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# Preparation of Foamed Phosphogypsum Lightweight Materials by Incorporating Cementitious Additives

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As a byproduct of phosphoric acid industry, phosphogypsum has many environmental problems. In order to recycle phosphogypsum to manufacture lightweight building materials, cementitious additives including fly ash, ground granulate blast-furnace slag and Portland cement were added to improve strength and water-resistance and different volume of foam was added to reduce the bulk density. The results show that hydrated lime can improve mechanical strength and water resistance of PG paste and the optimal dosage of hydrated lime is 6 %. Higher addition of fly ash or ground granulated blast-furnace slag improves the fluidity and delays the setting time of PG paste. The addition of 10 ~ 20 % fly ash results in a little reducing influence and 10 % ground granulated blast-furnace slag leads to an increase of 20.7 % for 28 days compressive strength of hardened PG specimen. The higher addition of Portland cement results in the better mechanical strength and water resistance of PG specimens. The 28day compressive and flexural strength reaches 25.9 MPa and 8.9 MPa respectively for the 25 % Portland cement mixture. PG based lightweight building materials can prepared by the addition of 60 % volume of air foam, with compressive strength of 1.7 MPa, bulk density of 521.7 kg/m$^3$ and thermal conductivity of 0.0724 W/(m·K).

*Keywords:* phosphogypsum, cementitious additives, air foam, strength, microstructure.

## 1. INTRODUCTION

Phosphogypsum (PG) is a byproduct from phosphoric acid production by the wet acid method [1, 2] and contains variable impurities like phosphorus oxide, sulphates, fluorides, radioactive elements [3]. It was reported that about five tons of PG is generated with a ton of phosphoric acid product. Around 100 – 280 million tons of PG was released all around the world in 2014, but only 5 % of the total amount was recycled [4 – 6]. In China, it was reported that around 22 million tons of waste PG was produced and the utilization ratio reaches only 2 – 3 % [6]. The unrecycled PG leads to occupation of cultivated land, air pollution and contamination of water resource [7]. Therefore, different recycling methods have been developed such as agricultural fertilizes [8, 9], cement retarder [10], soil stabilization [11, 12] and cementitious binder [13]. Due to the similarity between PG and natural gypsum, PG has been extensively used to replace natural gypsum in building materials such as gypsum blocks or plates [14, 15].

It was reported that cementitious mineral admixtures can decrease water requirement, improve fluidity, adjust setting time and improve mechanical strength and water resistance of gypsum paste. Singh and Garg [15, 16] proposed a cementitious binder by mixing calcined phosphogypsum, fly ash and hydrated lime with a ratio of 40:40:20, which is suitable for masonry mortars, tiles and bricks, etc. Yun Huang [17] developed a cementitious material with compressive strength of 40 MPa by a mixture of 45 % PG, 10 % steel slag, 35 % ground granulated blast-furnace slag and limestone. Escalante-García [18] found that the addition of 30 – 50 % ground granulated blast-furnace slag significantly improves strength and water resistance of gypsum mortar.

To reduce bulk density and enhance heat and sound insulation, different types of lightweight building materials can be manufactured by the addition of air bubbles. Lin Yang [19] presented a non-autoclaved aerated concrete based on PG by adding aluminum powder as air-forming agent. In the 1990s, Jones [20] developed a kind of pre-foamed bubbles to add into cement mortar for manufacturing cellular concrete. K.Ramamurthy and Y.H. Mugahed Amran systematically investigated foam concretes and summarized study progresses in this field [21, 22]. Z Zhang [23] presented a geopolymer foam concrete with fly ash, slag, foam and NaOH solution as the alkali activator. On the other hand, little attention has been paid to prepare foamed gypsum-based lightweight materials.

Therefore, this paper aims to study the modification of PG matrix by incorporating several cementitious additives including fly ash, ground granulate blast-furnace slag and Portland cement. On the other hand, different volume of foam was incorporated to prepare PG based lightweight material. The measured properties include fluidity, setting

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time, mechanical strength, water-resistance and microstructure observation.

## 2. MATERIALS AND METHODS

### 2.1. Materials

Phosphogypsum was from one phosphoric acid company in Guizhou Province of China. It is a light grey powder (as shown in Fig. 1) mainly containing CaSO4·2H2O, phosphorus oxide, fluoride and other impurities.


**Fig. 1.** Visual appearance of PG

The main chemical composition of dehydrated phosphogypsum was determined by X-ray Fluorescence as shown in Table 1 and the size distribution was analyzed by BT-2100 Laser Particle Size Analyzer as shown in Fig. 2.

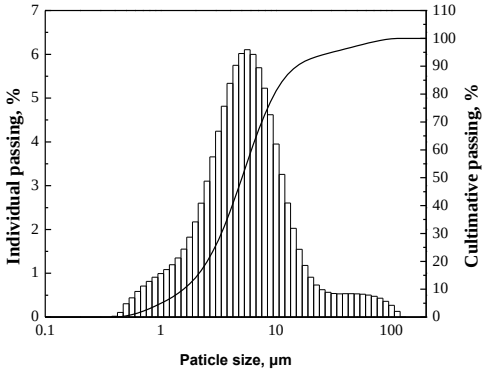



**Fig. 2.** Particle size distribution of PG

It can be found that the particle size of this used phosphogypsum ranges from 0.4 to 100 μm. It contains 81 % of total mass below 10μm and has a mean particle size of 5.063 μm. Fly ash was supplied by Harbin Shuangda Company in Heilongjiang Province of China. Ground granulate blast-furnace slag (SL) was used as another mineral admixture with specific surface area of over 400 $m^2/g$ according to GB/T18046-2008 [24]. Ordinary Portland cement (OPC) with the strength grade of 42.5 MPa according to Chinese standard GB175-2007 was used in this study [25].

The chemical compositions of fly ash (FA), ground granulate blast-furnace slag and OPC are indicated in Table 2. Hydrated lime was also used as an additive with a chemical purity. A polycarboxylate-based superplasticizer (SP) used in this study was supplied by Harbin Qiangshi Company with water reduction range of more than 30 % and solid content of about 40 %. A locally available plant-based foaming agent was diluted by tap water with a ratio of 1:30 and was then used to generate air foam with a density of 30 $kg/m^3$ – 40 $kg/m^3$.

### 2.2. Mixture preparation

PG was dehydrated at temperature of 140℃ for 9 h and then stored in a sealed container for at least 5 days before use. For all mixtures, the water to binder ratio was kept at 0.8. As shown in Table 3, four serials of mixtures were designed. In Serial A, hydrated lime was added by 2 %, 4 %, 6 %, 8 % and 10 % of total binder weight to eliminate the negative effect of impurities in PG and fly ash was added by 18%. Based on experimental results, the dosage of hydrated lime was selected as 6% and then fly ash or ground granulate blast-furnace slag was added to replace PG by different levels of 10 %, 20 %, 30 % and 40 % as shown in Serial B and C. On the other hand, 5 % – 35 % of cement was added to replace PG by volume in Serial D. Finally, the ratio of PG: fly ash: cement: hydrated lime was selected at 49: 20: 25:6, and air foam was added at different volume levels of 10 %, 20 %, 30 %, 40 %, 50 % and 60 %.

Based on the mixture proportions described above, gypsum paste was prepared by a planetary mixer. Prisms with size of 40 mm × 40 mm × 160 mm were then prepared for determining compressive strength, flexural strength, and water resistance. All specimens were cured in a room temperature with molds for the first day. After being demolded, prismatic specimens were kept in a chamber with room temperature (20 ± 2 ℃) and relative humidity of around 80 % until the day of testing.

### 2.3. Measurement methods

The setting time was evaluated according to Chinese standard for gypsum [26]. Fresh paste was cast in a cylinder mould immediately after mixing and then measured the penetration depth of a special steel needle with time. To determine the fluidity of fresh paste, the mixture was cast into a mini-cone mold uniformly.

**Table 1.** Chemical composition of PG, wt.%

| $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $Fe_2O_3$ | BaO | F | Cl |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.077 | 0.059 | 0.471 | 4.129 | 0.865 | 48.618 | 0.063 | 44.891 | 0.179 | 0.207 | 0.335 | 0.018 |

**Table 2.** Chemical composition of cementitious materials, wt.%

| Types | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | MgO | CaO | $SO_3$ | $R_2O$ |
|---|---|---|---|---|---|---|---|
| OPC | 20.86 | 5.47 | 3.94 | 1.73 | 62.23 | 2.66 | 0.48 |
| FA | 58.29 | 21.50 | 4.73 | 1.36 | 7.94 | 0.16 | 6.02 |
| SL | 34.17 | 13.7 | 15.33 | 8.11 | 26.61 | 0.26 | 3.51 |

**Table 3.** Addition dosages of cementitious additives

| Serial | A | B | C | D |
|---|---|---|---|---|
| Hydrated lime, % | 2, 4, 6, 8, 10 | 6 | 6 | 0 |
| FA, % | 20 | 10, 20, 30, 40 | 0 | 0 |
| SL, % | 0 | 0 | 10, 20, 30, 40 | 0 |
| OPC, % | 0 | 0 | 0 | 5, 10, 15, 20, 25, 30, 35 |

Then the mold was lifted vertically and two diameters perpendicular to each other of the plate were measured.

Three-point flexural test was carried out with a loading rate of 1.5 kN/s and the average of three 40 mm × 40 mm × 160 mm specimens for each mixture was reported as flexural strength. Compressive strength was conducted on six broken samples after flexural test and the average of six samples was reported as the tested compressive strength. As required in Chinese standard GB/T 17671, the deviation limit of strength test values is 10 % [27]. In other words, all datum with 10 % deviation from the average value in each group were regarded as invalid samples.

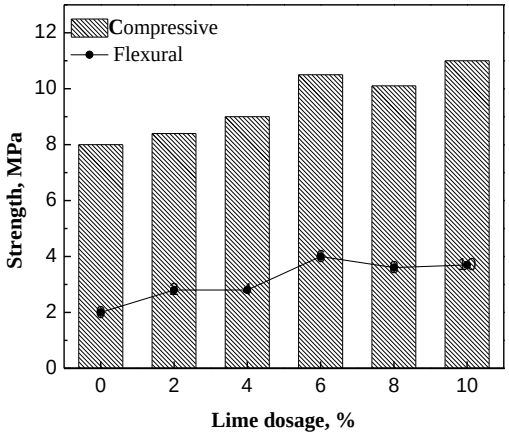
To determine the water resistance performance, compressive strength was firstly carried out on specimens that were oven-dried to constant weight under temperature of 40 ± 4 °C. And then strength measurement was carried out on specimens after immersion in water for 24 hours. The water resistance was determined by the softening coefficient that was defined as the ratio of wet strength to dry strength. The thermal conductivity was measured on dried specimens by using TC3000 heat wire thermal conductivity instrument. The selected samples were oven-dried to constant weight (when the weight variation during 24 h is less than 0.2 %) under temperature of 40 ± 4 ℃. Microstructure measurements were performed by a JEOL SX-4 scanning electron microscope (SEM) on freshly fractured surfaces of selected samples after gold coating treatment. And air bubble or void distribution was evaluated by a Super Depth of Field Microscope (OM,OLYMPUS, DSX500).

## 3. RESULTS AND DISCUSSION

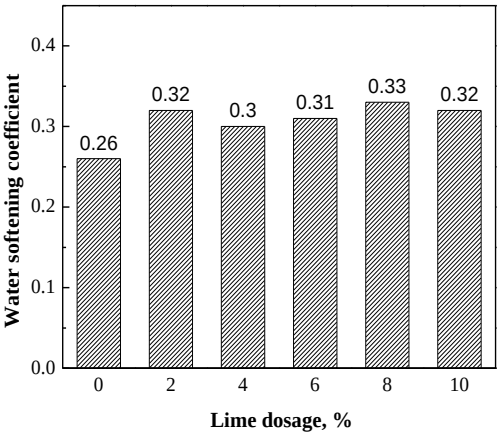
### 3.1. Influence of hydrated lime

Compressive and flexural strengths of PG pastes containing different dosages of hydrated lime are presented in Fig. 3 a. It can be found that compressive and flexural strength increases with the more addition of hydrated lime from 2 % to 6 % and then tends to be steady when the dosage of hydrated lime is increased from 6 % to 10 %. This is due to the facts that lime doesn't have a cementitious ability and acts only as alkali activator and filling particles in PG paste [19]. Therefore, both too high or too low alkalinity is not desirable for mechanical strength of PG paste [17]. Based on the experimental results, the optimum dosage of hydrated lime was selected at 6 % in the following tests.

The softening coefficients of mixtures containing different dosage of hydrated lime are indicated in Fig. 3 b. It is obviously seen that the water softening coefficient fluctuates around 0.3 and the addition of hydrated lime just has a little improvement on the water resistance of PG pastes. Overall, the hydrated lime modified PG specimens performs non-hydraulic feature.



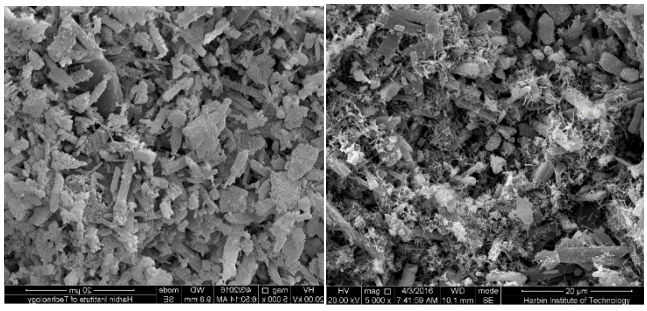

a




b

**Fig. 3.** Influence of hydrated lime on: a – mechanical strength; b – water resistance

Fig. 4 shows SEM images of the control and 6 % hydrated lime samples.



a b

**Fig. 4.** Influence of hydrated lime on microstructure: a – sample without lime; b – sample with 6 % lime

There are some many cylindrical and lamellar gypsum crystals which are crossing and connecting in the control sample, being attributable to the strength formation. It is indicated that tiny acicular ettringite crystals distribute intensively around gypsum crystals and fill voids formed by packing of cylindrical and lamellar gypsum crystals [14]. Therefore, the mechanical strength is improved a little by the addition of hydrated lime. On the other hand, hydrated lime leads to a higher alkalinity in pore solution which is advantageous for the pozzolanic reactivity of fly ash. As a result, the water resistance of hydrated lime added PG specimen is superior to the control one as described above.

## 3.2. Influence of mineral admixtures

The fluidity and setting time of PG paste was measured when different dosage of fly ash or ground granulate blast-furnace slag was incorporated. The testing results are presented in Fig. 5.

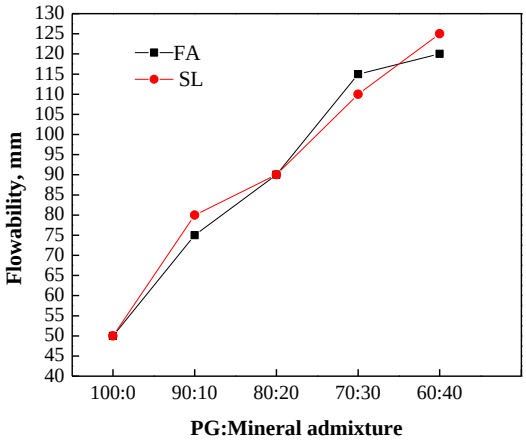



**Fig.5.** Influence of FA and SL on: a – fluidity; b – setting time

It can be seen that the fluidity is increased by the higher dosage of these two mineral admixtures. The flow diameter of PG paste was increased by 50 % and 60 % respectively when 10 % of FA and SL were added. When the dosage of FA and SL were increased to 40 %, the flow diameter exceeded to 120 mm and 125 mm, being 2.4 times and 2.5 times of that of the control one (50 mm). This is due to that mineral admixtures dilute the content of semi-hydrated gypsum in the system and have a ball effect to increase the fluidity of fresh paste.

It is illustrated in Fig. 5 b that the initial setting time is increased by 3 times from 6 min to 18 min while the final setting time is increased by 4 times from 11 min to 43 min when fly ash is added by 40 %. At the same time, the interval between initial and final setting times becomes larger with the increase of FA content. This can be explained by the less amount of $CaSO_4 \cdot 1/2H_2O$ in paste, inducing the less formation of hydrated product $CaSO_4 \cdot 2H_2O$ to build up a crystal network in the paste system.

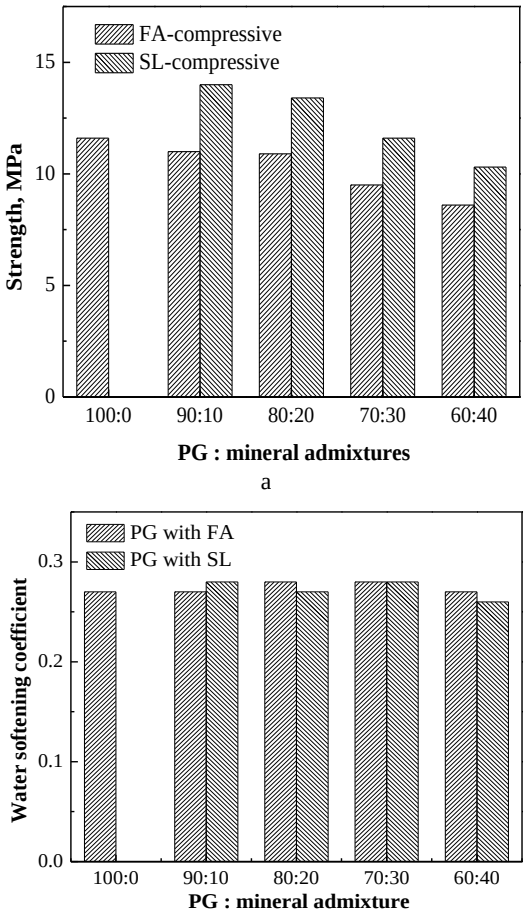
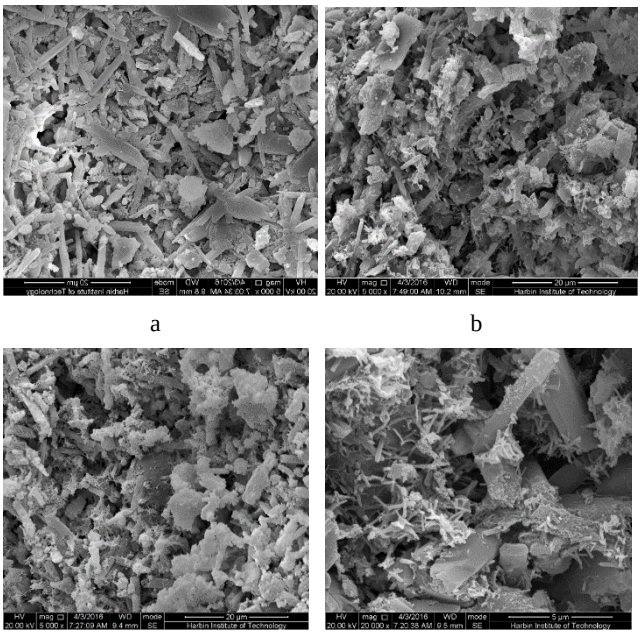



**Fig. 6.** Influence of FA and SL on: a – compressive strength; b – water resistance

The 28-days compressive strength strength of specimens containing different content of FA and SL are shown in Fig. 6 a. It is clearly illustrated that the addition of 10 ~ 40 % fly ash results in the reduction of 5.2 ~ 25.9 % on compressive strength of PG specimens. The addition of less than 20 % fly ash leads to a limited reduction on compressive strength. However, the addition of 10 ~ 30 % SL has an improving effect on compressive strength. The best dosage of SL is 10 %, inducing an increase of 20.7 % compressive strength. This is due to the facts that fly ash has a much lower pozzolanic reactivity than SL. It should be given more attention that some micro cracks formed on the surface of specimens containing SL. It is possibly because of the much more formation of expansive ettringite crystals in pores [28, 29]. Other reasons and preventive methods

should be researched in future. Therefore, SL was not used to prepare foamed PG materials in the later experiments.

It is presented in Fig. 6 b that the water resistance of all specimens fluctuates around 0.27. Therefore, the addition of 10 ~ 40 % fly ash or ground granulate blast-furnace slag has little influence on the water softening performance of PG paste specimens.



a b c d

**Fig. 7.** SEM images of PG paste samples: a – pure PG (magnification of 5000×); b – 10 % SL (magnification of 5000×); c – 20 % FA (magnification of 5000×); d – 20 % FA (magnification of 20000×)

SEM microstructure observation of typical samples are shown in Fig. 7. As expected, $CaSO_4 \cdot 2H_2O$ is the main hydration product of PG paste with needlelike crystal shape. There are some ettringite crystals and C-S-H products that are formed by the reaction of gypsum and mineral admixture. These products are filling the void between $CaSO_4 \cdot 2H_2O$ crystals with bigger size and then influence the mechanical strength. The exact hydration products in different samples should be further studied by XRD and DTA-TG.

## 3.3. Influence of ordinary Portland cement

The influence of OPC on compressive and flexural strength of PG paste is presented in Fig. 8 a. As expected, the higher addition of cement leads to the better mechanical strength. Furthermore, the strength growth from 7 days to 28 days is much increased by the incorporation of OPC, due to the continuous hydration reaction of Portland clinker minerals. Therefore, PG mainly contributes to the early age strength and OPC plays a more important role on long-term strength growth. As for flexural strength, the addition of 10 % OPC increases 7 d and 28 strength by 25 % and 43.8 % respectively, but no significant improvement can be found with the further higher dosage of OPC.

The water softening coefficients of PG specimens containing different dosage of cement are presented in Fig. 8 b. It can be observed that the addition of OPC improves the water resistance and the highest value is 0.48 when 25 % of OPC is used. It is due to the much better stability of hydration products of OPC (AFt crystals and C-S-H gel) than hydrated gypsum.

From SEM image shown in Fig. 9, it can be found that there are visible quantities of C-S-H gel and ettringite formed in OPC added samples. These hydration products fill the pore structure of paste to build up a stronger and denser framework, inducing the higher mechanical strength and better water resistance as described above [30].

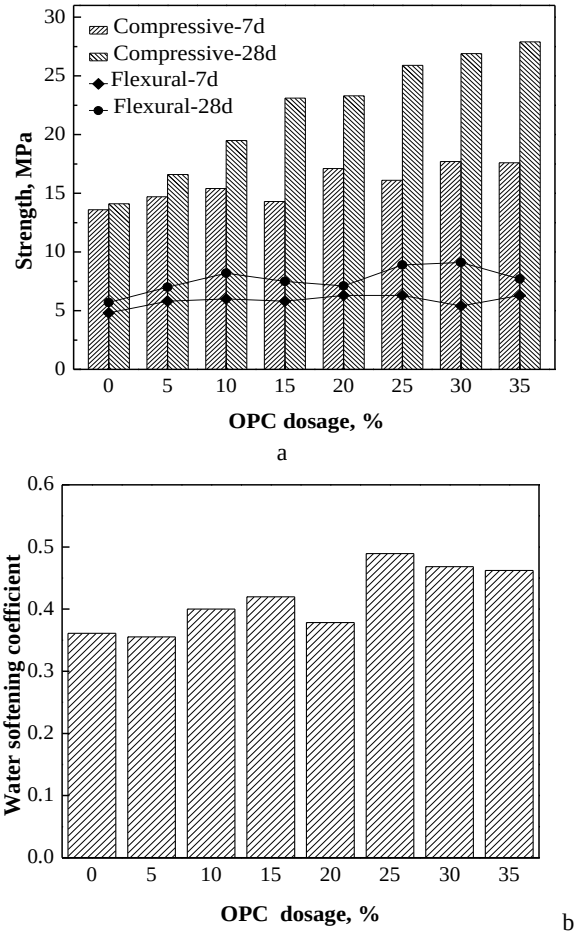
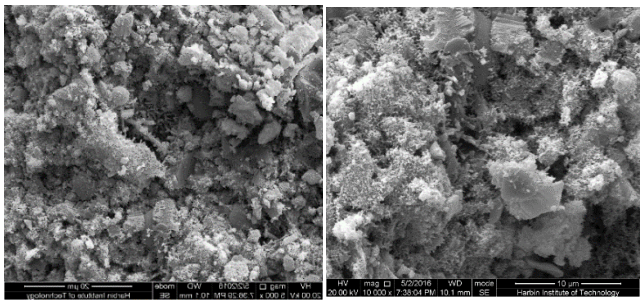



**Fig. 8.** Influence of OPC on properties of PG specimen: a – mechanical strength; b – water resistance



a b

**Fig. 9.** SEM images of PG samples: a – sample with 20 % cement (5000×); b – sample with 20 % cement (10000×)

## 3.4. Preparation of PG lightweight materials

Because fly ash is cheaper and easily accessible than SL in China and the utilization of FA has a more important environment significance. Therefore, 20 % fly ash was used

to replace PG to prepare foamed phoshogypsum lightweight material. On the basis of the above experimental results, OPC and hydrated lime were added by 25 % and 6 % respectively. When the ratio of PG: fly ash: cement: hydrated lime was kept at 49: 20: 25:6, different lightweight gypsum specimens were prepared by adding variable volume of air foam. Compressive strength, bulk density and thermal conductivity of these mixtures were measured as shown in Fig. 10. It can be found that when the compressive strength decreases from 17.9 MPa to 1.7 MPa, the bulk density is reduced from 1404.9 kg/m$^3$ to 521.7 kg/m$^3$ and the thermal conductivity is decreased from 0.4246 W/(m·K) to 0.0724 W/(m·K) when the content of foam increases from 0 % to 60 %.

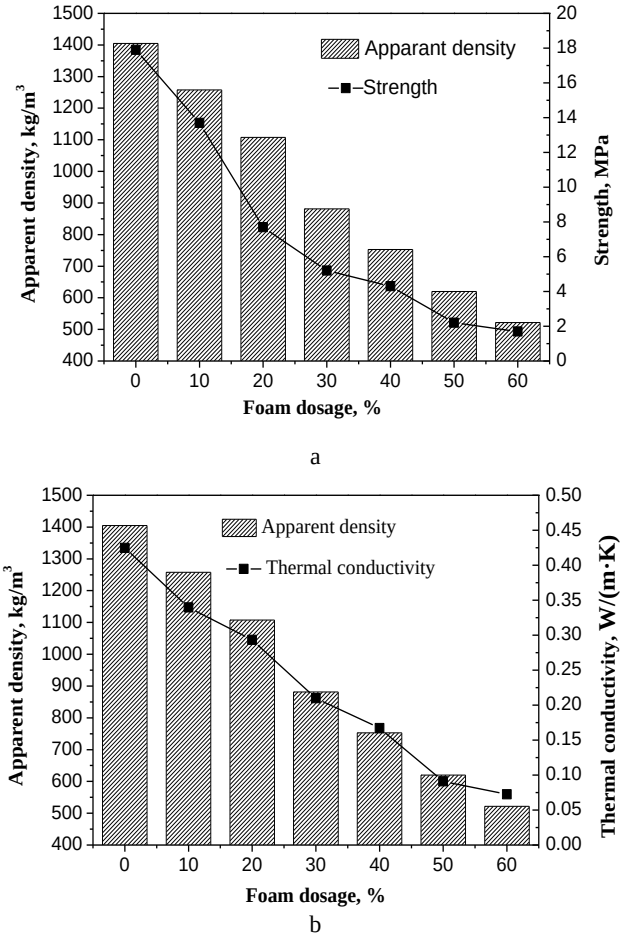



**Fig. 10.** Effect of foam on: a – apparent density and strength; b – apparent density and thermal conductivity

On the other hand, microstructure observation was carried out by optical microscope to characterize void distribution in different samples. As shown in Fig. 11, the total porosity is much lower than the added volume of air foam when the foam content is less than 40 %, being possibly attributed to damage of air foam during mixing. When the foam addition content is increased to 50 % and 60 %, the total porosity is sharply increased and is higher than the expected foam volume fraction, being attributed to the high porosity of gypsum paste matrix. On the other hand, most of air bubbles exists isolated in the observed section, being favorable for good thermal and acoustic insulation.

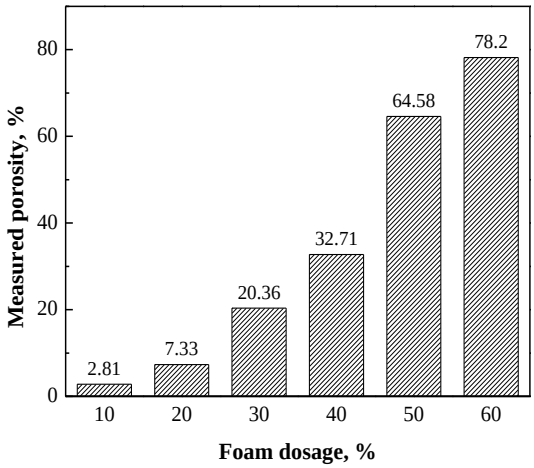



**Fig. 11.** Total porosity with different dosage of foam

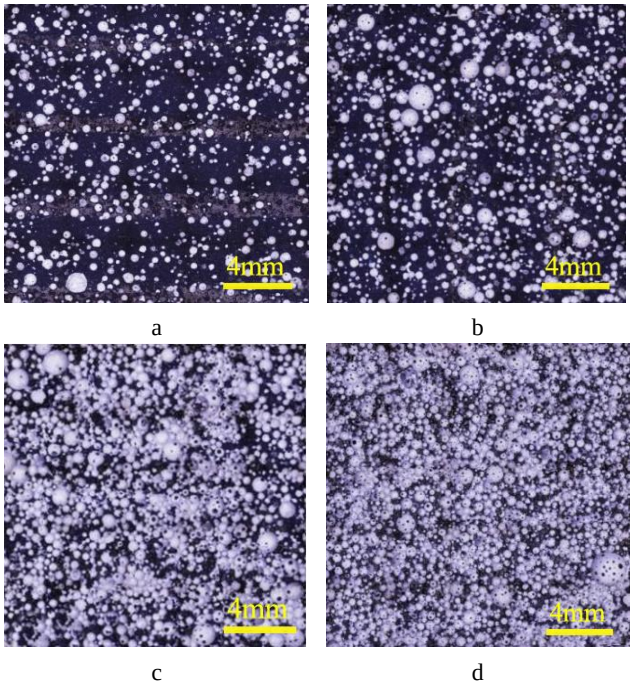



**Fig. 12.** Air bubble distribution for samples containing different foam content: a – 30 %; b – 40 %; c – 50 %; d – 60 %

## 4. CONCLUSIONS

Based on the above experimental results, the following conclusions can be drawn:

1. Addition of hydrated lime can improve mechanical strength and water resistance of PG paste, and the optimal dosage of hydrated lime is 6 %.
2. Higher addition of fly ash or ground granulated blast-furnace slag improves the fluidity and delays the setting time of phosphorus gypsum paste. The addition of 10 ~ 20 % fly ash results in a little reducing influence and 10 % ground granulated blast-furnace slag leads to an increase of 20.7 % for 28 days compressive strength of hardened PG specimen.
3. The higher addition of OPC results in the better mechanical strength and water resistance of PG specimens. The 28 days compressive and flexural

strength reaches 25.9 MPa and 8.9 MPa respectively for the 25 % Portland cement mixture.

4. PG based lightweight building materials can be prepared by the addition of 60 % volume of air foam, with compressive strength of 1.7 MPa, bulk density of 521.7 kg/m3 and thermal conductivity of 0.0724 W/(m·K).
5. PG based lightweight material provides a new recycling method for phosphogypsum waste and the possible application of this material in buildings should be paid more attention.

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