



# CO<sub>2</sub> sequestration by direct gas–solid carbonation of fly ash with steam addition

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## ABSTRACT

Mineral carbonation using alkaline industrial solid wastes is a promising CO<sub>2</sub> sequestration technology. In this work, the effects of temperature, CO<sub>2</sub> content, steam content, and reaction time on CO<sub>2</sub> sequestration at atmospheric pressure were studied by the direct gas–solid carbonation of circulating fluid bed fly ash in a thermogravimetric analyzer and a fixed bed reactor system. Results indicate that increasing the temperature and the content of CO<sub>2</sub> and H<sub>2</sub>O<sub>(g)</sub> can improve the CO<sub>2</sub> sequestration efficiency of the circulating fluid bed fly ash. However, the effect of CO<sub>2</sub> content is not as significant as that of temperature and H<sub>2</sub>O<sub>(g)</sub>. The maximum CO<sub>2</sub> sequestration capacity of 60 g CO<sub>2</sub>/kg fly ash with a maximum sequestration efficiency of 28.74% was achieved at 600 °C with 20% H<sub>2</sub>O<sub>(g)</sub> addition. The Brunauer–Emmett–Teller surface area and the pore volume decreased with the increase in average pore size after carbonation, due to the formation of a dense carbonate protective layer and pore blockage. With steam addition, the surface area and the pore volume increase, which contributes to the conversion of CaO to CaCO<sub>3</sub>. The production of Ca(OH)<sub>2</sub> or transient Ca(OH)<sub>2</sub> and the enhancement of CO<sub>2</sub> molecular mobility account for the promotion of steam in the carbonation process. The promotion mechanism of steam in different stages of reaction and temperature were explored in detail. Given the rich reserves of fly ash in China, it shows a good application prospect to use the Chinese fly ash as a mineral carbonation feedstock, which may not only reduces CO<sub>2</sub> emission, but also stabilizes the wastes.

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## 1. Introduction

In recent years, the development of efficient and economical techniques for reducing anthropogenic CO<sub>2</sub> emissions has been of great importance due to the severe global climate change problem. Mineral carbonation (MC), which was originally proposed by Seifritz (1990) is a potential Carbon Capture and Storage (CCS) technology for sequestering CO<sub>2</sub> from small/medium emitters (Sanna et al., 2014). MC ensures the permanent and inherently safe storage of CO<sub>2</sub> by forming thermodynamically stable carbonates and has the advantages of extensive material sources, low cost, and low energy consumption (Tian and Jiang, 2012).

MC generally contains two routes: direct gas–solid carbonation and aqueous carbonation. The efficiency of aqueous carbonation is relatively high (Ben et al., 2015), but the increase in reaction temperature and the complicated operating procedure limit the application of this technology (Tian and Jiang, 2012). Direct gas–solid carbonation can prevent the leaching of heavy metals in water without additional chemicals, thereby minimizing the environmental consequences (El-Naas et al., 2015). In addition, the heat from the gas–solid reaction can be applied usefully (Huijgen, 2003). However, the reaction rate is low and therefore usually focused on temperature (Wang et al., 2008), pressure (Mazzella et al., 2016), and H<sub>2</sub>O (Reddy et al., 2011) to accelerate the direct gas–solid carbonation process.

Alkaline wastes as an alternative source of feedstock are rich in reserves, which reduce the costs of mining and transportation (Sanna et al., 2014). Several researchers have studied the use of alkaline wastes to sequester CO<sub>2</sub> in the dry route, such as fly ash

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(Tamilselvi Dananjayan et al., 2016), steel-making bag house dust (El-Naas et al., 2015), air pollution control system residues (Tian and Jiang, 2012), and waste concrete and anorthosite tailings (Ben et al., 2015). Others suggested that the dry route rate is too low to scale up the process economically (Ben et al., 2015).

Fly ash, as a basic industrial solid waste, shows great potential for MC due to its rich production despite its relatively low free CaO content (Ji et al., 2017). The global production of fly ash is estimated at 700 million tons per year (Izquierdo and Querol, 2012). In China, coal-fired power plants have generated over 580 million tons annually by 2015, which accounted for over 50% of the worldwide production (Yao et al., 2015). Among these fly ash, circulating fluid bed (CFB) fly ash normally has a higher CO<sub>2</sub> sequestration capacity, which contains 20%–30% CaO due to 30%–45% desulfurization efficiency (Jia and Anthony, 2000). More than 50 million tons of CFB fly ash and slag are discharged annually in China (Zhao et al., 2015). The coal fly ash from the pulverized coal furnace that mainly contains quartz (SiO<sub>2</sub>), hematite (Fe<sub>2</sub>O<sub>3</sub>), mullite (Al<sub>6</sub>Si<sub>2</sub>O<sub>13</sub>), magnetite (Fe<sub>3</sub>O<sub>4</sub>), and some lime (CaO) (Tamilselvi Dananjayan et al., 2016), can be applied in the construction industry, for instance, as a mineral admixture in cement mixtures (Ebrahimi et al., 2017). The CFB fly ash with high free CaO contains dehydrated silicate minerals (quartz, cyanite, and muscovite) and iron-containing minerals (hematite and pyrrhotite) is rarely used in the construction industry, but is useful for zeolite synthesis (Grela et al., 2016). The disposal of CFB fly ash is difficult due to the volumetric expansion problem (Patel et al., 2017). In circulating fluid bed combustion (CFBC) fly ash, the sulfur exists mainly as II-CaSO<sub>4</sub> (II-anhydrite), and easily leads to great volume expansion. When CFBC fly ash is mixed with water, II-anhydrite not only hydrates to form gypsum but also reacts with activated alumina and calcium hydroxide to form ettringite. While ettringite forms with apparent solid volume increased by 125% (Zhang et al., 2012). So by the MC process, the calcium hydroxide could be reacted with CO<sub>2</sub> to form stable carbonates, thus alleviating the volumetric expansion problem. Using the CFB fly ash as a MC feedstock may not only reduce CO<sub>2</sub> emission, but also stabilize the wastes, thereby achieving the goal of “waste control by waste.”

Studies on the gas–solid carbonation of fly ash are limited. CO<sub>2</sub> can be captured using fly ash in two ways: adsorption and carbonation. The former is seldom investigated due to its low adsorptive capacity (Siriruang et al., 2016). Temperature, pressure, and H<sub>2</sub>O are the key factors of gas–solid carbonation. On one hand, increasing temperature and pressure under dry conditions is considered the most efficient measure to promote the MC process. At 45 °C, a CO<sub>2</sub> uptake capacity of over 18 wt% that corresponds to a maximum carbonation efficiency of 74% is achieved by the gas–solid carbonation treatment of coal fly ash in the pressure range 1–15 bar (Mazzella et al., 2016). The maximum sequestration capacity was found to be 26.3 g of CO<sub>2</sub>/kg of coal fly ash (CFA) at 10 bar for 1 h at room temperature (Tamilselvi Dananjayan et al., 2016). Meanwhile, no noticeable carbonation occurs without pressurizing when temperature is below 400 °C (Wang et al., 2008). Experiments have been carried out for dry fly ash at 500 °C–750 °C (Patel et al., 2017). On the other hand, the reaction rate could be improved significantly with the existence of H<sub>2</sub>O (gaseous or liquid). Wang et al. (2008) investigated the carbonation reactions in the CFB fly ash under the oxy-fuel condition and found that the addition of steam (8%, 15%) enhances the reaction even at 250 °C. Patel et al. (2017) found that hydrated fly ash shows a similar or even a higher capture level than dry fly ash even at 30 °C–80 °C. Evidently, the gas–solid carbonation of fly ash is a viable route, especially under humid conditions. However, the mechanism of the carbonation reaction remains unclear due to the complicated mineral phases of fly ash. The enhancement of water vapor in the

fly ash carbonation process is complicated and has not been studied clearly.

However, many researchers have applied significant improvement on the carbonation of CaO-based sorbents by steam addition. The mechanism of the influence of steam on carbonation is controversial (Liu et al., 2012). This may be attributed to the catalytic effect of H<sub>2</sub>O through the formation of Ca(OH)<sub>2</sub>, which is more reactive than CaO (Manovic and Anthony, 2010). In addition, the core expands and particles fracture because the molar volume of Ca(OH)<sub>2</sub> is greater than that of CaO (Blamey et al., 2010). Another reason may be the enhancement of the solid state diffusion in the product layer (Manovic and Anthony, 2010). Pontiga et al. (2013) reported that the addition of hydrophilic nanosilica further enhances the CO<sub>2</sub> fast sorption capacity with Ca(OH)<sub>2</sub> due to the high amounts of free molecular water. Otherwise, pore structure is developed by hydration treatment, which yields high sorbent reactivity (Li et al., 2016). Both surface area and pore volume increase with steam presence (Chen et al., 2016).

There are many studies on the mineralization of CFB fly ash, but these studies often focused on the aqueous carbonation route (Sanna et al., 2014). Studies on the gas–solid carbonation of fly ash are limited, especially for studies on the effects of steam. The chemical composition and physical structure of fly ash have a significant effect on the carbonation process. With steam addition, the changes is more complicated. In different stages of reaction and temperature, the promotion mechanism of steam may be different, which is closely related to the physical and chemical structure of fly ash. But there is little literature from this perspective to study the carbonation process of fly ash. In addition, given the rich reserves of CFB fly ash in China, it is important to study more about characteristics of Chinese CFB fly ash. Using the CFB fly ash as a MC feedstock may not only reduce CO<sub>2</sub> emission, but also stabilize the wastes.

In this work, the CFB fly ash was evaluated as a possible feedstock for CO<sub>2</sub> sequestration through direct gas–solid carbonation. The effects of temperature, CO<sub>2</sub> content, steam content, and reaction time were studied using a thermogravimetric analyzer (TGA) and a fixed bed reactor system. The reaction mechanism, especially with steam addition, was investigated by TGA, X-ray fluorescence (XRF), X-ray diffractometer (XRD), N<sub>2</sub> adsorption, and scanning electron microscopy equipped with an energy dispersive system (SEM-EDS). With steam addition, the chemical composition and physical structure of the CFB fly ash were studied in detail. And in different stages of reaction and temperature, the promotion mechanism of steam was revealed. This work mainly aimed to estimate the sequestration potential of the CFB fly ash under dry conditions with steam addition and clarify the promotion mechanism of steam on the mineral carbonation process of CFB fly ash.

## 2. Materials and methods

### 2.1. Materials

The CFB fly ash used in this experiment was collected from a Chinese CFB power plant in Yuanping, Shanxi province (YP), which is based on a low-rank coal with high sulfur and ash in Shuozhou, Shanxi province. Before the experiments, fly ash of 125 μm particle size was obtained by sieving and then sealed in a dry dish. However, the residual carbon in fly ash may interfere with the sample weight in the TGA, and free CaO may react with H<sub>2</sub>O(g) and CO<sub>2</sub> in the air in the storage process. At room temperature, in the air containing saturated water vapor (2.3 kpa), over 95% of CaO will change to Ca(OH)<sub>2</sub> after 38 h (Blamey et al., 2010). Consequently, the ash was heated in a muffle furnace at 800 °C for 1 h, and was dried overnight prior to the experiments (YP-C). The purity of the gases (CO<sub>2</sub>,

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