Construction and Building Materials 110 (2016) 65–69

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Fabrication of slaked lime compacts (plasters) with high compressive strength using a warm press method

Shinobu Hashimoto *, Wataru Shimoda, Hayami Takeda, Yusuke Daiko, Sawao Honda, Yuji Iwamoto

Nagoya Institute of Technology, Department of Environmental and Materials Engineering, Gokiso-cho, Showa-ku, Nagoya 466-8555, Japan

HIGHLIGHTS

- A new warm press technique was employed to form dense slaked lime compacts.
- A compact fabricated 150 °C and 240 MPa under uniaxial pressure for 90 min reached an average compressive strength of 57 MPa.
- Warm press treatment led to mass transport occurred through a sintering-like process during warm pressing.

ARTICLE INFO

Article history:
Received 16 June 2015
Received in revised form 15 December 2015
Accepted 2 February 2016
Available online 10 February 2016

Keywords:
Slaked lime
Plaster
Warm press
Compressive strength
Chemical thermodynamics

ABSTRACT

So far dense calcium hydroxide body (slaked lime plaster) with high mechanical strength has not been fabricated yet. In this study, novel method was tried in order to fabricate the dense calcium hydroxide body. As a result, a calcium hydroxide body with a dense and high mechanical strength was obtained. Pure calcium hydroxide powder was placed in a cylindrical steel mold 15 mm in diameter, and then simultaneously pressed uniaxially at 60–240 MPa and heated to 100–250 °C for various lengths of time. This compacting process is referred to as a warm press. After warm pressing, hardened compacts composed of pure calcium hydroxide were obtained. Increasing both the applied uniaxial pressure and the heating temperature increased the compressive strength of the resulting hardened compacts. Furthermore, increasing the time spent in warm press conditions also increased the compressive strength of the hardened compacts. However, these increases diminished over time, and the compressive strength reached a nearly constant value after approximately 90 min. When the uniaxial pressure, heating temperature, and duration were 240 MPa, 250 °C and 90 min, respectively, the compressive strength of the resulting hardened compact was 57 MPa. Finally, in this study, the phase stability relationship between calcium hydroxide and calcium carbonate is discussed on the basis of chemical thermodynamics. This warm press technique for the fabrication of dense hydroxide ceramics should help clarify the formation mechanism of natural hydroxide minerals.

1. Introduction

Lime plaster has been a widely used construction and building material worldwide since ancient times. The primary raw material of lime plaster is slaked lime, or calcium hydroxide: $Ca(OH)_2$. In the ancient cities of Central and South America, the urban infrastructure was mainly built using lime plaster [1–4]. Even in the Middle Ages in Europe, plaster technology was used in buildings and fresco paintings [5–9]. In Japan, ancient powerful families used plaster in murals (decorations) for their tumuli (tombs) [10]. Furthermore, medieval rulers (shoguns) built castles with white plaster walls. One of the national treasures of Japan is Himeji-jo (castle), which was built approximately 400 years ago and is registered as a UNESCO world heritage site [11]. Since the outer wall of Himeji-jo is a beautiful white, due to the use of a large amount of lime plaster, Himeji-jo is also called White Heron Castle. After lime plaster technology was used in several castles in the Japanese middle ages, its use spread to temples, shrines, and storehouses of wealthy merchants, and it is still in common use today.

In Japan, a slaked lime paste slurry is generally used for the construction of a lime plaster wall. The work is done by highly skilled craftsmen, who apply a paint containing a slaked lime paste slurry and a reinforcement filler or humectant, materials empirically selected by the individual craftsman. Over approximately one hundred years, the slaked lime paste on a wall gradually changes to

* Corresponding author.
E-mail address: hashimoto.shinobu@nitech.ac.jp (S. Hashimoto).

http://dx.doi.org/10.1016/j.conbuildmat.2016.02.009
0950-0618/

calcium carbonate, or lime plaster, which has a higher mechanical strength, through the following reaction [12–14];

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (1)$$

Therefore, the mechanical strength of slaked lime immediately after it is applied to a wall is not very high. However, slaked lime, or calcium hydroxide, has several highly desirable properties as a construction and building material, such as aesthetic appearance, antimicrobial capability (inactivation of avian influenza virus) [15,16], and high humidity control [17]. If slaked lime compacts, i.e., unfired titles with higher mechanical strength, could be easily fabricated, the use of slaked plaster tiles in conventional construction and/or buildings would increase. So far, since calcium hydroxide (slaked lime) is thermodynamically unstable phase in air, fabrication of the hardened body of pure phase of slaked lime has not been employed. However the carbonization of slaked lime takes a long time, therefore the dense body of slaked lime is expected to be used as not only tiles but also daily use construction ceramic materials such as furniture.

In this study, slaked lime compacts with a higher density and mechanical strength were fabricated from calcium hydroxide powder as the sole starting material, using a novel warm press technique. This method was developed to rapidly fabricate geopolymer products with high densities and high mechanical strengths [18]. In this method, uniaxial pressures up to 300 MPa and heating temperatures up to 300 °C were employed to the starting powder compact samples at the same time. Although the intrinsic mechanism is not clear, calcium hydroxide which is one of hydroxide compounds can be also hardened by the action of hydroxyl ions or water vapor in air during warm press processing. The resulting slaked lime compacts will continue to carbonize in air for a hundred years, which will continue to increase their mechanical strength. Furthermore, the phase stability relationship between calcium hydroxide and calcium carbonate under atmospheric pressure and under warm press conditions is discussed, since their phase stability will change under the condition of not only temperature but also pressure. In this experiment, changes in the relative density, crystal phase, microstructure, and compressive strength of hardened compacts fabricated from calcium hydroxide powder under various warm press conditions were examined.

2. Experimental procedure

Reagent grade calcium hydroxide powder (Kishida Chemical Co., Ltd., Osaka, purity above 96%) was used as a staring material. The powder was placed in a cylindrical steel mold 15 mm in diameter, and then the mold was placed in a warm press machine, which had top and bottom pressboards equipped with electric resistance heaters. The maximum heating temperature and uniaxial pressure were 250 °C and 240 MPa, respectively. The heating of this machine was controlled by a simple on/off switch. When the machine was switched on, the temperature of the pressboards reached a fixed value within 10 min. When the temperature of the steel mold reached a fixed value, an experiment was initiated. The duration of the heating was counted from the time when the temperature reached the desired fixed value. On the contrary, pressing starts from the room temperature, since only pressing has no effect on densification of the samples. That is, actual heating and pressing were performed simultaneously. The prepared samples were exposed to warm press conditions for various lengths of time, the longest of which was 180 min. All warm press experiments with this machine were performed in air.

After warm pressing and cooling to room temperature, the samples were removed from the steel mold. The compressive strength of the samples was examined using a universal testing machine (INSTRON5582: INSTRON, Norwood, MA, USA). The crosshead speed was 1 mm/min. At least three samples were used from each set of warm press conditions to conduct compressive strength tests. The crystal phases of the samples before and after warm pressing were analyzed by X-ray diffraction (XRD: X'pert-MPD; PHILIPS, The Netherlands), and the micromorphology of the samples was observed by scanning electron microscopy (SEM: JSM-6010LA; JEOL, Japan).

3. Results and discussion

3.1. Formation of hardened slaked lime compacts

When the slaked lime compacts were fabricated by simple heating to 250 °C without continuous pressing, hardened bodies could not be obtained. In other words, the compressive strength of slaked lime bodies after merely heating them to 250 °C was very low. When the slaked lime compact bodies were fabricated by continuous pressing at 240 MPa without heating, the strength was improved slightly, but still not sufficiently strong. The latter process of pressing without heating is a conventional powder compacting process used for the production of fine ceramics before sintering. In contrast, when we applied a combination of heating and pressing, also called a warm press treatment, the outcomes were much better. Fig. 1 shows an optical photograph of the appearance of the hardened slaked lime compacts after warm pressing at 150 °C with 120 MPa of uniaxial pressure for 90 min. The resultant hardened bodies resembled sintered bodies, and had high mechanical strength. Fig. 2 shows the compressive strengths of slaked lime hardened compacts fabricated at various heating temperatures for different lengths of time at 120 MPa. The results indicate that increasing the heating temperature and pressing time increased the compressive strength of the hardened compacts. The compressive strength remained relatively constant after 90 min, and the difference between heating at 150 and 250 °C was slight, so we selected a heating temperature of 150 °C and a warm press time of 90 min as a suitable standard condition to study the effect of pressure during the warm heating.

Fig. 3 shows the changes in compressive strength and density of hardened compacts obtained by heating at 150 °C for 90 min in the uniaxial press. With increasing uniaxial pressure under the same warm press conditions, the compressive strength of the hardened compacts increased. Specifically, when 60 MPa of uniaxial pressure was applied, the average compressive strength of the hardened compacts was 17.5 MPa. However, when the uniaxial pressure was 240 MPa, the compressive strength of the hardened compacts increased to 57 MPa. The increase in the compressive strength must be owing to the increase in the density of the products as shown in Fig. 3. When 60 MPa of uniaxial pressure was applied, the density of the resulting hardened body was approximately $1.3 \times 10^3\ kg \cdot m^{-3}$. In contrast, when 240 MPa was applied, the density of the hardened body reached $1.8 \times 10^3\ kg \cdot m^{-3}$.

After hardening by warm press treatment, the crystal phases of the hardened compacts were analyzed. Fig. 4 shows XRD patterns for hardened compacts obtained by heating at 150 °C for 90 min

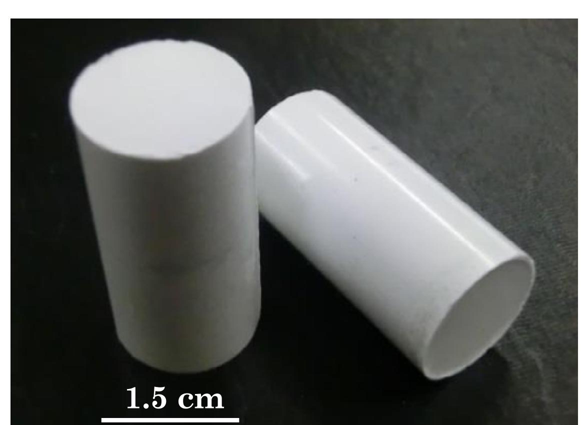

Fig. 1. Optical photograph of hardened slaked lime compacts after warm pressing at 150 °C and 120 MPa for 90 min.