Effect of the phosphogypsum calcination time on the compressive mechanical properties of phosphogypsum-based composite cementitious materials

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Abstract
In this study, phosphogypsum-based composite cementitious materials (PGCMs) were prepared by adding fixed proportions of additives to calcined phosphogypsum (PG). Samples with dimensions of 40 × 40 × 80 mm and 150 × 150 × 300 mm were used to study the effect of the PG calcination time on the PGCM compressive strength, stress–strain relationship, and failure mode and its mechanism. The test results indicated that the PGCM compressive strength gradually increased as the calcination time increased. When the PG calcination time was 180 min, the compressive strengths of the smaller and larger samples increased by 3 and 3.6 times, respectively, compared with the strengths at a calcination time of 20 min. The main reason for the strength increase was the formation of ettringite and hydrated calcium sulfate dihydrate in the PGCM gel system. Additionally, the PGCM compressive strength was significantly related to the sample size, and its reduction coefficient was between 0.60 and 0.69 at different PG calcination times. As the PG calcination time increased, the peak stress of the PGCM stress–strain curve and the corresponding axial strain increased gradually; moreover, brittle failure became more evident.

1. Introduction
Phosphogypsum (PG) is one of the byproducts of the phosphate fertilizer industry [1]. About five tons of PG are produced for every ton of phosphoric acid produced. The global annual PG production is about 100 to 280 million tons [2], but only 15% of it is recovered and recycled [3]. Currently, the annual PG emission of China is nearly 75 million tons, and the accumulated stockpiles exceed 500 million tons. However, the comprehensive utilization rate is only 40% [4]. Although the utilization rate has increased, a large gap still exists between the output and consumption weight. In the past 10 years, PG has been mainly used in the construction industry as a cement retarder based on the PG utilization paths and proportions in China [5–7].

It can also be made into gypsum products, such as gypsum board [8, 9] or gypsum bricks [10–12]. Moreover, in some studies, phosphogypsum-based composite cementitious materials (PGCMs) have been prepared by mixing PG with other additives and corresponding activators [13]. PGCMs have advantages such as great heat preservation, heat insulation, sound insulation, and high PG utilization ratio [14]. However, low strength has become the main factor limiting the wide application of PGCMs. Hence, research work exploring PGCM properties has attracted much attention, including studies regarding setting time [15, 16], water resistance [17, 18], and other physical or mechanical properties [19, 20].

Phosphogypsum-based composite cementitious materials can be mainly divided into anhydrous PGCM, hemihydrate PGCM, and dihydrate PGCM. Anhydrous gypsum needs to be calcined under high temperature, i.e., 360 °C to 1000 °C. The calcination product has a low strength, but the calcination process requires high energy consumption and can easily cause secondary contamination to the environment [21]. Hemihydrate gypsum is formed through the removal of 1.5 crystal water molecules from dihydrate gypsum under 100 °C to 360 °C [22]. Two types of hemihydrate gypsum materials exist: α-type hemihydrate gypsum and β-type
hemihydrate gypsum. The α-type hemihydrate gypsum is formed through the heating of dihydrate gypsum under pressurized steam or in acid or salt solution, which are both complicated processes [23]. The β-type hemihydrate gypsum is formed through the dehydration of dihydrate gypsum in a dry environment, but its products feature low mechanical strength, vulnerability to temperature, and poor waterproof performance [24]. Dihydrate PGCMs are prepared by mixing dihydrate gypsum with several additives, including fly ash, slag, cement, and activators [25]. The mechanical strength of most cementitious materials mixed with untreated dihydrate PG might be reduced, but the mechanical strength can be improved via the calcination and dehydration of PG [1, 26, 27]. Therefore, to solve the non-gelling and self-hardening issues of original PG [28], calcination or dehydration methods can be used to form hemihydrate PG. Certain additives can be subsequently added to prepare PGCMs. This approach is effective for promoting the comprehensive utilization of PG, because it not only improves the matrix strength but also increases the PG utilization rate.

Currently, adding additives is one of the major ways to improve the strength of PGCMs. Additives such as cement, fly ash, quicklime, and slag all have a positive effect on improving cement strength. Jin et al [24] prepared hardened samples using a mixture of PG and cement to study the compressive properties of the samples. The compressive strength increased as the cement content increased, and the maximum compressive strength could reach 20 MPa. Kumar et al [29] studied the effect of fly ash on the strength of PG hollow blocks and the influencing mechanism. The calcium sulfoaluminate and silicate hydrate formed after the addition of fly ash effectively increased the matrix strength. Liu et al [30] studied the effect of cement, slag, quicklime, and other additives on PG strength. Through orthogonal experiments, an optimal proportion of each additive was found, and the highest strength was found as 41.70 MPa. Additionally, PG has been calcined in other studies to introduce gelling and self-hardening effects, and the effect of the calcination temperature on the PGCM strength has been revealed. In these studies, the overall law was that the material strength increased with the calcination temperature. Meanwhile, the additive type also influenced the optimal calcination temperature. Bumanis et al [31] studied PG containing rosin resin (plasticizer) under calcination temperatures between 100 °C and 180 °C and concluded that the cementitious material strength increased with the temperature. Yang et al [32] investigated the strength of calcined PG cementitious materials containing fly ash under different temperatures (135 °C, 430 °C, and 800 °C) and found that the material calcined at 430 °C exhibited the highest strength. Ren et al [33] calcined PG for 2 h under 400 °C, 500 °C, 600 °C, 700 °C, and 800 °C. They prepared cementitious materials by mixing PG at a proportion of 33% with fly ash, ordinary silicate cement, lime, and chemical activators. The study investigated the effect of PG calcination temperature on cementitious material strength, and the PG optimal strength was achieved at 700 °C. The above studies show that the PG calcination temperature has a profound impact on the PGCM strength, and the temperature can be controlled to optimize the material strength. Besides calcination temperature, calcination time is another important parameter. One study [34] has shown that a too-long or too-short calcination time can significantly decrease the PG quality. Moreover, Geraldo et al [35] studied the effect of calcination time (1 to 6 h) on gypsum strength under a calcination temperature of 150 °C and concluded that the maximum strength was achieved under 3 h calcination time. However, the existing studies mainly focus on the effect of PG calcination temperature on strength. There are few studies on the effect of PG calcination time; therefore, it is necessary to study the effect of calcination time on the PGCM strength.

At present, the reference standards for obtaining the compressive strength of PGCMs are mainly GB/T 17671 ‘Method of Testing Cements—Determination of Strength (ISO Method)’ [36] and GB/T 50081 ‘Standard Test Methods for Mechanical Properties of Ordinary Concrete’ [37]. The considered sample sizes were 40 × 40 × 80 mm and 150 × 150 × 300 mm. Generally, the strength of quasi-brittle materials such as concrete, mortar, and rock is related to their size [38], and the studies on the size effect on PGCM compressive strength are few. Therefore, revealing the size effect on PGCM strength and establishing the size–strength relationship of samples for two different sizes will provide a reference for PGCM application under different working conditions.

This study investigated PGCMs prepared by mixing calcined PG with fixed proportions of additives. A uniaxial compression test was used to measure the PGCM compressibility, investigate the effect of PG calcination time on compressible strength and clarify the stress–strain relationship. Scanning electron microscopy (SEM) coupled with x-ray diffraction (XRD) was used to analyze the microstructural change and reveal the compressibility enhancement mechanism of the PGCMs.

2. Experimental process

Original PG, dehydrated PG, ash-calcium-powder, and cement, as raw materials, were used to prepare the PGCMs. The chemical compositions of the original PG, dehydrated PG calcined at 160 °C for 120 min, and the additives (ash-calcium-powder and cement) are presented in table 1.
The pH and water content of the original PG were 6.82 and 18.5%, respectively. The materials containing hardened blocks were crushed and passed through a square-hole sieve with a hole size of 0.315 mm, and the thickness of each plate was maintained at 10 mm. The materials were placed in a 160°C constant-temperature electric heating blast drying oven for calcination. Different PG calcination times were adopted: 20 min, 30 min, 60 min, 90 min, 120 min, and 180 min, respectively.

PGCM was a mixture of PG, ash-calcium-powder, a polycarboxylate water reducing agent, a protein-type gypsum retarder, and water. The calcined PG was mixed with fixed proportions of additives. The mass ratio of ash calcium powder to PG was 6:94, and the mixture contained 9% cement, 0.6% polycarboxylate water reducing agent, and 0.3% protein-type gypsum retarder. The amount of water equivalent to a flow expansion degree of 220 ± 5 mm was needed, and such amount was confirmed based on GB/T 17669.4–1999 [39]. The evenly stirred slurry was then poured into test molds of 150 × 150 × 300 mm and 40 × 40 × 160 mm, respectively.

The molds were vibrated 5 to 10 times, and then the slurry surface was scraped. After the slurry was left to stand for 12 h, the molds were removed, and the samples were cured under natural conditions for 28 days before they were used for the compression test. According to GB/T 17671 Method of Testing Cements—Determination of Strength (ISO Method) [36], the 40 × 40 × 160 mm sample was broken into two prisms, and one of them (40 × 40 × 80 mm) was subjected to a compression test. The pressed surfaces were the two sides with an area of 40 × 40. There were six groups of samples for each size, and three samples in each group.

Table 1. Chemical composition of materials (w/%).

| Material          | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO  | SO₃ | P₂O₅ | K₂O | SrO | F     | Cl  |
|-------------------|------|-------|-------|------|-----|------|-----|-----|-------|-----|
| Original PG       | 2.678| 0.299 | 0.368 | 39.522| 55.280| 0.887| 0.065| 0.061| 0.648 | /   |
| Dehydrated PG     | 2.709| 0.292 | 0.382 | 41.840| 53.601| 0.864| 0.065| 0.057| /     | /   |
| Ash-calcium-powder| 0.399| 0.140 | 0.111 | 98.292| 0.238| 0.008| 0.581| 0.014| /     | 0.014|
| Cement            | 19.897| 5.155 | 4.456 | 61.713| 3.962| 0.169| 1.725| 0.158| /     | 0.036|

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PGCM was a mixture of PG, ash-calcium-powder, a polycarboxylate water reducing agent, a protein-type gypsum retarder, and water. The calcined PG was mixed with fixed proportions of additives. The mass ratio of ash calcium power to PG was 6:94, and the mixture contained 9% cement, 0.6% polycarboxylate water reducing agent, and 0.3% protein-type gypsum retarder. The amount of water equivalent to a flow expansion degree of 220 ± 5 mm was needed, and such amount was confirmed based on GB/T 17669.4–1999 [39]. The evenly stirred slurry was then poured into test molds of 150 × 150 × 300 mm and 40 × 40 × 160 mm, respectively.

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Based on GB/T 17671 Cement Mortar Strength Test Method (ISO Method) [36] and GB/T 50081–2002 Standard Test Methods for Mechanical Properties of Ordinary Concrete [37], an electrohydraulic servo universal testing machine was used to conduct uniaxial compression test on samples of different sizes (40 × 40 × 80 mm and 150 × 150 × 300 mm), and the sample loading rate was 2 kN/s. The test equipment is displayed in figure 1.

Scanning electron microscopy (Model: ZEISS MERLIN Compact) was adopted to scan the microscopic morphology of the original materials and samples. X-ray diffraction (Model: Bruker D8 Advance) was used for phase analysis, and x-ray fluorescence spectrometry (Model: Zetium, PAN analytical, Netherlands) was employed for chemical composition analysis of the original materials.

3. Results and discussion

3.1. Effect of calcination time on PG compressive strength

The performances of PG materials calcined under different conditions are presented in table 2.

According to the specifications [40], both PG-5 and PG-6 sample groups met the mechanical performance standards for level 1.6 phosphorous building gypsum (i.e., two-hour compressive strength ≥3.0 MPa, two-hour
flexural strength $\geq 1.6$ MPa). Moreover, the standard water to cement ratio values were all greater than 0.65, which was one of the reasons for the low mechanical strength of the PG hardened body.

The microscopic morphologies of the original PG and the PG calcined under 160 °C for 120 min are presented in figure 2. The original PG featured rhombus blocks as its main morphology and long and thick plates. After calcination under 160 °C for 120 min, vertical microfractures occurred inside the PG and on its original surface. Some PGs with small particle size also appeared, and the specific surface area increased. Wu et al [41] also found that the hemihydrate PG had a larger specific surface area than the original PG. The phase composition of PG at different calcination times was analyzed via XRD.

Figure 3 shows the phase composition of PG at different calcination times under a 2θ range of 10° to 60°. As shown in the figure, the calcination temperature caused the phase change of PG and promoted the removal of crystal water in dihydrate PG. As the PG calcination time increased, the dihydrate PG diffraction peak gradually...
weakened, and the peak intensity decreased as well. When the PG calcination time was 90 min, the peak intensity of the dihydrate PG was equivalent to that of the hemihydrate PG. When the PG calcination time was 120 min, the diffraction peak of dihydrate PG completely disappeared. Only the hemihydrate PG diffraction peak and the anhydrous PG peak were detected, which was the key reason for the dramatic increase in the PG strength. When the calcination time was 120 min and 180 min, the intensity of the hemihydrate PG peak stabilized. The study by Değirmenci et al. showed that the mechanical performance of the hardened cementitious body mixed with the original PG decreased, but the compressive strength of the cementitious material mixed with calcined PG increased. Therefore, this study investigated the PGCM with calcined PG to reveal the effect of the PG calcination time on its compressive strength.

3.2. Effect of calcination time on PGCM compressive strength

Figure 4 shows the compressive strengths of the PGCM samples (with dimensions of 150 × 150 × 300 mm) after 28 days of natural curing and at different PG calcination times. The PGCM compressive strength increased as the PG calcination time increased. When the calcination time increased from 20 min to 180 min, the compressive strength increased from 7.87 MPa to 31.41 MPa, approximately a three-fold enhancement. This
occurred because as the PG calcination time increased, 1.5 water molecules were removed from calcium sulfate dihydrate, and hemihydrate PG was formed. The hemihydrate PG content gradually increased, and hemihydrate PG reacted with water and additives to form minerals such as ettringite and calcium sulfate dihydrate. The formation of these minerals enhanced the matrix strength. Hence, the hemihydrate PG content was an important factor influencing the PGCM strength.

Figure 5 shows the compressive strengths of the PGCM samples with dimensions of 40 × 40 × 80 mm after 28 days of natural curing and at different PG calcination times. The sample compressive strength increased as the PG calcination time increased. With an increase in the calcination time from 20 min to 180 min, the compressive strength increased from 11.41 MPa to 52.60 MPa, about 3.6-fold enhancement. The strength enhancement mechanism of the sample was similar to that of the large sample (150 × 150 × 300 mm). As the PG calcination time increased, 1.5 water molecules escaped from calcium sulfate dihydrate, and calcium sulfate hemihydrate was formed. As the hemihydrate PG content gradually increased, the formed minerals such as ettringite and hydrated calcium sulfate dihydrate enhanced the compactness of the hardened body, thereby increasing the matrix strength.

The surface morphology of the sample (with dimensions of 40 × 40 × 160 mm) revealed that pores appeared on the surfaces of the PGCM samples at the calcination times of 20 and 30 min. However, when the calcination time was 60 min, the surface porosity dramatically decreased. This was also the reason the compressive strength at this calcination time was 132.4% higher than that at 30 min calcination time. Overall, with longer PG calcination time, the PGCM sample surface became more compact, and the compressive strength increased.

Figure 6. Scanning electron microscopy images of PGCMs under different calcination conditions.
The SEM images of PGCM hardened body samples at different calcination times are presented in figure 6 to further reveal the effect of calcination time on the sample compressive strength.

The PGCM hydration products calcined under the different times all contained ettringite (3CaO·Al₂O₃·3CaSO₄·32H₂O, AFt) and calcium sulfate dihydrate (CaSO₂·2H₂O). As the calcination time increased, more hydration products were formed, and the structure became denser. When the calcination time was 20 min (figure 6(a)) and 30 min (figure 6(b)), the sample network structure was loose. The evaporation of water near the overlap points of the gypsum crystals resulted in the formation of many pores in calcium sulfate dihydrate. Meanwhile, a small amount of acicular ettringite existed. The crystal surface was covered with a fluffy and flocculent substance, C-S-H (Ca₅Si₆O₁₆(OH)₄·4H₂O). The interlocking phenomenon was very weak.

Moreover, dihydrate gypsum with low crystallinity and incompletely dissolved hemihydrate gypsum formed a loose microstructure, which increased porosity. Such a phenomenon was consistent with the sample surface morphology analysis. Therefore, loose structure and large porosity are the major reasons for the low compressive strength of the samples calcined at 20 min and 30 min.

When the PG calcination time was 60 min (figure 6(e)) and 90 min (figure 6(d)), the hydration products increased dramatically but porosity decreased substantially. The interlocking between ettringite and gypsum crystals increased, and the crystallinity increased. The increased interlocking was the direct cause of the significant strength increase at 60 min. With an increase in the calcination time from 30 min to 60 min, the compressive strength increased dramatically, by 138.5% and 132.4%, respectively. Needle-like ettringite and C-S-H hydration products occurred in the samples calcined at 120 min (figure 6(e)) to 180 min (figure 6(f)). The two products were closely connected, connecting the PG particles with the hydration products. Hence, a dense microstructure was formed, which improved the interface bonding strength and resulted in a significant increase in the PGCM compressive strength during long-time sintering. In addition, the SEM morphology analysis results show that when the calcination time exceeded 60 min, the fraction of brittle failure increased. Since brittle failure usually requires higher energy, the sample compressive strength also increases. In summary, the PG calcination time directly affected the fractions of ettringite and calcium sulfate dihydrate hydration products and hemihydrate PG. Such phenomena further affected the material microstructure, porosity, and failure mode, increasing the PGCM compressive strength with the calcination time.

The compressive strengths of samples with different dimensions (figures 4 and 5) were compared. From figures 4 and 5, the compressive strengths of both samples increased with the calcination time. The compressive strength of the small sample (40 × 40 × 80 mm) increased more dramatically than that of the large sample (150 × 150 × 300 mm). Figure 7 shows the strength reduction coefficient of the two samples.

At the same calcination time, the compressive strength of the small sample was higher than that of the large sample. The strength reduction factor was between 0.60 and 0.69. Thus, the PGCM strength was closely related to the sample size. The smaller sample showed the higher strength. One study [42] indicated that the mechanical strength of ultra-tough cementitious composites also increased as the sample size decreased. Moreover, when the calcination time was above 30 min, the strength reduction factor decreased gradually as the calcination time increased, and the PGCM size effect increased.
3.3. Stress–strain curve

Figure 8 shows the stress–strain relationship of the large PGCM sample (150 × 150 × 300 mm) under uniaxial compression. The three stress–strain relationship curves in each figure were obtained from three samples at a constant PG calcination time.

From the figure, the PGCM stress–strain curve mainly included three stages. The first stage is the elastic stage, where the stress–strain relationship was linear. The second stage is the fracture initiation and propagation stage, where fractures developed inside the sample, defects expanded, fracture surfaces formed inside the sample, and strain increased at a faster rate; also, the stress–strain relationship was non-linear. The third stage is the accelerated crack propagation and penetration stage, where the fracture propagation inside the sample accelerated, and macroscopic fractures were formed through the sample. Fractures occurred at the sample surface, and the stress–strain relationship entered the descending stage. As the PG calcination time increased, the sample peak stress and its corresponding axial strain increased gradually. When the PG calcination time reached 30 min, the stress–strain curve declined by a small extent, while the extension line was longer. As the calcination
time increased, the stress–strain curve after the peak declined by a large extent. Such a phenomenon indicates that the PGCM became more brittle as the PG calcination time increased, which is consistent with the results shown in figure 7.

Figure 9 shows the stress–strain relationship of the small PGCM sample (40 × 40 × 80 mm) under uniaxial compression. At a constant PG calcination time, three samples were tested to obtain the stress–strain relationship curves.

As the calcination time increased, the sample peak stress and its corresponding axial strain increased gradually, and the sample brittle failure became more obvious. This was because the longer calcination led to lower porosity and crack fraction; hence, the PGCM densification effect became greater, and the compressive peak load became higher. However, when the maximum compression strength was exceeded, cracks propagated rapidly, and the sample underwent brittle failure.

A comparison of figures 8 and 9 indicates that the stress–strain relationship curves of the two samples were different. Significant size effect was observed for the PGCM stress–strain relationship. Under the same calcination time, the fracture initiation and propagation of the large sample were more significant than those of the small sample, which was manifested as the shorter extension section of the larger sample. This was mainly because the large sample contained more internal defects. Hence, the microstructure was the direct reason for the size effect on the PGCM compressible strength and the stress–strain relationship.

4. Conclusions

In this study, PGCMs were prepared using calcined PG at different calcination times. The effects of calcination time on the calcined PG strength and the PGCM compressive strength and stress–strain relationship were investigated. Scanning electron microscopy coupled with XRD was used to reveal the mechanism of strength enhancement based on the microscopic structure. The major conclusions are as follows.

(1) The calcination time had a profound impact on the compressive strength of the calcined PG and the PGCM. As the calcination time increased, the compressive strengths of the calcined PG and the PGCM increased gradually. When the calcination time was 180 min, the mechanical strength of the PGCM sample of 150 × 150 × 300 mm was 31.41 MPa, and that of the 40 × 40 × 80 mm sample was 52.60 MPa. The values were 3 times and 3.6 times the mechanical strengths at 20 min calcination time, respectively. The PGCM compressive strength was significantly related to the sample size, and the compressive strength reduction coefficient was between 0.60 and 0.69.

(2) The PG calcination time directly influenced the hemihydrate PG content in the PGCM. The contents of ettringite and hydrated calcium sulfate dihydrate increased as the calcination time increased, which reduced the matrix porosity and made the microstructure denser. This was the key cause of the increase in the PGCM compressive strength.

(3) As the PG calcination time increased, the peak stress of the PGCM stress–strain curve and the corresponding axial strain increased gradually; moreover, the slope of the rising section of the stress–strain curve increased gradually, the falling section of the curve became steeper, and the brittle failure became more obvious.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.
Declaration of interest statement

We declare that we do not have any commercial interests or associations that represent a conflict of interest in connection with this work.

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