

# Effects of humidity on calcite block fabrication using calcium hydroxide compact

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## Abstract

Calcite has attracted attention as an artificial bone replacement material and as a precursor for the fabrication of carbonate apatite, which is also an artificial bone replacement material. In this study, the effect of humidity on calcite block fabrication was investigated using calcium hydroxide (Ca(OH)<sub>2</sub>) compact. Ca(OH)<sub>2</sub> compact and Ca(OH)<sub>2</sub> paste compact were exposed to CO<sub>2</sub> at room temperature under 0%, 50%, and 100% humidity for two weeks. No carbonation was observed when Ca(OH)<sub>2</sub> compact was exposed to CO<sub>2</sub> under 0% humidity. In contrast, Ca(OH)<sub>2</sub> compact transformed into pure calcite under 100% humidity. Forty percent of the Ca(OH)<sub>2</sub> compact transformed into calcite under 50% humidity, while 30% of the Ca(OH)<sub>2</sub> paste compact transformed into calcite. Interestingly, the diametral tensile strength of the Ca(OH)<sub>2</sub> paste compact was four times higher than that of the Ca(OH)<sub>2</sub> compact when both were exposed to CO<sub>2</sub> under 100% humidity, despite the paste compact's lower conversion into apatite. After exposure to CO<sub>2</sub>, SEM observations revealed that in the case of the paste compact, the Ca(OH)<sub>2</sub> powder was bridged with a precipitate, whereas in the case of Ca(OH)<sub>2</sub> compact, no precipitate was found. Results obtained in this study demonstrated that carbonation of the Ca(OH)<sub>2</sub> compact at room temperature was the result of a dissolution–precipitation reaction. Ca(OH)<sub>2</sub> powder was dissolved into water to supply the Ca<sup>2+</sup>, and CO<sub>3</sub><sup>2-</sup> was supplied for the calcite precipitation from the interaction of CO<sub>2</sub> and water. Excess humidity from the paste compact was the key to the precipitation of the calcite bridge. The presence of the calcite bridge resulted in a higher mechanical strength for the calcite block.

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## 1. Introduction

Calcium carbonate (CaCO<sub>3</sub>) has become an important artificial bone substitute [1–5]. Marine coral, which contains CaCO<sub>3</sub> as aragonite, has been used as an artificial bone substitute [6–13]; however, usage of coral as a bone substitute can lead to the destruction of natural environments and can cause serious medical complications, including an inflammatory response. The limitations of natural aragonite's use as a biomaterial are thought to be caused by the impurities from coral [9]. Therefore, fabrication of an artificial CaCO<sub>3</sub> has been a long-awaited scientific achievement. One of the methods used to fabricate CaCO<sub>3</sub> is the carbonation of Ca(OH)<sub>2</sub> [14–16]. Lin et al. investigated the effect of the compacting

pressure on calcite block fabrication using Ca(OH)<sub>2</sub> compact. When Ca(OH)<sub>2</sub> was compacted uniaxially at 1 MPa and exposed to CO<sub>2</sub> for 3 days, the Ca(OH)<sub>2</sub> transformed into calcite. However, Ca(OH)<sub>2</sub> compacted at 15 MPa did not completely convert into calcite, even after 72 h. This incomplete conversion is thought to be caused by limited penetration of the CO<sub>2</sub> gas through the intergranular space due to dense packing of Ca(OH)<sub>2</sub> at higher compacting pressure. In other words, channels that allow CO<sub>2</sub> penetration inside the Ca(OH)<sub>2</sub> compact are required for Ca(OH)<sub>2</sub> carbonation [17–20]. Less is known about the effects of humidity on the carbonation of Ca(OH)<sub>2</sub>, even though 100% humidity is employed during Ca(OH)<sub>2</sub> carbonation at room temperature [16,21–23].

Ca(OH)<sub>2</sub> can be carbonated into CaCO<sub>3</sub> as shown in the following equation:



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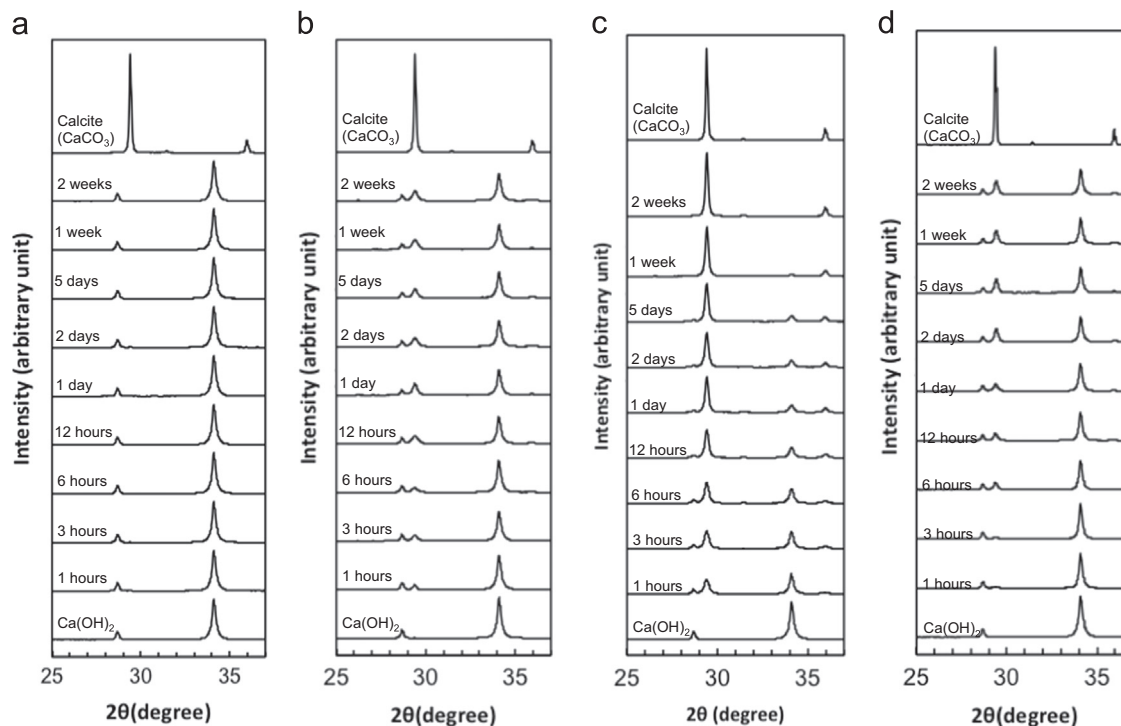


Fig. 1. X-ray diffraction patterns of the  $\text{Ca(OH)}_2$  compacts when exposed to  $\text{CO}_2$  at room temperature under (a) 0%, (b) 50%, and (c) 100% humidity and (d) the  $\text{Ca(OH)}_2$  paste compact when exposed to  $\text{CO}_2$  at room temperature under 100% humidity.

In other words,  $\text{Ca(OH)}_2$  theoretically can be converted into  $\text{CaCO}_3$  when exposed to  $\text{CO}_2$ , even in the absence of humidity, and  $\text{H}_2\text{O}$  is produced during the carbonation reaction. In this study, the effect of humidity on the carbonation of  $\text{Ca(OH)}_2$  compacts was examined to improve understanding of calcite block fabrication.

## 2. Materials and methods

### 2.1. Calcium hydroxide compact preparation

Commercially available  $\text{Ca(OH)}_2$  powder (Wako Chemicals, Osaka, Japan) was used in this study.  $\text{Ca(OH)}_2$  powder, 0.1 g, was placed in a stainless steel mold and pressed uniaxially under 20 MPa with an oil pressure press machine (Riken Power, Riken Seiki, Japan) at room temperature. Columnar specimens, 6 mm in diameter and 3 mm in height, were obtained.

$\text{Ca(OH)}_2$  compact was also made from a  $\text{Ca(OH)}_2$  paste by mixing 0.1 g of  $\text{Ca(OH)}_2$  powder with distilled water at a powder-to-liquid ratio of 0.56, in order to simulate the conditions of excess humidity. In the following text,  $\text{Ca(OH)}_2$  compact made from  $\text{Ca(OH)}_2$  paste is referred to as  $\text{Ca(OH)}_2$  paste compact.

### 2.2. Carbonation of calcium hydroxide compact

$\text{Ca(OH)}_2$  compacts and paste compacts were placed within  $\text{CO}_2$  reaction vessels (TMF-300, As One Corp., Osaka, Japan) at room temperature for up to 2 weeks.  $\text{CO}_2$  gas was introduced into the vessel at a flow rate of 200 mL/min under different relative humidity conditions (0%, 50%, and 100%). In the case of the  $\text{Ca(OH)}_2$  paste compact, used to model excess humidity, the carbonation reaction was examined only at 100% humidity.

( $\text{OH})_2$  paste compact, used to model excess humidity, the carbonation reaction was examined only at 100% humidity.

### 2.3. X-ray diffraction analysis

Composition of the specimens was determined using an X-ray diffractometer (XRD: D8 Advance, Bruker AXS GmbH, Karlsruhe, Germany) operated at 40 kV and 40 mA. The diffraction angle was continuously scanned, from  $10^\circ$  to  $60^\circ$ , in  $2\theta$  at a scanning rate of  $2^\circ/\text{min}$ . In Fig. 1, only  $25\text{--}37^\circ$  was shown, as no important peaks were observed in the excluded regions. Quantitative analysis was performed based on the obtained XRD patterns. A calibration curve for the quantitative analysis of calcite was constructed using a mixture of  $\text{Ca(OH)}_2$  (2 QUOTE = 28.67) and calcite (2 QUOTE = 35.96). The amount of calcite formed from  $\text{Ca(OH)}_2$  carbonation was calculated from the integrated area ratio of the XRD peaks using MDI Jade 5.0 Software (Materials Data Inc., Livermore, CA, USA).

### 2.4. Mechanical strength

The mechanical strength of the  $\text{Ca(OH)}_2$  compacts, before and after exposure to  $\text{CO}_2$ , was evaluated in terms of diametral tensile strength (DTS). The diameter and height of the specimens were measured by a micrometer (MDC-25MU Mitutoyo Co. Ltd., Kanagawa, Japan). The specimens were crushed using a universal testing machine (AGS-J, Shimadzu Co., Kyoto, Japan) at a constant cross-head speed of 1 mm/min.

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