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Short communication

Preparation of specific gypsum with advanced hardness and bending strength by a novel In-situ Loading-Hydration Process

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ABSTRACT

An interesting approach named as "In-situ Loading-Hydration Process" is proposed and demonstrated to develop a new type of compact gypsum product with load-bearing ability, benefited from excellent hardness and bending strength. In detail, bassanite ($CaSO_4 \cdot 0.5H_2O$) is press-formed in a mold and in-situ hydrated into gypsum ($CaSO_4 \cdot 2H_2O$) under the control of loading-pressure. The maximum hardness of 1.47 GPa and bending strength of 17.8 MPa are achieved at the loading pressures of 50 and 20 MPa, respectively. These outstanding mechanical properties of the processed gypsums are mainly attributed to the low-level porosities and interlocking crystal microstructures.

1. Introduction

Gypsum ($CaSO_4 \cdot 2H_2O$) is one of the oldest inorganic materials and has been widely used in buildings and constructions [1–5]. Conventional gypsum products are generally produced by a casting process. Bassanite ("plaster of Paris", calcium sulfate hemihydrate, $CaSO_4 \cdot 0.5H_2O$) is mixed with water, casted and then hydrated into gypsum (calcium sulfate dihydrate, $CaSO_4 \cdot 2H_2O$) under the natural condition [6–9]. Nevertheless, considerable pores form inside the gypsum after the excess water evaporates and result in a high porosity (generally 20–40%), which adversely affects the mechanical performance of gypsum products [10–14]. A lot of studies have been carried out to improve the mechanical properties of the gypsum product. For example, inorganic or organic agents were added to reinforce the structure, and the nano-hemigypsum was used to control the crystal growth. Of particular interest, a method of forcing water out of the molded green block by a pressure immediately after casting, was used for reducing the porosity and enhancing the mechanical performance of gypsum. However, in spite of these perfections, the hardness and bending strength of as-obtained gypsums are still not higher than 0.6 GPa and 3 MPa with the corresponding density of about 1.6 g/cm^3 [15–22].

The conventional casted gypsum is produced into plasterboards and mainly used to construct compartment walls, in view of their light-weight and fireproofing features [1,7,8,12]. Nevertheless, if a new method can be designed and developed to greatly improve such properties as compactness, hardness and bending strength, the application scope of gypsum will be significantly expanded, e.g. using as the load-bearing wall or pole.

For this purpose, a novel process named as "In-situ Loading-Hydration Process" was proposed in this work for preparing gypsum products with greatly improved mechanical performance. Bassanite ($CaSO_4 \cdot 0.5H_2O$) filled in a mold was press-formed and in-situ hydrated into the gypsum block ($CaSO_4 \cdot 2H_2O$) under the control of loading–pressing. The hardness and bending strength of gypsum blocks prepared at different loading-pressures were investigated. The crystalline phase, microstructure and porosity were measured to reveal the mechanism controlling the mechanical performance.

2. Materials and methods

The procedures of "In-situ Loading-Hydration Process" are schematically shown in Fig. 1 and detailed as follows:

(1) The raw gypsum powder ($CaSO_4 \cdot 2H_2O$) was dried and dehydrated into the bassanite powder ($CaSO_4 \cdot 0.5H_2O$) at 150 °C; (2) 25 g bassanite was filled into a mold of 35 mm in width and 60 mm in length, laid on WAW1000D Microcomputer-controlled electro-hydraulic servo universal testing machine (UTM), and press-formed into a compacted block with the thickness of 5–10 mm at a loading pressure of 1, 10, 20, 30, 40, 50 or 60 MPa; (3) The crucial operation: the loading-pressure was maintained at the destined value controlled by UTM. Along the gap between the press head and the mold, water was added drop by drop at a rate of 10 drops per minute into the compacted block for 3 h, in which course, bassanite dissolved continuously into water and then new gypsum crystals precipitated in situ to form a gypsum block with high compactness; (4) The pressure was unloaded and the as-obtained block was naturally dried for 7 days to become the final product.

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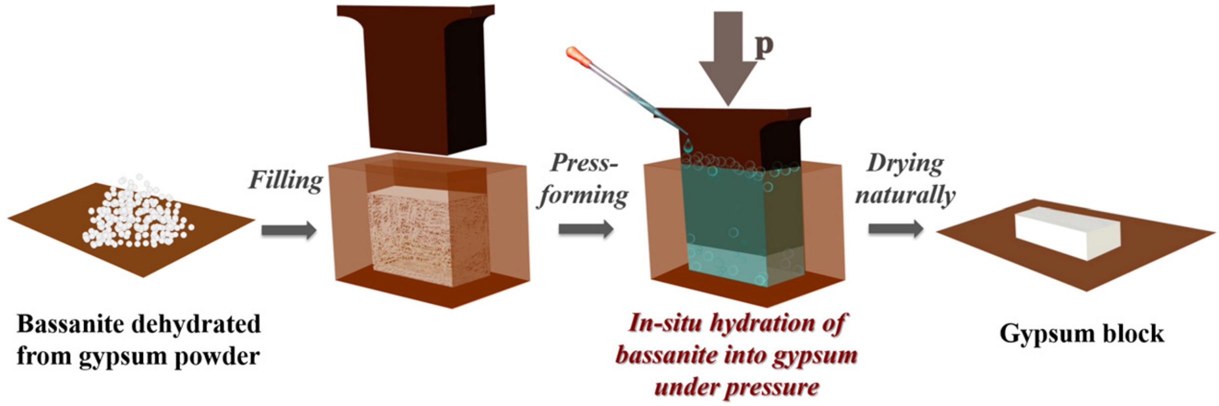

Fig. 1. The schematic diagram of preparing gypsum blocks by the In-situ Loading-Hydration Process.

The hardness of gypsum specimens was identified by Vickers Indenter (HVS-1000) with 300N indentation. The bending strength was measured by the three-point method with a 40 mm span at a crosshead speed of 1 mm/min on WAW1000D Microcomputer-controlled electro-hydraulic servo universal testing machine. The bulk density and porosity were determined by the Archimedes method using the aviation kerosene as the medium according to the national standard of PR China (GB/T9966.3-2001) on MDMDY-300 Automatic density meter. The crystalline phase was determined by X-ray Powder Diffractometer (XRD; D/Max-3b, Rigaku) with Cu Ka radiation, at 35 kV and 40 mA with 10 s scanning time. The microstructure of fracture surface of the specimens after coating with gold was observed by field emission scanning electron microscope (FE-SEM, Hitachi SU8010) in the vacuum environment. Five specimens were prepared at each loading-pressure and used for testing.

3. Results and discussion

3.1. Optimal performances

Both the hardness and bending strengths of as-prepared gypsum blocks versus loading-pressures are plotted in Fig. 2. It shows that, a maximum hardness of 1.47 GPa is achieved when the loading pressure lies in 50 MPa, which approaches the theoretical value of gypsum crystals (1.5 GPa) as reported by Soroka et al. [23]. On the other hand, Fig. 2 shows that, an encouraging maximum bending strength of 17.5 MPa is attained under the loading pressure of 20 MPa, far higher than the standard C40 concrete (about 5.5 MPa) and even close to normal ceramic tiles (≥15 MPa, ISO 13006:1998).

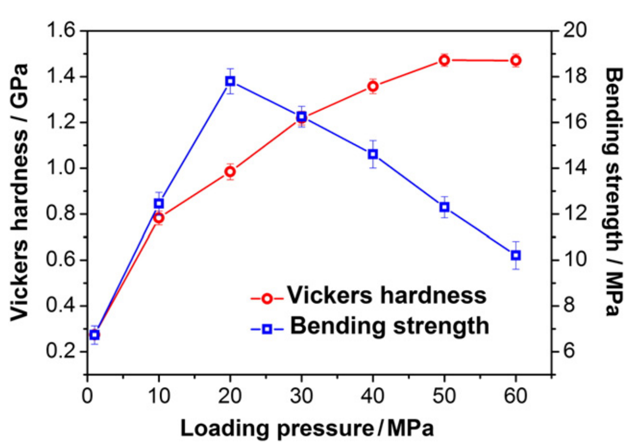

Fig. 2. The Vickers hardness and bending strengths of gypsum blocks versus loading pressures.

It is noted that the maximum hardness and bending strength are not obtained at the same loading-pressure, which will be fully discussed in the latter part of the paper. Because the mechanical strength of the material is a preferential and key property for considering its application, the optimal loading-pressure lies in 20 MPa, which provides the gypsum block with a bending strength as high as 17.5 MPa and an appropriate hardness of 0.98 GPa, and endows the gypsum product with the potential as a candidate of load-bearing material.

3.2. Vickers hardness

Fig. 2 shows that, the Vickers hardness increases gradually to 1.47 GPa as the loading-pressure increases to 50 MPa, and it keeps constant at the further increased pressure of 60 MPa.

The XRD diffraction on the sample surface (shown in Fig. 3a) confirms that, the surfaces of samples prepared at different pressures are identical with each other in crystalline phase and are all composed of gypsum ($CaSO_4 \cdot 2H_2O$). Moreover, these gypsum crystals have the same cell parameters as the standard gypsum (JPCDS 06-0046) as proved by their same peak locations in XRD patterns (Fig. 3a). Thus, the application and control of loading-pressure during the hydration of bassanite into gypsum do not change the crystal type of gypsum, and the increase of hardness of gypsum blocks hydrated under higher pressures does not result from the crystal type.

Fig. 4 shows the porosities of all samples as a function of their loading-pressures. It demonstrates that, the porosity drastically decreases from 32.5% to 3.5% when the loading-pressure increases from 1 to 30 MPa, and it becomes relatively changeless at further increased pressures. Correspondingly, the bulk densities of these samples show a change trend just reverse to that of porosities. The porosity change trend can be visually observed from the cross-section photographs of gypsum blocks prepared at the different loading pressures of 1 MPa (Fig. 5a), 20 MPa (Fig. 5b) and 50 MPa (Fig. 5e). The inset of Fig. 4 further plots the Vickers hardness of gypsum blocks versus their porosities. It shows that, the decrease of porosity is accompanied with an accelerated growth of the Vickers hardness. Therefore, it is believed that, the hardness of the as-prepared gypsum block is directly relative to its porosity, and the superior hardness of gypsum blocks processed at higher pressures can be mainly attributed to their lower porosities.

3.3. Bending strength

Fig. 2 shows that, the changing trend of bending strengths of the gypsum blocks versus loading-pressures is different from the continuous-increasing trend of hardness. The bending strength first increases to the maximum value of 17.5 MPa as the loading-pressure increases to 20 MPa, and then gradually declines at further increased loading-pressures.