



Mechanical properties and microstructural analysis of slag based cementitious binder with calcined dolomite as an activator



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HIGHLIGHTS

- Calcined dolomite at complete decarbonation temperature (900 °C) potentially used as a slag activator.
- Optimum mixture of 20 wt% dolomite activated slag has a 28-day compressive strength of 30.71 MPa.
- Dynamic moduli were determined by means of non-destructive resonant frequency and damping analysis (RFDA).
- The main hydration products are C-S-H, portlandite, quartz, calcite, and magnesium calcite.

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ABSTRACT

Blast furnace slag (BFS) incorporating with natural dolomite calcined at temperature 900 °C for producing the no-cement alkali-activated cementitious binder was investigated. Test results indicate that the hardened paste specimen with 20 wt% dolomite has the highest compressive strength of 30.71 MPa at age of 28 days. The addition of calcined dolomite has an apparent effect on increasing the thermal conductivity of hardened paste specimen at 28 days by about 7.03, 12.79, 22.41, and 28.77% with the added amount of calcined dolomite of 10, 20, 30, and 40 wt% and on the increasing dynamic Young's properties of the specimens with calcined dolomite of 10, 20, 30, and 40 wt% at 28 days by 40.68, 55.07, 61.32, and 70.17% than the pure slag paste. The X-ray diffraction (XRD) indicated the main hydration product of pure slag paste was only the calcium silicate hydrate (C-S-H). However, the hydration product of slag-dolomite paste were the C-S-H, portlandite, quartz, calcite, and magnesium calcite, together with the appearance of quartz in the specimen with the calcined dolomite more than 20 wt%, which is a major factor for the increased compressive strength.

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1. Introduction

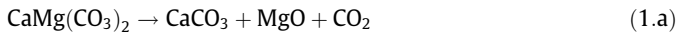
Concrete as construction materials made with hydraulic binders based on the utilization of Portland cement has become worldwide popular since its development over 175 years ago [1]. The production of Portland cement produces a significant amount of carbon dioxide (CO₂) emission that harms the environment, with approximately 0.87 kg of CO₂ for every kg of cement produced [2]. Many researchers have focused on increasing the usage of pozzolanic materials from industrial by-product as a partial replacement of Portland cement to develop alternative binders for concrete. On the contrary, the alkali-activated binders are potential to be an alternative to the Portland cement because of their high strength, durability, and low environmental impact, being made by mixing

solid pozzolanic materials such as fly ash, blast furnace slag, or metakaolin with an alkaline activating solution [1]. Without an activator, the blast furnace slag (BFS) could still react with water at a very slow rate of hydration, but can achieve higher compressive strength once the alkaline activator is used [3]. Sodium hydroxide (NaOH), sodium silicate (Na₂SiO₃), and sodium carbonate (Na₂CO₃) are the most common alkalis used as activators. Although those activators are widely available, economical, and effective to produce a better performance of alkali-activated binders those chemicals have a high level of toxicity. This has led to researchers to find alternative activator from natural resources that have a lower toxicity level. Ideal dolomite (CaMg(CO₃)₂) is a naturally occurring substance formed from a sedimentary rock as a massive layer on the ancient rock with abundant reserves around the world, low cost, less toxicity, and environmental friendliness [4]. It is an end-member mineral consisting of alternate layers of calcite (CaCO₃) and magnesite (MgCO₃) [4–6]. Dolomite is a double

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carbonate of Ca and Mg and theoretically, it crystallizes in the rhombohedral unit cell [7]. The calcined dolomite is important in the industries. It serves as a flux in metallurgy, a source of magnesium in glass and ceramic manufacturing industries as well as construction materials [5,7]. The thermal decomposition of dolomite is divided into two stages. The formations of calcite and magnesium oxide (MgO) takes place at a lower temperature (Eq. (1.a)), whereas the decomposition of calcite takes place at a higher temperature to form calcium oxide (CaO) (Eq. (1.b)) [8,9].



The calcination of dolomite at the high temperatures forms CaO and MgO, both which are alkaline earth activator. Either chemical oxide could serve as an effective BFS activator [10,11]. Gu et al. [12] had verified that the natural dolomite calcined at 800 °C and 1000 °C in air was a potential activator for ground granulated blast furnace slag. The XRD patterns of selected dolomite activated slag samples revealed the presence of C-S-H and hydrotalcite-like phases in all samples. The durability tests showed that those slags activated by dolomite calcined at 800 °C were more resistant to Na₂SO₄ attack than those of slag activated by Portland cement, and those slags activated by calcined dolomites were more resistant to MgSO₄ [8]. Jauffret et al. [13] studied the calcined dolomite as a replacement of Portland cement, which showed that the half-burnt dolomite, i.e., dolomites being calcined 800 °C in 30 min, could preserve the 28 days compressive strength of cement paste with 20–25 wt% addition of half-burnt dolomite and induce even higher maximum compressive strength with lower substitution dolomite around 10–15 wt%. Yang et al. [14] studied the light-burnt dolomite, a dolomites being calcined at 900–1000 °C as an addition to Portland blast furnace slag cement, which showed that the addition of light-burnt dolomite improved the 28-day compressive strength by 22–24% and delayed the initial setting time. Peng et al. [15] studied the alkali-activated cement synthesized by calcining bentonite with dolomite and Na₂CO₃. The alkali-activated cement pastes cured at 80 °C reached a 28-day compressive strength of 38.3 MPa, but the strengths of all those cured at room temperature were very low. Although these mentioned literatures have showed that the natural calcined dolomite used as a potential activator is feasible, some important characteristics of resulting slag based cementitious binder with calcined dolomite, such as the optimal addition, thermal properties, microstructural hydration products, etc., still lack of studies.

In this study, the slag-dolomite paste specimens produced by the BFS activated by calcined dolomite at different amounts from 0 to 40 wt% to determine the optimum addition with respect to the development of compressive strength were investigated. In addition, associated physical properties were explored, such as the workability, setting time, ultrasonic pulse velocity (UPV), thermal conductivity, and dynamic properties by impulse excitation. The microstructural analyses using the thermogravimetric analysis/differential thermal analysis (TGA/DTA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), X-ray diffraction (XRD) on both the raw materials and the hydration products of the slag-dolomite paste were also examined.

2. Experimental program

2.1. Materials and mix proportions

2.1.1. Blast furnace slag (BFS) powder

The white blast furnace slag (BFS) powder with Blaine fineness of 6000 cm²/g used in this research was supplied by CHC Resources Corporation, Kaohsiung, Taiwan, as shown in Fig. 1(a). The chemical composition of BFS powder is shown in

Table 1. From the microstructure examination, the shape of the BFS powder particle is irregular, as shown in Fig. 2(a).

2.1.2. Raw and calcined dolomite powders

The raw dolomite powder used in this research was supplied by Taimax Material Co. Ltd., New Taipei City, Taiwan, with the chemical composition shown in Table 1. The calcined dolomite was obtained through laboratory calcination by heating up the raw dolomite powder in a high-temperature electric furnace, in which the raw dolomite powder, approximately 150 g, was placed in the individual clay crucibles, heated at a rate of 5 ± 0.2 °C/min from room temperature in stagnant air to the desired temperature at 900 °C and maintained for 60 min. After the calcined dolomite powder cooled itself down to the room temperature, it was ground until all passed through the 150-µm sieve. The color of the raw dolomite was pure white, but the calcined dolomite was light brown, as shown in Fig. 1(b) and (c), respectively. The micrographs of dolomite powders before and after calcination are shown in Fig. 2(b) and (c), respectively, which reveal that the shape of raw dolomite powder was irregular before calcination and particles were stuck together after calcination, resulting in the enlarged particles with the fractures and porous at the surface of the calcined dolomite. To explain this phenomenon, Ngamcharussrivichai et al. [4] mentioned that through micrographic investigation the fractures appearing on the surface of the calcined dolomite at 900 °C was due to a severe volume reduction of unit cell after the complete decarbonation. Sulistiyono et al. [16] reported that the porous surface of calcined dolomite was caused by CO₂ released from dolomite grain and that such calcination reaction occurred not only on the surface of dolomite but also in all part of dolomite. Particle size analyzer (Mastersizer 2000) with calculation range from 0.040–2000 µm was used for determining particle size distribution of both raw and calcined dolomite, as shown in Fig. 3. Particle size distributions are found at the approximate range of 0.136–120.226 µm for raw dolomite and 0.275–275 µm for calcined dolomite. The mean particle sizes (\bar{x}) of the raw and calcined dolomite are 28.689 and 20.069 µm, respectively.

2.1.3. Mixture proportions

Mix proportions of the slag-dolomite paste were calculated based on the volume method with the concept that the total volume of the mixture was unity. The total mixing water requirement (W_w) in kg/m³ was calculated with Eq. (2)

$$W_w = \frac{0.4 \cdot \rho_w \cdot \rho_s \cdot \rho_d}{\beta \cdot \rho_w \cdot \rho_s + \alpha \cdot \rho_w \cdot \rho_d + 0.4 \cdot \rho_s \cdot \rho_d} \quad (2)$$

where α and β were the weight percentage (wt%) of slag and dolomite, and ρ_w , ρ_s , and ρ_d were the density (kg/m³) of the water, BFS powder, and calcined dolomite powder, respectively. In this study, the water-to-binder ratio was fixed as 0.40. The mixture proportions of slag-dolomite pastes are presented in Table 2. The BFS and calcined dolomite powder in the slag-dolomite paste mixture are denoted as S and D, respectively, and the following number represents the weight percentage of materials. For example, the mixture S70D30 has $\alpha = W_s/(W_s + W_d) = 70$ wt% and $\beta = W_d/(W_s + W_d) = 30$ wt%, where W_s and W_d are the weight of BFS and calcined dolomite powder in kg/m³, respectively.

2.2. Specimen preparation and test methods

2.2.1. Specimens, mixing procedure, and curing condition

The 50-mm cubic specimens were used for determining the development of compressive strength, whereas the cylindrical specimen of 50 mm in diameter and 100 mm in height were used in the non-destructive tests such as ultrasonic pulse velocity and thermal conductivity. The rectangular specimens of 100 × 50 × 15 mm were used in the dynamic moduli measurements with resonant frequency and damping analysis (RFDA).

Mixing procedure for slag-dolomite pastes was illustrated as follows. Firstly all BFS and calcined dolomite powders were placed in a steel container and mixed for 1 min at slow speed to allow both powders evenly mixed, then the water was added gradually into the mixing container for mixing for additional 2 min, and the mixer was stopped for 15 s, during which any sticking paste on the sides of the container was scrapped down. The mixer was continuously running at slow speed for 2 min until the fresh paste evenly mixed, and then running for 1 min at medium speed. After the fresh paste mixture was poured into the mold and tamped to decrease the air bubble, it was covered by the clinging wrap to prevent the loss of water. The specimens were demolded after 24 h, except for those with pure slag paste removed from molds on the third day, because at the early age the specimens were not strong enough for demolding. The pure slag and slag-dolomite paste specimens were cured in the saturated lime water at temperature of 25 ± 2 °C after demolding.

2.2.2. Testing of physical and chemical properties of materials and specimens

The densities of BFS, raw dolomite, and calcined dolomite powder were determined in accordance with ASTM C188-15 [17]. Both raw and calcined dolomites were scanned by X-ray diffraction (XRD) to determine the phases. The changes of physical and chemical properties of raw dolomite powder after heating at a constant rate were characterized by the thermogravimetric analysis/differential

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