

Effect of Calcination Temperature of Dolomite on Properties of Self-insulation Wall Materials

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Abstract: The environmental pollution problem of Sinochem Chongqing Fuling Chemical Co., Ltd. is serious. Fuling District Party committee and Fuling District government relocated the chemical plant as a whole and started the ecological restoration project. However, the investigation found that soil covering greening failed to fundamentally eliminate the harm caused by phosphorus slag to the environment. Therefore, the resource utilization of phosphorus slag is imperative. The application of dolomite rich phosphorus slag in the construction industry not only improves the environment, but also reduces the production cost of self-insulation wall materials. In this experiment, dolomite rich phosphorus slag was calcined at 600 °C, 700 °C and 800 °C respectively to obtain light calcined dolomite, which was mixed into modified fly ash cement composite mortar to prepare self-insulation wall material. The mechanical properties, water absorption, bulk density and thermal insulation of the specimens were tested after curing for 7 and 28 days respectively. The conclusions are as follows: when the calcination temperature of dolomite rich phosphorus slag is 700 °C, the specimen has the best mechanical properties, the lowest water absorption, the largest bulk density and the best thermal insulation.

Keywords: Phosphate slag, Calcination temperature, Water soaking maintenance, Self-insulating wall materials.

1. Introduction

1.1. Research Background of Dolomite and Self-Insulation Wall Materials

1.1.1. Research Background of Dolomite

The environmental pollution problem of Sinochem Fuling Chongqing Chemical Industry Co., Ltd. is serious, which has a great impact on the surrounding vegetation and water source. Fuling District Committee and District Government relocated the chemical plant as a whole and initiated the ecological restoration project. However, it is found from the investigation that overlaying greening fails to fundamentally eliminate the harm caused by phosphate slag to the environment. Therefore, the resource utilization of phosphate slag is imperative. It is found that the phosphorite slag in tailings mine mainly includes phosphorus-rich gypsum phosphorite slag ($\text{CaSO}_4 \cdot \text{H}_2\text{O}$) and dolomite-rich phosphorite slag ($\text{CaCO}_3 \cdot \text{MgCO}_3$). The dolomite-rich phosphate slag is used in this experiment.

1.1.2. Research Background of Self-Insulation Wall Materials

In the modern era of vigorously advocating energy conservation and environmental protection, building energy conservation has been greatly impacted. In traditional buildings, the insulation wall is built with brick wall and cement, the insulation effect is poor, and residents have to consume more resources to obtain a comfortable living environment; the new construction materials themselves can achieve the effect of energy conservation and insulation, so as to effectively reduce energy consumption. The new building materials can be divided into exterior wall insulation materials, interior wall insulation materials and self-insulation wall materials. The exterior wall insulation materials have high requirements for fire resistance and durability; the interior wall insulation materials occupy the house area and the construction is relatively complex; while the self-insulation wall materials themselves have the

function of heat preservation and heat insulation, and its utility can also solve the shortcomings of exterior wall and interior wall insulation materials in heat preservation.

1.2. Current Research Status of Dolomite and Self-Insulation Wall Materials at Home and Abroad

1.2.1. Current Research Status of Dolomite At Home and Abroad

Lianyungang Jinping Phosphorite was discovered in China as early as the Tongzhi Period of the Qing Dynasty, and was officially mined in 1914. It is the earliest phosphorite discovered and mined in China with the longest history, and is also the first national key project of the five-year plan in China. It is the first large phosphorite in New China. However, after the enterprise announcing bankruptcy, its stacked phosphorite slag has seriously threatened the surrounding environment. Li Mingdong studied the production of bubble mixed light soil by phosphate slag, and developed appropriate methods to consume phosphate slag to a certain extent [1]. In foreign countries, experts and scholars have formed a relatively perfect theoretical system for the problem of phosphate slag pollution of the environment, and its research time is earlier than that in China. In the 1990s, Salah proposed treatment measures for phosphate slag pollution of the environment [2].

1.2.2. Current Research Status of Self-Insulation Wall Materials at Home and Abroad

The building insulation system originated in Europe in the 1960s, and after the first energy crisis broke out in the early 1970s, building insulation began to receive attention and development, when the European building insulation system was the most advanced. At present, the exterior insulation system method of thinning plastering with exterior insulation plate is widely used in Europe. The initial use of exterior insulation systems in Europe was to compensate for the cracks existing in the wall, while it was later found that exterior insulation systems not only solved the wall cracks

problem, but also reduced the building energy consumption and improved the building insulation performance, which is because the composite wall has good insulation performance and heat insulation performance [5]. Serres in France studied a new wall material, COB block, with low LCC, and showed that traditional materials can be used on modern buildings by studying the thermal properties of COB blocks compared with ordinary concrete insulated block walls [8]. At present, the research on new wall materials, especially self-insulation blocks, is increasing in China. In terms of thermal bridge research, research in China has developed relatively late, and Zhao Lingling of Anhui University of Technology studied the thermal performance of self-insulation walls [10].

1.3. Study Purpose and Significance of Dolomite and Self-insulation Wall Materials

The mixture of MgO and CaCO₃ was obtained by light burning of phosphate slag. The mixture was incorporated into the modified fly ash-cement based self-insulation wall material. Without changing the performance of self-insulation wall material, or with little change, the waste was reduced, and the cement dosage was saved to a certain extent. Although some scholars have studied the performance of dolomite-rich phosphate slag calcined at different temperatures incorporated into modified fly ash-cement based self-insulation wall material to form self-insulation wall material, and found that dolomite-rich phosphate slag calcined at different temperatures has a good improvement effect on the performance of modified fly ash-cement based self-insulation wall material, but the research work is relatively few, the relevant research on the mechanical properties and insulation properties of the material is not comprehensive enough, and the relevant research work still needs to be carried out systematically and deeply. Therefore, this study will carry out research on it.

1.4. Research Content of Dolomite and Self-insulation Wall Materials

1.4.1. Experimental Contents

In this experiment, the dolomite-rich phosphate slag was first investigated and collected on-site, and dried, sieved and milled. Then the dolomite-rich slag was heat-treated at 600 °C, 700 °C and 800 °C to obtain light-burned dolomite-rich slag powder, and XRD phase analysis was performed. The formula design was carried out with light-burned dolomite-rich phosphate slag, fly ash, natural medium sand and PO42.5 cement as the main raw materials. Taking K12 as the foaming agent and EPS particles as the insulation medium for the formulation design. Dolomite-modified fly ash-cement based wall insulation materials were prepared by the processes of burdening, chemical foaming, stirring, molding, demoulding and maintenance. The mechanical properties, water absorption rate, volume density and insulation properties of dolomite-rich phosphate slag modified fly ash-cement based self-insulation wall materials cured by water immersion for 7 days and 28 days were examined to study the effects of different calcination temperatures of phosphate slag on the

development rules of mechanical properties, water absorption rate, volume density and insulation properties, as well as the relationship between mechanical properties, water absorption rate, volume density and insulation properties.

2. Experiment

2.1. Experiments Materials and Formula

90 g dolomite-rich phosphate slag (industrial solid waste produced by Sinochem Chongqing Fuling Chemical Industry Enterprise), 450 g PO42.5R grade common Portland cement (Huaxin Cement Chongqing Fuling Co., Ltd.), 1,350 g natural river sand (medium sand), 225 mL common tap water, 45 g fly ash (10 wt%, grade II produced by Chongqing Zhongxian Power Plant), 200 mL EPS particles and 2.25 g K12 foaming agent (0.5 wt%, sodium dodecyl sulfate).

2.2. Experimental Equipment and Instruments

- (1) 300 mm × 300 mm × 30 mm cement sand-binder test mould;
- (2) 40 mm × 40 mm × 160 mm cement sand-binder test mould;
- (3) IOS.679 model cement sand-binder mixer;
- (4) DKZ.5000 electric folding tester (Shenyang Changcheng Electromechanical Equipment Factory);
- (5) ZT.96 model cement sand-binder shake stand (Wuxi Huaxi Building Materials Laboratory Instrument Co., Ltd.);
- (6) DYE.2000 model electro-hydraulic pressure tester;
- (7) USA.HZ&HUAZHI electronic scale (accuracy of 0.01g);
- (8) National standard inspection sieves (60 mesh, 100 mesh, 120 mesh and 200 mesh, respectively);
- (9) DHG-9245A electrothermal oven (the temperature can reach 230°C, the control accuracy is ± 3°C, with temperature automatic controller);
- (10) SK.G12133 degree energy-saving box electric furnace (1600°C energy-saving box electric furnace).

$$c_2 = a_2 + b_2. \quad (1)$$

3. Study Results and Analysis

In this experiment, the blank sample number was K, the sample number of sample with 20 wt% uncalcined dolomite-rich phosphate slag was B0, the sample number of sample with 20 wt% calcined at 600 °C was B600, the sample number of sample with 20 wt% calcined at 700 °C was B700, and the sample number of sample with 20 wt% calcined at 800 °C was B800.

3.1. Effect of Calcination Temperature on The Phase Composition of Dolomite

The phosphate slag was calcined at the same calcination time and different calcination temperatures. After calcination, the phosphate slag calcination products were manually grinded in an agate sand-binder to the required fineness, and the grinded components were analyzed by X-ray diffraction, and the results were shown as Figure 1.

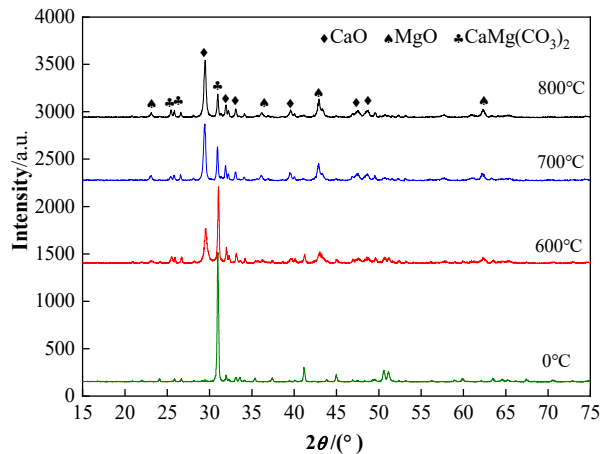


Figure 1. Dolomite with different calcination temperatures

It can be seen from Figure 3 that when the dolomite-rich phosphate slag was not calcined, the dolomite-rich phosphate slag almost did not decompose with the increase of diffraction angle, and its main components were $MgCO_3$ and $CaCO_3$; when the calcination temperature of the dolomite-rich phosphate slag was $600\text{ }^\circ\text{C}$ and the holding time was 3 h, the dolomite-rich phosphate slag only decomposed a small part of the dolomite with the increase of diffraction angle, and the main components of the calcination product were undecomposed dolomite, and its main components were $MgCO_3$, $CaCO_3$ and MgO generated by the calcination decomposition of a small part of dolomite-rich phosphate slag; when the calcination temperature of the dolomite-rich phosphate slag was $700\text{ }^\circ\text{C}$ and the holding time was 3 h, the dolomite-rich phosphate slag decomposed with the increase

of diffraction angle, and most of the calcined products were MgO and $CaCO_3$;

3.2. Effect of Calcination Temperature of Dolomite on Physical Properties of Self-insulation Wall Materials

3.2.1. Volume Density Analysis

In this experiment, dolomite-rich phosphate slag (20wt%) calcined at different temperatures was incorporated into modified fly ash-cement based self-insulation wall material to make specimens, and after 7 days and 28 days of water immersion curing, the volume density experimental test was performed. The volume density results were analyzed as shown in Figure 2:

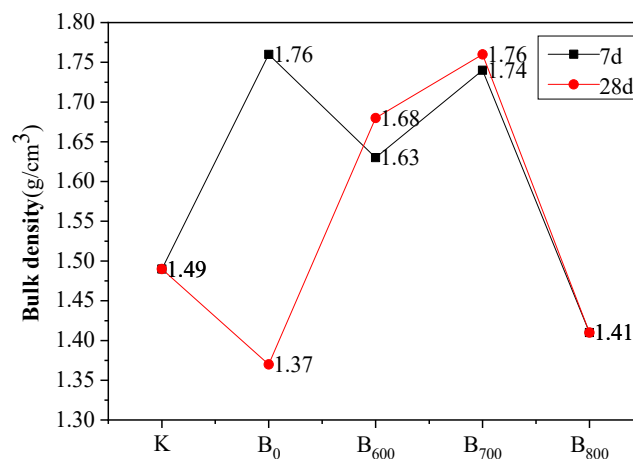


Figure 2. Average volume density

Seen from Figure 2, K specimen was the control group, and the volume density of the specimen did not change with the increase of curing age due to the absence of dolomite-rich phosphate slag powder, and its average volume density was 1.49 g/cm^3 at 7 days and 28 days of curing under water immersion conditions; the volume density of the other B₀, B₆₀₀, B₇₀₀ and B₈₀₀ specimens changed to some extent during curing. The incorporation of dolomite-rich phosphate slag into modified fly ash-cement based self-insulation wall

materials changed the volume density of the specimen to a certain extent. Since modified fly ash and dolomite-rich phosphate slag had many spherical glass beads, it reduced the interior friction resistance of dolomite-modified fly ash-cement based self-insulation wall materials and reduced the water consumption to a certain extent; in addition, the macropore ($>100\text{nm}$) content of dolomite-modified fly ash-cement based self-insulation wall materials after doped dolomite-rich phosphate slag was reduced, and the capillary

pore content was increased, indicating that dolomite-rich phosphate slag could replace the water in the large pores of cement, with the pores gradually filled with modified fly ash and dolomite-rich phosphate slag, the displaced water content was reduced, and the adsorbed water volume of modified fly ash and dolomite-rich phosphate slag was increased.

3.2.2. Mechanical Performance Analysis

In this experiment, the fracture-compression resistance

strength test of dolomite-modified fly ash-cement based self-insulation wall materials was experimentally tested after 7 days and 28 days of water immersion curing of dolomite-rich phosphate slag calcined at different temperatures (20 wt%) incorporated into dolomite-modified fly ash-cement based self-insulation wall materials, and the fracture-compression resistance strength results of dolomite-modified fly ash-cement based self-insulation wall materials were analyzed as shown in Figure 3 and Figure 4:

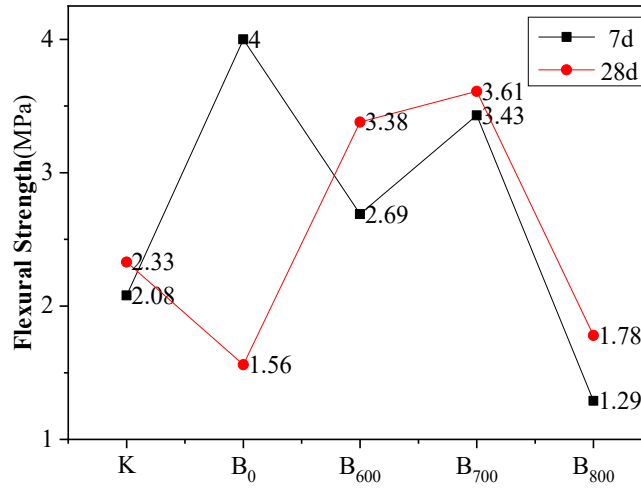


Figure 3. The fracture resistance strength of test piece

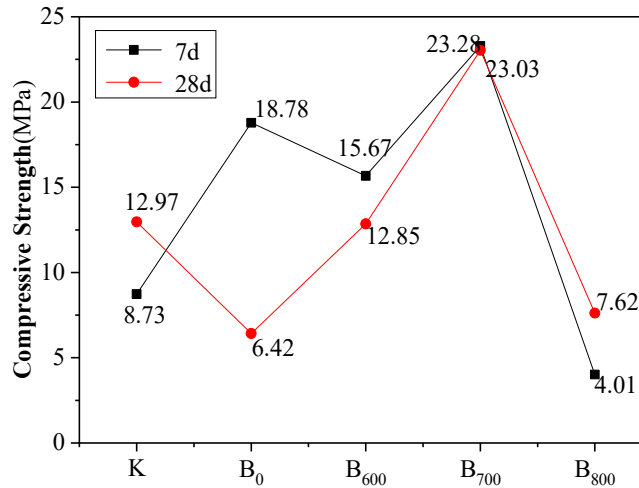


Figure 4. The compression resistance strength of test piece

Seen from Figures 3 and 4, the K specimen was the control group, and the fracture-compression resistance of the specimen increased with the increase of curing age due to the absence of dolomite-rich phosphate slag powder, and its fracture-compression resistance was 2.08 MPa and 2.33 MPa under water immersion conditions for 7 days and 28 days, respectively; seen from the B₀, B₆₀₀, B₇₀₀, and B₈₀₀ groups of specimens in Figure 3. with the change of calcination temperature of dolomite-rich phosphate slag, the fracture-compression resistance of the specimen gradually increased, but the fracture-compression resistance of the dolomite-rich phosphate slag decreased after the calcination temperature

exceeded a certain value. Due to the different calcination temperature of dolomite-rich phosphate slag, there were differences in its products, which led to differences in the amount of MgO-MgSO₄-H₂O gels generated by calcination modification of dolomite-rich phosphate slag and ternary reaction of modified fly ash in cement, thus causing several contradictory effects. First, these gels increased the solid volume and produced extrusion to the surrounding area, thus forming a dense transition zone to improve the weak interface area, and finally changed the fracture-compression resistance strength to varying degrees. Second, solid particles such as CaCO₃ in dolomite-rich phosphate slag that were not

involved in the hydration reaction could be considered as aggregates, and dolomite-rich phosphate slag and modified fly ash were later hydrated to form hydration products, which increased the interfacial bond between dolomite-modified fly ash-cement based self-insulation wall materials and aggregates, thereby improving the fracture-compression resistance strength of the specimen. Third, the incorporation of dolomite-rich phosphate slag and modified fly ash into dolomite-modified fly ash-cement based self-insulation wall materials correspondingly diluted the MgO content per unit mass of cement base, reduced the probability of contact between Mg²⁺ and SO₄²⁻ in the solution and MgO hydration layer, and thus reduced the fracture-compression resistance strength per unit volume.

In summary, for the water-soaked cured specimens, the

calcination temperature of dolomite-rich phosphate slag is 700 °C as the best, when the fracture-compression resistance strength of the specimens is the best.

3.2.3. Analysis of Water Absorption

In this experiment, after 7 days and 28 days of water immersion curing of the specimen mixed with dolomite-rich phosphate slag calcined at different temperatures (20 wt%) into the modified fly ash-cement based self-insulation wall material, the specimen was removed, the surface moisture of the specimen was wiped off with the wrenched cloth, its mass was weighed, and the water absorption rate was experimentally tested. The water absorption rate experimental results were analyzed as shown in Figure 5:

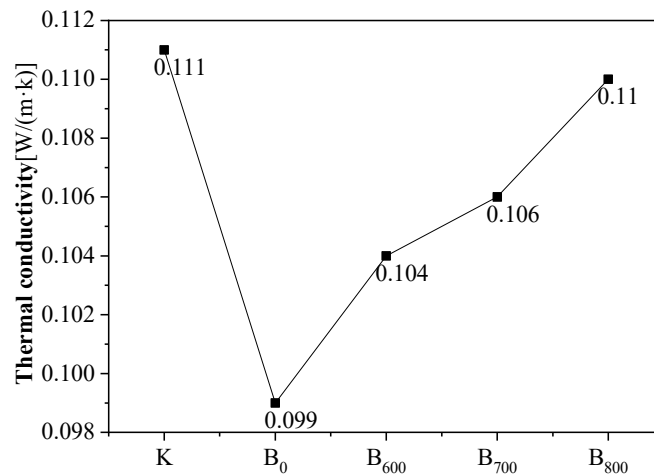


Figure 5. Water absorption rate

Seen from Figure 5, the K specimen was the control group, and the water absorption rate of the specimen increased with the increase of curing age due to the absence of dolomite-rich phosphate slag powder, and its water absorption rate was 10.23% and 11.05% at 7 days and 28 days of curing under water immersion conditions, respectively; seen from the B₀, B₆₀₀, B₇₀₀, and B₈₀₀ specimens in Figure 5, with the change of calcination temperature of dolomite-rich phosphate slag, the water absorption rate of the specimen gradually increased when dolomite-rich phosphate slag was incorporated into the modified fly ash-cement based self-insulation wall material, but the water absorption rate of the dolomite-rich phosphate slag appeared an extreme value after the calcination temperature exceeded a certain value. K12 and EPS particles were added to all the experimental specimens, so that a large number of pores were formed in the specimens. When the specimens were in contact with moist air or solution, the free state water molecules continuously entered the interior of the specimens through diffusion, capillarity and surface adsorption; when the specimens were added with treated dolomite-rich phosphorite residues, the moisture formed a very strong affinity with the hydrophilic groups in the dolomite to make the expansion of the specimens undergo subtle deformation. The more pores of the material, the higher

the water absorption rate.

In summary, for the samples cured by water immersion, the addition of dolomite-rich phosphate slag remained unchanged (20 wt%) and the calcination temperature was 700 °C as the best. At this time, the ternary reaction generated by the addition of dolomite-rich phosphate slag into the modified fly ash-cement based self-insulation wall material generated the most MgO-MgSO₄-H₂O gel, and the water absorption rate of the samples was low, which was more in line with the performance requirements of self-insulation wall material.

3.3. Thermal Insulation Performance Analysis of Dolomite Calcination Temperature on Self-insulation Wall Materials

In this experiment, the 300mm×300mm×30mm thin plate specimens calcined at different temperatures with dolomite-rich phosphate slag (20 wt%) incorporated into the modified fly ash-cement base were slowly and carefully removed from the water tank after 28 days of water immersion curing, and after drying, the thermal conductivity experimental test was performed, and the thermal conductivity results were analyzed as follows:

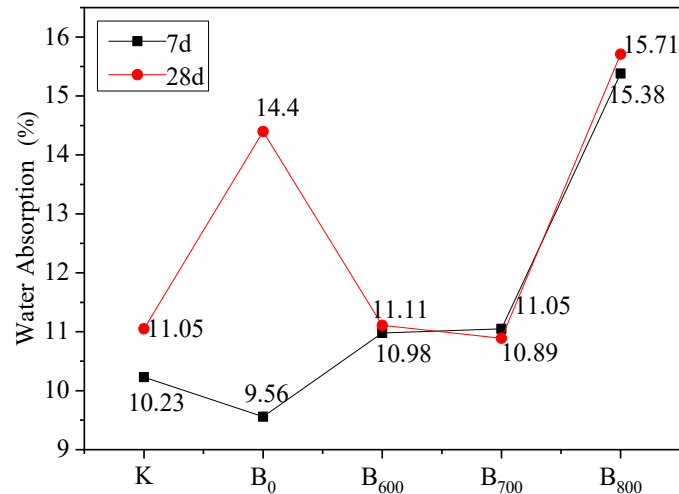


Figure 6. Thermal conductivity

Seen from Figure 6, K specimen was the control group, no dolomite-rich phosphate slag powder was added, the specimen was cured for 28 days under water immersion conditions, the mean value of thermal conductivity λ was the largest with 0.111 W/(m·k); the mean value of thermal conductivity λ of the other B₀, B₆₀₀, B₇₀₀ and B₈₀₀ specimens changed to some extent during the curing process. From the above analysis, it can be concluded that the thermal conductivity changes of these specimens are caused by dolomite-rich phosphate slag. Because the test sample is added with K12 foaming agent, EPS particles and fly ash under the same conditions form a hollow hole, the hollow sample has a higher thermal resistance and good insulation performance due to the small thermal conductivity of the air layer. Appropriate increase of the porosity of the specimen is beneficial to improve the thermal insulation performance of self-insulation wall materials, but the porosity cannot always increase. According to the above, the calcination temperature of dolomite-rich phosphate slag remains unchanged (20wt%) calcination temperature at 700°C is the optimal calcination temperature of dolomite rich phosphate slag in this experiment.

4. Conclusion

Through the analysis of experimental data and line diagram, the effect of calcination temperature of dolomite-rich phosphate slag on the performance of self-insulation wall materials was studied. The following four conclusions are drawn:

(1) The molecular ratio of dolomite MgO and CaO in phosphate slag is about 1:1, and the thermal decomposition process can be divided into two steps. By controlling the calcination temperature of dolomite-rich phosphate slag, it can be seen that the highest activity of MgO and CaCO₃ is obtained when the calcination temperature is 700 °C. With the increase of temperature, MgO grains form agglomerated structures and the activity decreases.

(2) Under the conditions of water-sand-binder ratio 1:2, sand-binder ratio 1:3 and dolomite-rich phosphate slag addition of 20 wt%, the mechanical properties of dolomite-modified fly ash-cement based self-insulation wall materials

increase with the increase of water immersion curing age, and reach the maximum when the calcination temperature of dolomite-rich phosphate slag is 700 °C, here the flexural resistance strength is 3.61MPa and the compression resistance strength is 23.28 MPa.

(3) When the calcination temperature of dolomite-rich phosphate slag is 700 °C and the curing time is 28 days, the volume density of dolomite-modified fly ash-cement based self-insulation wall materials is the largest and the water absorption rate is the lowest, here the volume density is 1.76 g/cm³ and the water absorption rate is 10.89%.

(4) The thermal conductivity of dolomite-modified fly ash-cement based self-insulation wall material increases with the increase of calcination temperature of dolomite-rich phosphate slag, and the thermal conductivity of self-insulation wall material is the lowest when uncalcined dolomite is used, here the thermal conductivity is 0.099 W/(m·k).

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