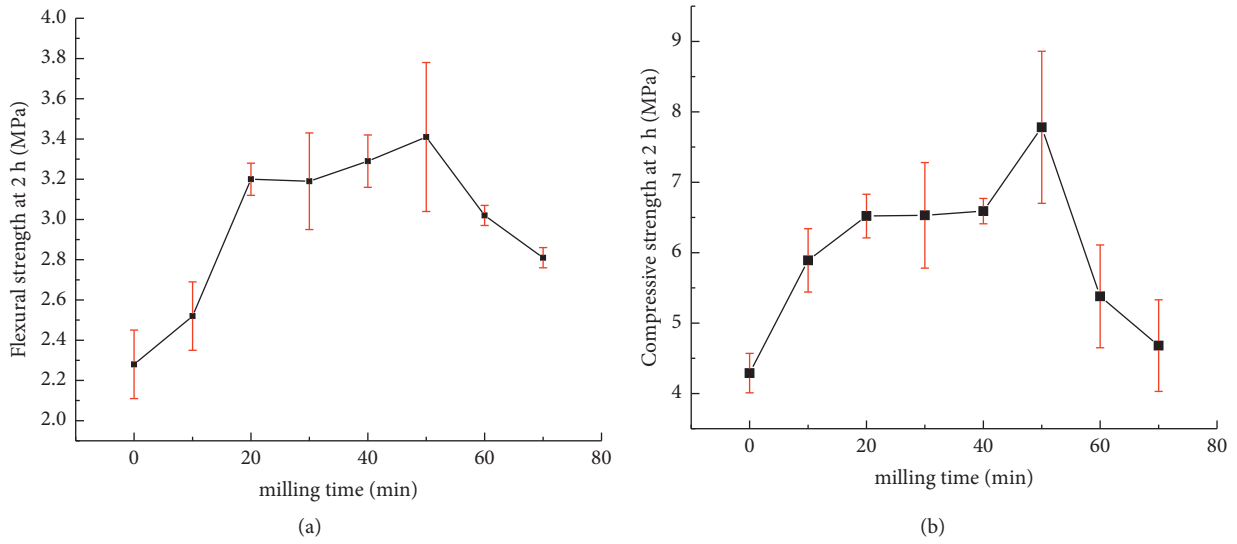


FIGURE 4: Variation of the 2 h strength of PBG with milling time. (a) Flexural strength. (b) Compressive strength.

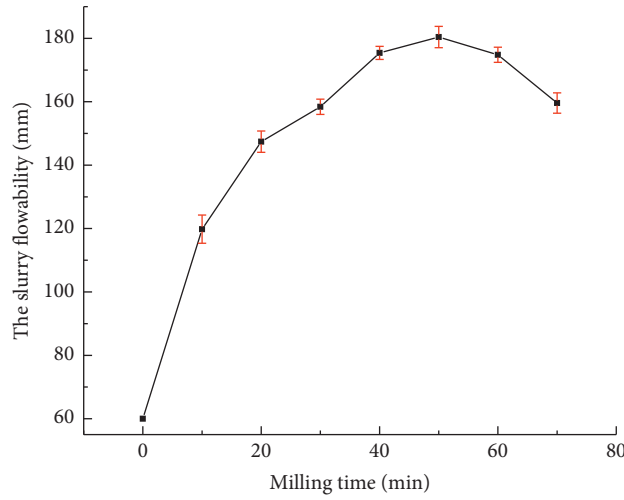


FIGURE 5: Variation of the slurry flowability of PBG with ball milling time.

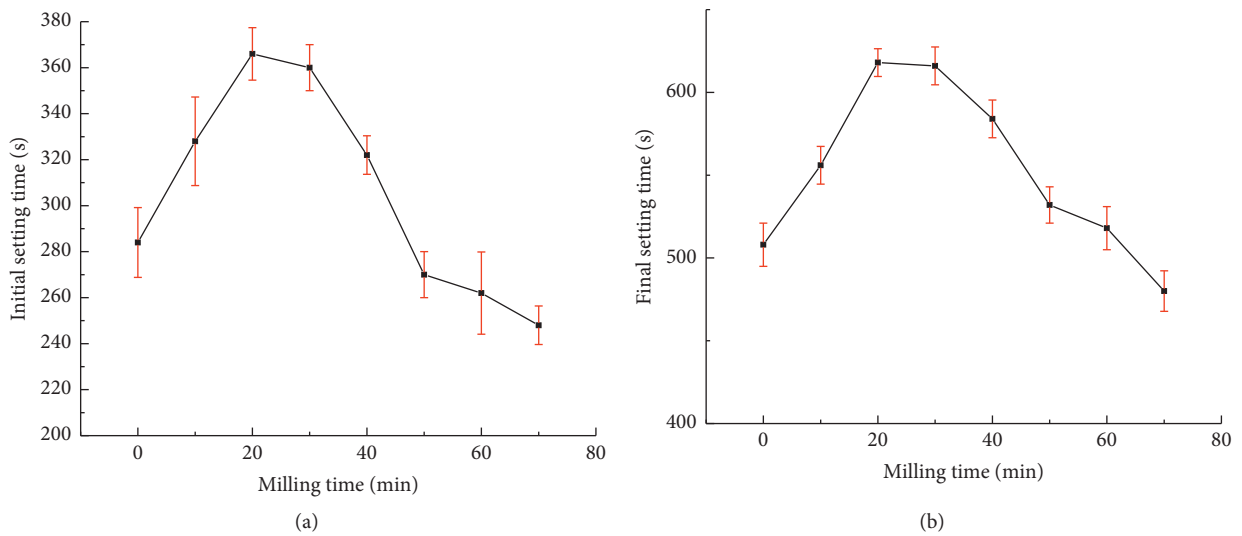


FIGURE 6: Variation of the setting time of PBG with ball milling time. (a) Initial setting time. (b) Final setting time.

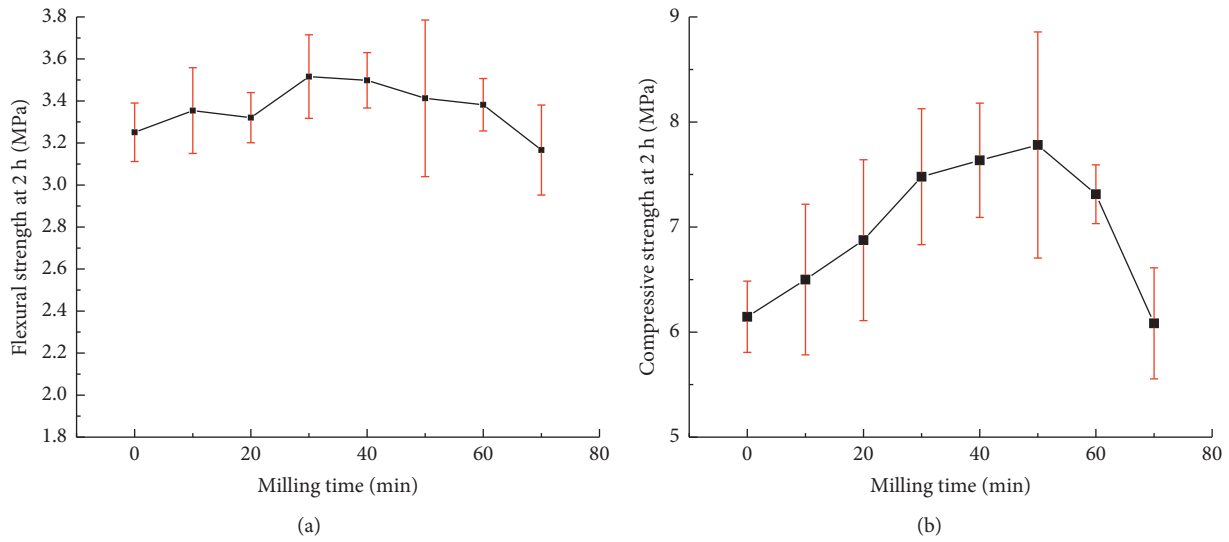


FIGURE 7: Variation of the strength of PBG at 2 h. (a) Flexural strength. (b) Compressive strength.

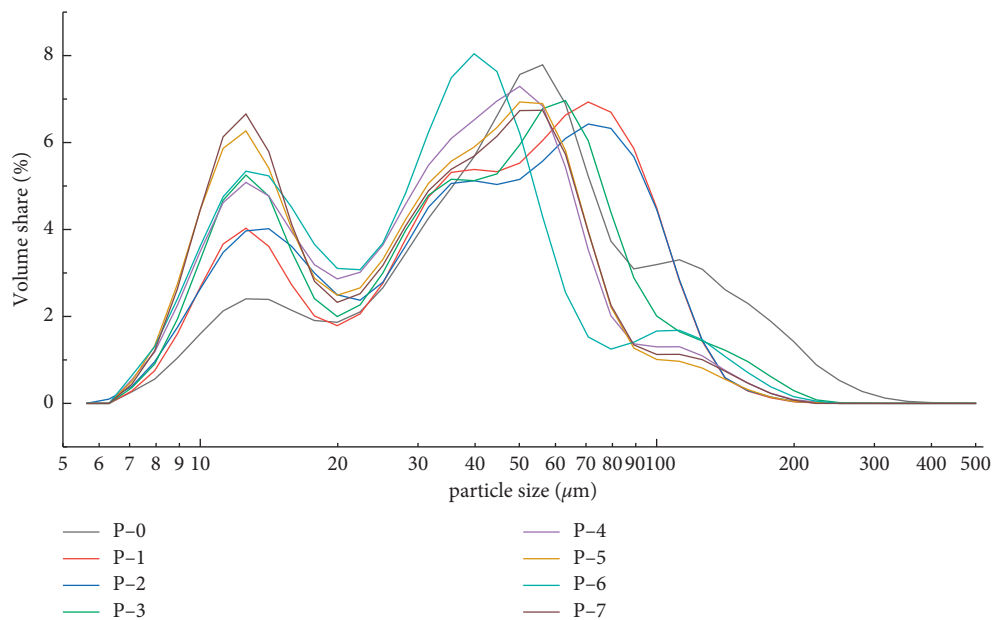


FIGURE 8: Particle size distribution of PBG.

and average size (DAV) were obtained. Figure 9 shows the variations of the four parameters with the ball milling time.

With the increase of ball milling time, D10, D50, D90, and DAV change to different degrees.

As the ball milling time increases, D10 decreases from 13.9 to 10.2 μm. At a ball milling time of 50 min, D10 reaches a minimum value of 10.2 μm, a decrease of 26% than the D10 PBG without ball-milling treatment. When the ball milling time is 10 min, D10 has the largest decline. When the ball milling time is >10 min, D10 variation decreases at >50 min, and the variation of D10 is about ~3% (Figure 9(a)).

With an increase in the ball milling time, D50 decreases from 47.5 to 30.3 μm. When the ball milling time is 60 min,

D50 reaches the minimum value of 30.3 μm, a 36% decrease compared with the value before the ball-milling treatment. When the ball milling time is 50 min, D50 decreases nearly linearly; above 50 min, D50 fluctuates with a variation of ~2% (Figure 9(b)).

With the increase in the ball milling time, D90 decreases from 126.5 to 65.2 μm. When the ball milling time is 50 min, D90 reaches the minimum value of 65.2 μm, a 48% decrease relative to the value before the ball milling. When the ball milling time is 10 min, D90 has the largest decrease. With 10 min of ball milling, D90 decreases by 61%. Above 10 min of ball milling, D90 decreases; moreover, after 40 min, it fluctuates by ~6.5% (Figure 9(c)).

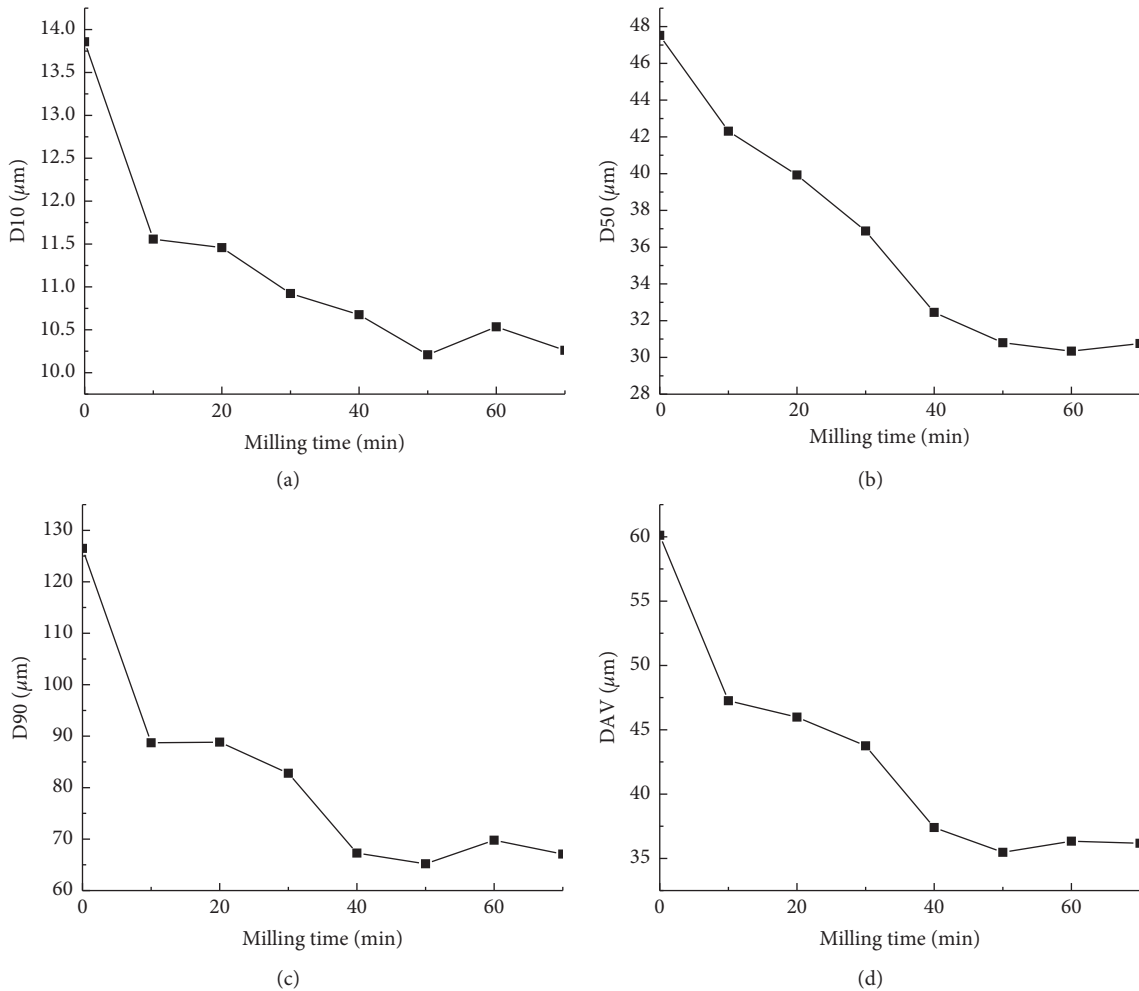


FIGURE 9: Trend of parameters of particle size of PBG. (a) D10. (b) D50. (c) D90. (d) DAV.

With an increase in ball milling time, DAV decreases from 60.1 to 35.5  $\mu\text{m}$ . When the ball milling time is 50 min, DAV reaches the minimum value of 35.5  $\mu\text{m}$ , a 41% decrease compared with the value before ball milling. When the ball milling time exceeds 50 min, DAV fluctuates with a variation of  $\sim 3\%$  (Figure 9(d)).

**3.3.2. Specific Surface Area.** Figure 10 shows the variation in the specific surface area of PBG with an increase in ball milling time.

The specific surface area of phosphate gypsum gradually increases as the ball milling time increases, reaching a maximum value of 2813.65 (S/V) at the ball milling time of 50 min, which is a 55% increase compared with the value (1815.23 (S/V)) before the ball milling. The specific surface area increases nearly linearly when the ball-milling treatment time is within 50 min; moreover, the D50 increases by  $\sim 2\%$  when the ball milling time exceeds 50 min. When the treatment time exceeds 50 min, the specific surface area fluctuates with a variation of  $\sim 2\%$ .

**3.3.3. Particle Morphology.** The particle morphology of the PBG was observed using SEM (Figure 11).

Before ball milling, the particles of the PBG are diamond-shaped with apparent angles. After ball milling, rhombic particles are observed, and the angles are reduced. When the ball milling time reaches 50 min, the particles lose their original shape and become irregularly shaped. When the ball milling time reaches 70 min, the particles are almost oval-shaped.

### 3.4. Mechanism of the Effect of Ball-Milling Treatment on the Properties of PBG

**3.4.1. Mechanism of the Effect of Ball-Milling Treatment on the Water Consumption of Normal Consistency and Slurry Flowability at Fixed Water Consumption.** Varying the time of ball-milling treatment can change the crystal particles of PBG from diamond-shaped flakes to oval-shaped crystal particles. However, the relative thickness of the particles decreases. When water is added to PBG to form a slurry, particles with an oval shape and smaller relative thickness

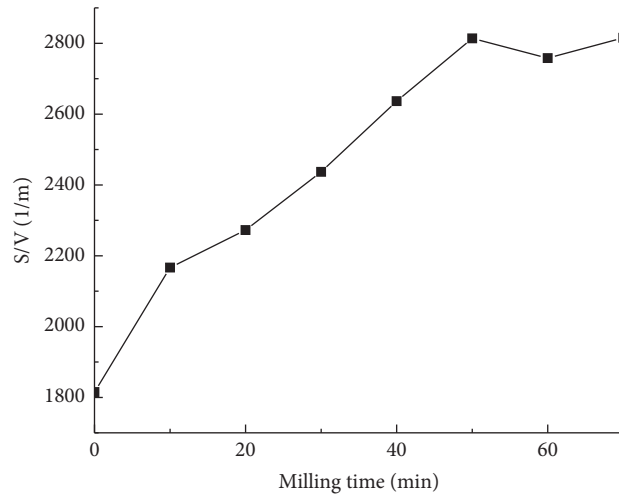


FIGURE 10: Variation of the specific surface area of PBG.

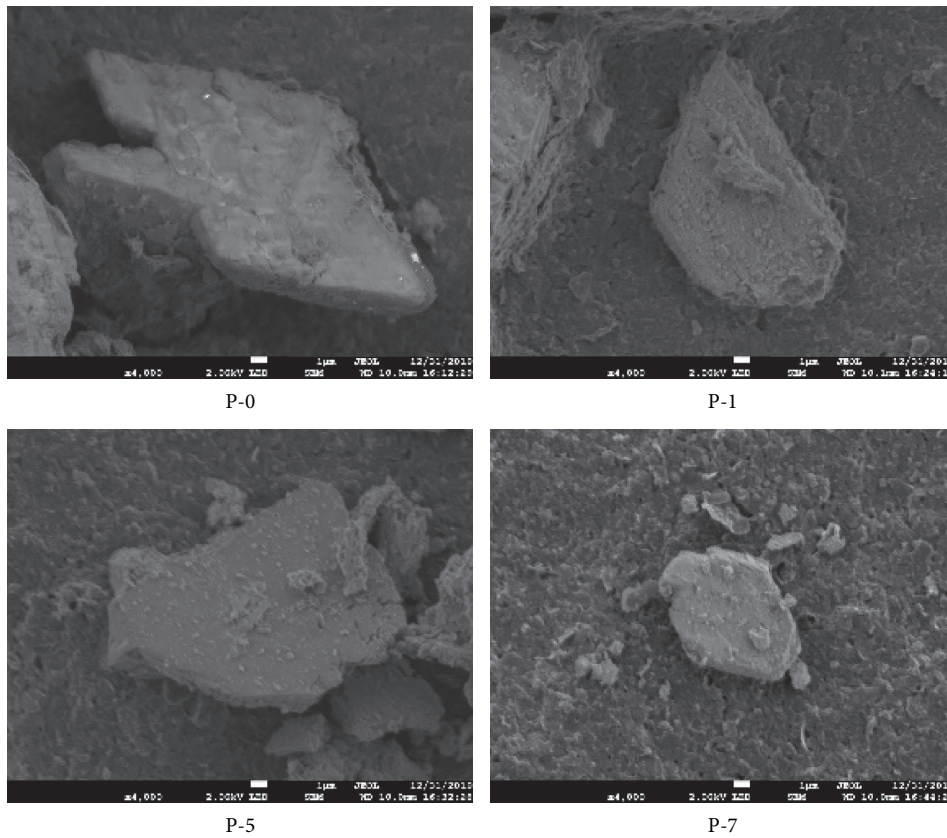


FIGURE 11: Particle morphology (4000×).

after ball milling would have better flowability. The fine-end particles increase after ball milling. These fine-end particles (around 10 μm) form a “ball effect,” which further increases the fluidity of the slurry and reduces the water consumption of normal consistency.

3.4.2. Mechanism of the Effect of Ball Milling on the Setting Time. As shown in Figure 3, under the water consumption

of normal consistency, ball-milling treatment has a larger effect on the setting time of PBG. However, Figure 6 shows that the effect of ball milling on the setting time of PBG exhibits a trend of increasing first and then decreasing. Ball-milling reduces the water consumption of normal consistency of PBG. Hence, setting time can be reduced by reducing water consumption. Moreover, ball milling decreases the particle size of the material and increases the specific surface area, thereby increasing the dissolution rate of



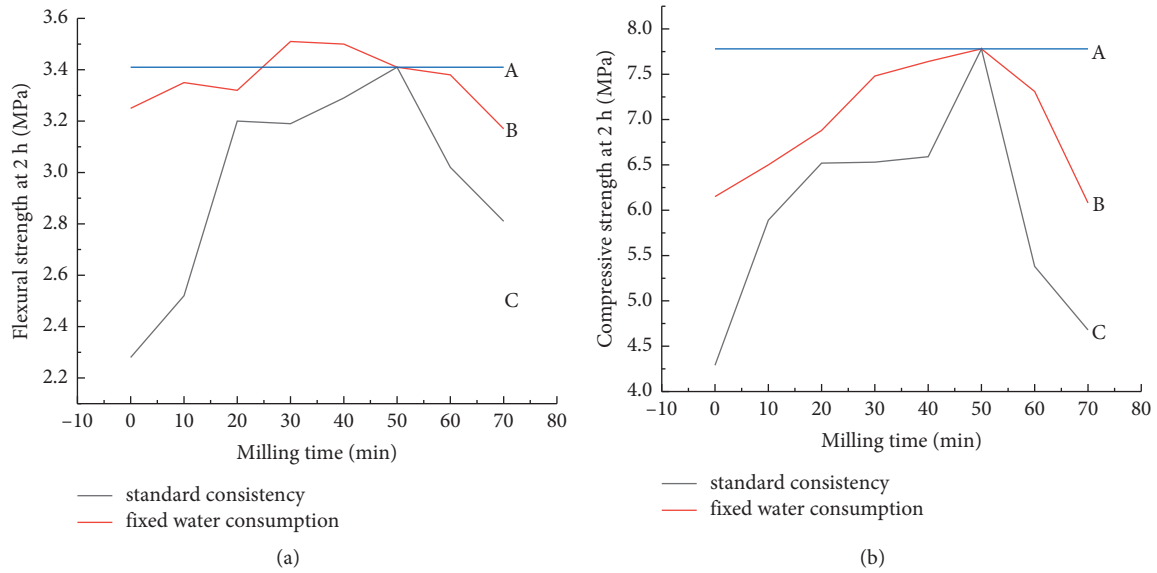


FIGURE 12: Trend of the strength of PBG at 2 h. (a) Flexural strength (b) Compressive strength.

TABLE 3: Comparison of the effect of ball-milling treatment and other treatment methods.

Performance index	Here	[9]	[9]	[18]	[19]	[23]
Treatment methods	Ball milling	Ball milling and sieving	Ball milling and washing	Ball milling	Ball milling and aging	Aging
The water consumption of normal consistency (%)	60	80	74	68	76	82
Initial setting time (min)	4.5	3.5	5	-	8	9.5
Final setting time (min)	8.87	6	7.5	-	13	15.5
Flexural strength at 2 h (MPa)	3.41	2.55	2.61	2.39	2.28	2.21
Compressive strength at 2 h (MPa)	7.78	4.62	5.53	5.47	4.06	4.43

TABLE 4: Comparison of ball milling PBG with national standards.

Performance index	Untreated PBG	PBG after 50 min of ball milling	“Building gypsum” (GB/T 9775-2008) (level 1)
Fineness (0.2 mm square hole sieve residue) (%)	2	0.2	≤10
Initial setting time (min)	6.73	4.5	≥3
Final setting time (min)	14.9	8.87	≤30
Flexural strength at 2 h (MPa)	2.28	3.41	≥3.0
Compressive strength at 2 h (MPa)	4.29	7.78	≥5.0

calcium sulfate hemihydrate crystals following the law of mechanical force chemistry [20] and further resulting in a higher hydration rate and less setting time of PBG.

As the particle size of PBG decreases, the release of eutectic phosphorus from the crystals is accelerated, which will retard PBG to some extent [20, 22]. However, in the fixed water consumption (60%) test, the release of eutectic phosphorus slowed the setting of the material at ball milling times of <20 min, indicating that the PBG setting time increases with an increase in ball milling time. When the ball milling time is >20 min, the particle size of the material decreases. The effect of accelerated hydration is more prominent; the overall setting time of the material

decreases with the increased ball milling time. Thus, the overall setting time is reduced with an increase in ball milling time.

3.4.3. Mechanism of the Effect of Ball Milling on the Strength at 2 h. Figure 12 compares the strength of PBG at the water consumption of normal consistency of 60%

Ball-milling treatment can improve the flexural and compressive strength at 2 h of PBG primarily by reducing the water consumption of normal consistency. In addition to the flexural strength at 2 h, which is improved by ball milling at fixed water consumption (60%). The effect on the floral

strength at fixed water consumption is little; the flexural strength fluctuates in a small range. However, the compressive strength at 2 h of PBG is enhanced.

#### 4. Potential Applications and Prospects of PBG

On the one hand, because the composition of phosphogypsum is complex, many impurities would affect the performance of phosphorus building gypsum; on the other hand, the poor particle shape and particle size distribution of phosphogypsum also adversely affects its performance. In recent years, most of the phosphogypsum would be treated by water washing or calcium oxide neutralization method during the production and be made to meet the index requirements in Chinese Standard GB/T 9775-2008. According to the results of this paper, without using methods as screening, washing, and aging, only by improving the particle shape and particle size distribution of phosphogypsum at the appropriate time for reducing the water consumption of normal consistency can obtain a good performance of building gypsum and meet the criterion of first-class requirements, as shown in Tables 3 and 4. Compared with treatments of screening, washing, and aging, the ball grinding not only has the advantages of simplicity, friendly to the environment, and low cost but also has a certain production practical significance.

#### 5. Conclusion

In this paper, the effects of ball-milling treatment on the properties of PBG were examined. From the obtained results, the following conclusions were drawn:

- (1) Ball-milling treatment can effectively reduce the particle size and increase the specific surface area of PBG. With a milling time of 50 min, the average particle size of PBG reaches a minimum of 35.5  $\mu\text{m}$ , which is 41% lower than the value of the unmilled PBG. The specific surface area reaches the maximum value of 2813.65 (S/V), which is an increase of 55% compared with the value of unmilled PBG. Ball-milling treatment can crush the diamond-shaped gypsum particles, reduce the edges and corners, and form elliptical flakes.
- (2) Ball milling can effectively reduce the water consumption of normal consistency and increase the 2 h strength of PBG. When the ball milling time is 50 min, the water consumption of normal consistency reaches a minimum of 60.07%, which is 21.5% lower compared with unmilled PBG. The flexural strength at 2 h reaches a maximum value of 3.41 MPa, an increase of 49.6% compared with the 2.28 MPa for the unmilled PBG. The compressive strength at 2 h reaches a maximum value of 7.78 MPa, which is an increase of 81.4% compared with the 4.29 MPa of the unmilled PBG.
- (3) Ball-milling treatment can effectively reduce the water consumption of normal consistency of PBG by improving the particle shape and particle-size distribution. Thus, the ball-milling treatment increases

the 2 h strength of the PBG. The effect results from the change in water consumption of normal consistency (compared with the case of fixed water consumption (60%)), and the ball-milling treatment enhances the compressive strength at 2 h of PBG.

#### 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 there are no conflicts of interest regarding the publication of this paper.

#### Acknowledgments

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