



# Morphological changes during enhanced carbonation of asbestos containing material and its comparison to magnesium silicate minerals



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## ABSTRACT

The disintegration of asbestos containing materials (ACM) over time can result in the mobilization of toxic chrysotile ((Mg, Fe)<sub>3</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>) fibers. Therefore, carbonation of these materials can be used to alter the fibrous morphology of asbestos and help mitigate anthropogenic CO<sub>2</sub> emissions, depending on the amount of available alkaline metal in the materials. A series of high pressure carbonation experiments were performed in a batch reactor at P<sub>CO2</sub> of 139 atm using solvents containing different ligands (i.e., oxalate and acetate). The results of ACM carbonation were compared to those of magnesium silicate minerals which have been proposed to permanently store CO<sub>2</sub> via mineral carbonation. The study revealed that oxalate even at a low concentration of 0.1 M was effective in enhancing the extent of ACM carbonation and higher reaction temperatures also resulted in increased ACM carbonation. Formation of phases such as dolomite ((Ca, Mg)(CO<sub>3</sub>)<sub>2</sub>), whewellite (CaC<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O) and glushinskite (MgC<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O) and a reduction in the chrysotile content was noted. Significant changes in the particle size and surface morphologies of ACM and magnesium silicate minerals toward non-fibrous structures were observed after their carbonation.

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## 1. Introduction

Safe disposal of asbestos, which is known to cause significant health problems such as asbestosis, lung cancer and mesothelioma [1], has been considered as one of the most difficult and costly challenges in environmental engineering and public health. Asbestos was extensively used in manufactured goods and building materials due to its high strength and heat resistant properties attributed by the fibrous chrysotile ((Mg, Fe)<sub>3</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>) component, which is also the main cause of health problems. For example, about 2 billion m<sup>2</sup> of cement-asbestos which is predominantly found in industrial and civil buildings, is present in Italy [2] which may degrade over time to release toxic fibers [3–7]. Products containing cement asbestos were manufactured such that a slurry containing 10–15 wt% asbestos fibers were mixed with ordinary Portland cement and dried slowly to obtain cement composites. Due to their high tensile strength, the asbestos fibers served as reinforcing

agents [3,7]. The trading and use of asbestos containing materials (ACMs) are now banned in several countries and has prompted the policy of the abatement and the disposal of ACM in controlled landfills. However, the landfill method cannot be regarded as the ultimate solution, as there is no guarantee that the landfilled fibers will not be dispersed into air or water. Technologies that can permanently alter the fibrous nature of asbestos are necessary to ensure safe handling and disposal of ACM. Furthermore, if the ACM treatment can be combined with CO<sub>2</sub> capture and storage schemes [8–10] that alter the morphological structure [11], the developed technology will have multi-faceted environmental benefits.

A number of asbestos treatment schemes that have been suggested in the past focused on the structural and chemical alteration of asbestos for the landfill disposal. Chemical treatment of asbestos with phosphoric acid [12], supercritical steam [13], 1–2 N HCl or a mixture of CaCl<sub>2</sub> and Na<sub>2</sub>SO<sub>4</sub> [14], oxalic acid and sulfuric acid solutions [15,16], and acidic gases such as HCl and HF [17] were effective in decomposing asbestos. While thermal treatment of asbestos at 620–750 °C can lead to dehydroxylation of chrysotile [18,19], heating asbestos to temperatures greater than 1000 °C can lead to enhanced rate of transformation of ACM [20–22] with the potential for applications in ceramics. In situ thermal treatment with atmospheric microwave air plasma technology [23], grinding [24,25] and

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**Table 1**  
Compositions of ACM and magnesium silicate minerals based on XRF analyses.

| Components                     | ACM (wt%) | Serpentinite (wt%) | Chrysotile (wt%) | Olivine (wt%) |
|--------------------------------|-----------|--------------------|------------------|---------------|
| CaO                            | 38.10     | 0.40               | 0.01             | 0.16          |
| MgO                            | 3.90      | 38.80              | 41.10            | 47.30         |
| Fe <sub>2</sub> O <sub>3</sub> | 1.90      | 7.10               | 2.40             | 13.90         |
| SiO <sub>2</sub>               | 17.70     | 39.70              | 41.70            | 39.70         |
| Al <sub>2</sub> O <sub>3</sub> | 3.90      | 1.80               | 1.10             | 0.20          |
| Na <sub>2</sub> O              | 0.21      | –                  | –                | 0.01          |
| K <sub>2</sub> O               | 0.32      | 0.01               | –                | <0.01         |
| TiO <sub>2</sub>               | 0.18      | 0.05               | –                | <0.01         |
| P <sub>2</sub> O <sub>5</sub>  | 0.07      | –                  | –                | <0.01         |
| MnO                            | 0.03      | 0.08               | 0.05             | 0.15          |
| Cr <sub>2</sub> O <sub>3</sub> | 0.07      | 0.37               | –                | 0.78          |
| V <sub>2</sub> O <sub>5</sub>  | 0.01      | 0.01               | –                | <0.01         |
| LOI <sup>a</sup>               | 33.60     | 12.07              | 13.60            | –0.70         |

Material is heated to 1000 °C until there is no change in the weight of the sample.

<sup>a</sup> LOI: loss of ignition.

ultrasonic treatment in a solution containing Mg and Fe-targeting chelating agents (i.e., oxalic acid) [26,27] also resulted in the complete breakdown of ACMs. Unfortunately, these technologies are often energy intensive. Thus, a need for a more environmentally benign and energy efficient method for ACM treatment has been suggested.

Recently, significant research has been conducted on the enhanced carbonation of magnesium silicate minerals that have very similar chemical compositions as chrysotile or actually contain chrysotile, for the purpose of CO<sub>2</sub> storage [28–34]. It was found that the morphological structures of these silicate minerals undergo significant changes during the mineral carbonation. Thus, this study proposes the utilization of CO<sub>2</sub> to treat ACM by converting it to non-hazardous, readily disposable or even re-useable carbonates. The morphological structures of produced mineral carbonates are determined based on the types of carbonates formed. Generally, nesquehonite (MgCO<sub>3</sub>·3H<sub>2</sub>O) and hydromagnesite (Mg<sub>5</sub>(CO<sub>3</sub>)<sub>4</sub>(OH)<sub>2</sub>·4H<sub>2</sub>O) are favored at low temperatures, while magnesite (MgCO<sub>3</sub>) is favored at higher temperatures. Thus, the mineralogy and the reaction conditions (e.g., temperature, CO<sub>2</sub> and H<sub>2</sub>O pressures, and/or chemical compositions of the solvent) are all important factors for enhanced mineral carbonation. Even at relatively low carbonation cases, the surface morphological structure of minerals could be significantly altered due to the carbonate layer formed directly on the surface of the mineral particles [32,33,35]. Therefore, this study investigated the effects of reaction temperature, the composition of reaction fluid and mineralogy on the morphological and chemical changes of ACM and magnesium silicates based on the aqueous mineral carbonation approach.

## 2. Materials and methods

### 2.1. Materials

Synthetic materials including ACM collected from rooftop tiles and a number of natural minerals were procured and ground for this study. Serpentinite (composed of 95% lizardite) and chrysotile samples were procured from a serpentinite outcrop from Montecastelli Pisano (Central-Southern Tuscany, Italy). Ground Twin Sisters olivine from Washington State (USA) was obtained from our collaborator at Albany Research Center. The chemical compositions of the studied materials were determined by X-ray Fluorescence (XRF) analyses. The major component of ACM was found to be CaO with smaller amounts of MgO, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>, whereas the magnesium silicate minerals were mostly composed of MgO followed by Fe<sub>2</sub>O<sub>3</sub>, in addition to SiO<sub>2</sub> (Table 1). The studied ACM is a sample of corrugated cement–asbestos slate collected from Italy. The mineralogical phase composition carried out by X-ray powder

diffraction (XRPD) showed calcite as the most abundant crystalline component, followed by an appreciable amount of clinochrysotile, quartz, plagioclase and minor gypsum (Fig. 2(a)). The weak low-angle reflection peaks are attributed to the scarce presence of phyllosilicates. The apparent absence of cement phases is due to carbonation in air, a slow process leading to the transformation of cement phases to calcite from portlandite and from the poorly hydrated crystalline phases of hydration in cement [6]. The match of the chemical and mineralogical composition of the investigated ACM sample and its comparison with some analogous Italian cement-asbestos manufactured goods, recently reported by Viani et al. [4] lead us to infer that this material is about 10 years old and contains around 10% clino-chrysotile as asbestoid mineral phase.

### 2.2. Carbonation of ACM and silicate minerals

A series of experiments were performed to determine the conversion and the changes in the chemical compositions and structural properties during the carbonation of different types of materials: ACM, olivine, serpentinite and chrysotile. Particularly, the effects of the reaction temperatures (i.e., 90, 125 and 185 °C) and the solvent compositions (i.e., deionized water, 0.1 M Na-oxalate, 0.1 M Na-acetate and 1.0 M Na-acetate) on ACM carbonation were investigated.

The carbonation experiments were performed in a high-temperature, high-pressure batch reactor manufactured by Autoclave Engineers (Fig. 1). The reactor was connected to a 500D Teledyne Isco high pressure syringe pump. A 50 ml of slurry containing 15 wt% of solids was charged to the reactor, which was then sealed. The stirring speed was set at 800 rpm throughout the experiment since it was the optimum speed for effective mass and heat transfer within the reactor. Once the reactor temperature set-point was reached, the reactor pressure was increased to 139 atm of CO<sub>2</sub>, which marked the start of the experiment. The reaction time was set to 3 h for all experiments to achieve a measurable extent of carbonation, which was based on the preliminary carbonation experiments performed using olivine. At the end of 3 h, the reactor was cooled to below 70 °C before taking liquid and solid samples. The filtered liquid samples were diluted ten times into 2% HNO<sub>3</sub> solution to prevent any post-reaction precipitation and the solid samples were dried at 70 °C for 12 h for further analyses.

### 2.3. Analyses of starting materials and carbonated products

Both starting materials and carbonated products were dried at relatively mild temperatures and analyzed using a battery of experimental techniques. The mineralogical and chemical compositions of solid samples were determined using XRF and XRPD. Electron

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