



Assessing the carbon sequestration potential of magnesium oxychloride cement building materials



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ABSTRACT

Magnesium oxychloride cement (MOC) boards have the potential to offset carbon emissions through carbon mineralization, a process whereby carbon dioxide (CO₂) is converted to carbonate minerals. Boards (0–15 years old) contained MOC phase 5 (21–50 wt%), brucite, primary (e.g., magnesite) and secondary (hydromagnesite and chlorartinite) carbonate minerals. Quantitative mineralogy, electron microscopy and carbon abundance data demonstrate that secondary carbonates form through the reactions of MOC and brucite with CO₂ within interfacial water layers after board manufacturing. Stable carbon isotopic data confirmed the source of sequestered CO₂ as being from the atmosphere. Average carbonation rates were approximately 0.07 kg CO₂/m² board/year or 9.8 kg CO₂/t board/year over 15 years, offsetting ~20–40% of estimated carbon emissions. In experiments using 10% and 100% CO₂ gas, carbonation was accelerated by approximately 400 and 1600 times in comparison to the passive rate. Integration of carbonation reactions into MOC board production could provide significant carbon offsets.

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1. Introduction

Manufacturing of cement products accounts for approximately 5% of global greenhouse gas (GHG) emissions [1]. For this reason, extensive research is ongoing to minimize GHG emissions in the cement industry with carbon sequestration being one field of investigation (e.g. [2,3]). Magnesium oxychloride cement (MOC) is formed through the reaction of magnesium oxide (MgO) with magnesium chloride (MgCl₂) solution. The major commercial applications are in industrial flooring, fire protection, grinding wheels, and wall panels [4]. MOC boards have some superior properties compared with gypsum or fiber-based boards such as greater fire resistance, lower thermal conductivity, improved resistance to abrasion, and greater strength [5–8]. The versatility of magnesium-based cement building materials has spurred considerable research into product characterization and development [8–15], including for improving MOC water resistance [16–18]. Reactive MgO is produced from calcining of magnesite (MgCO₃) at lower temperatures (e.g., <750 °C) than the sintering temperature

of cement clinker of Portland cement (~1450 °C), thereby using less fuel and emitting less GHGs [19,20]. Nevertheless, there are substantial carbon emissions associated with manufacture of all cement-based products, and thus, opportunity to reduce those emissions.

Carbon mineralization, also referred to as mineral carbonation, involves storing CO₂ as solid carbonate minerals, a process that is being increasingly studied in the cement industry for reducing GHG emissions and improving product quality [21–27]. In addition, numerous studies have examined the effects of carbonation on the physical and mechanical properties of cement-based construction materials [2,28–34]. Sequestration of CO₂ into cement acts as a stable carbon sink and contributes to the development of sustainable “green” building products. Under ambient conditions, the carbonation rate of conventional cement is relatively slow, yet over its lifetime 7.6%–57% of the CO₂ produced during the calcination process may be offset [19]. It is estimated that 4.5 Gt of carbon has been sequestered through carbonation of cement materials from 1930 to 2013, which accounts for 43% of the CO₂ released from calcination of carbonate rocks during this period. Regarding MOC cement, there are documented cases of carbonation and volume increases of polishing bricks and mortar [35,36], yet the carbonation processes are not fully understood and it is not known to what

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extent MOC building materials sequester CO₂ during their lifespan. In addition to sequestering CO₂, carbonation of MOC may offer other benefits including greater strength and toughness because of denser microstructure and microhardness as has been documented for Portland cement with added MgO [19,20,37].

The goals for this study were to: (1) identify mineral sinks for atmospheric CO₂, (2) elucidate carbonation processes, (3) determine the carbon sources to confirm their value as GHG offsets, (4) estimate the passive rate of atmospheric CO₂ sequestration in MOC boards under ambient factory conditions, and (5) determine to what extent carbonation of MOC boards can be accelerated using CO₂-rich gas. Starting materials (magnesium oxide and magnesium chloride) and boards ranging from 0 to 15 years old were analyzed in this study. Quantitative mineralogical analyses were used to understand carbonation pathways and distinguish between primary carbonates that have formed through geologic processes and secondary carbonates that have formed post-production, and are therefore of value as carbon offsets. To explore the potential for accelerating carbonation, boards were incubated in carbonation chambers containing CO₂-rich gasses over several weeks. A detailed understanding of MOC carbonation processes, including identification of reactants and products and the corresponding changes in microstructure will contribute to knowledge about the carbonation mechanisms and thereby assist in ongoing technology development efforts in the construction industry.

2. Analytical methods for characterization of materials and boards

2.1. Sample cataloguing

MagO Building Products Ltd., a distributor of MOC boards, provided starting materials and MOC boards of known ages from manufacturers. These samples included nine boards of between 0 and 15 years old, three magnesium oxide powders, magnesium chloride salt, and magnesium chloride brine (Table 1). All boards were obtained from Chinese manufacturers except for board#8 that was obtained from a Florida manufacturer. Variations in the initial board compositions and structures as well as storage conditions were expected to have influenced the rate and extent of carbonation. Board dimensions and masses were recorded for determining board density and normalizing carbon content to board surface area and mass. Samples were generally 10–15 cm in length and width and 0.55–1.50 cm in thickness.

2.2. X-ray diffraction methods

X-ray diffraction (XRD) methods are useful for characterizing cement building materials and quantifying their mineralogical compositions [38–40]. Mineral phases in MgO powders and MOC boards were identified using powder XRD. Samples were ground in a mortar and pestle with subsamples being pulverized under anhydrous ethanol for 3 min using a McCrone micronizing mill and agate grinding elements. A 10 wt% internal standard of annealed CaF₂ was added to quantify amorphous content, which is an important component of cement [41]. Micronized samples were dried at room temperature for ~24 h and gently disaggregated with an agate mortar and pestle. Powder mounts were prepared in back-loaded cavity mounts against rough, frosted glass to minimize preferred orientation [42]. All XRD data were collected using a Bruker D8 Focus Bragg–Brentano diffractometer with a step size of 0.04° over a range of 3–80° 2θ at 0.7 s/step. Fe monochromator foil, 0.6 mm divergence slit, incident and diffracted beam slits, and a Lynx Eye positive sensitive detector were used. A long fine focus Co X-ray tube was operated at 35 kV and 40 mA using a take-

off angle of 6°. Search-match software by Bruker (DIFFRACplus EVA 14) was used for phase identification [43]. Quantitative phase analysis was conducted using Rietveld refinement of XRD data, which provides a measure of the weight-percent (wt%) contribution of each mineral in a sample. Crystal structure data for Rietveld refinement were obtained from the International Centre for Diffraction Data PDF-4+ 2010. Rietveld refinement was completed using Topas Version 3 software [44].

2.3. Percent carbon and geochemical analyses

The percent carbon (%C) of the solid samples was determined using a Model CM501C4 CO₂ Coulometer from UIC Inc. Calcium carbonate standards were analyzed to ensure instrument calibration. This method is accurate to within ±0.05% by mass and has a detection limit of 0.10% C. Aliquots of known mass of the magnesium chloride were dissolved in deionized water and the magnesium chloride brine was diluted. These samples were analyzed for their anion and cation compositions using ion chromatography and inductively coupled plasma – optical emission spectroscopy by ALS Environmental in Burnaby, British Columbia, respectively.

2.4. Stable isotope analyses

Aliquots of ground boards and MgO powders were placed in Labco exetainers and acidified using 85% phosphoric acid (H₃PO₄). Sample vials were sealed and heated to 72 °C for 1 h prior to analysis. The CO₂ generated from the acidification of carbonate minerals was passed through an ethanol-dry ice cold trap and drawn into a Los Gatos Research (LGR®) off-axis integrated cavity output laser spectrometer [45]. The LGR® analyzer measures the absorption spectra of ¹²C¹⁶O¹⁶O, ¹³C¹⁶O¹⁶O, and ¹²C¹⁶O¹⁸O in the near-infrared wavelength spectrum. The stable carbon and oxygen isotope values are reported in the conventional δ notation in per mil (‰) relative to Vienna Pee Dee Belemnite (VPDB) and Vienna Standard Mean Ocean Water (VSMOW), respectively (Eq. (1)).

$$\delta^{13}\text{C}_{\text{sample}} = \left[\frac{^{13}\text{C}/^{12}\text{C}_{\text{sample}} - ^{13}\text{C}/^{12}\text{C}_{\text{std}}}{^{13}\text{C}/^{12}\text{C}_{\text{std}}} \right] \times 1000 \text{ in per mil (‰)} \quad (1)$$

In-house gas and mineral standards were used to correct for instrument drift and for two-point normalization to VSMOW and VPDB. One standard deviation uncertainties based on repeated measurement of a calcite standard, measured on the same day as the unknowns, were 0.2‰ and 0.3‰ for δ¹³C and δ¹⁸O, respectively. The δ¹⁸O values of Mg-carbonate sediments were corrected for reaction with phosphoric acid using the fractionation values from Das Sharma et al. [46]. The fractionation factor for magnesite was used as a proxy for hydrated Mg-carbonate minerals.

2.5. Scanning electron microscopy

Board samples were split using a razor blade to expose a cross-section for imaging. Scanning electron microscopy (SEM) was performed at the Centre for High-Throughput Phenogenomics at The University of British Columbia. Samples were coated with 8 nm of iridium using a Leica EM MED020 coating system and imaged using a FEI Helios NanoLab 650 operating at 1.0 kV voltage.

3. Experimental methods for accelerating carbonation

Two sets of experiments were conducted to determine MOC carbonation rates using CO₂-rich gases under controlled temperature and relative humidity. In the first experiment, six boards were selected for accelerated carbonation with initial carbon abundances

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