An experimental investigation of mesoporous MgO as a potential pre-combustion CO\textsubscript{2} sorbent

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Abstract We examined the CO\textsubscript{2} capture capacity of mesoporous MgO (325 mesh size, surface area = 95.08 \pm 1.5 m\textsuperscript{2}/g) as a potential pre-combustion CO\textsubscript{2} sorbent. Our results show that 96.96\% of MgO was converted to MgCO\textsubscript{3} at 350 °C and 10 bars CO\textsubscript{2} pressure. The sorbent could be completely regenerated at 550 °C under argon flow. The sorption rate parameters such as surface area and pore size were investigated.

Keywords Capture capacity • Mesoporous • Pre-combustion • Sorbent • Surface area • Pore size

Introduction

Fossil fuel accounts for the world’s major energy supply and its use is anticipated to be continued throughout the 21st century [1]. The use of fossil fuel is always accompanied with a vast emission of CO\textsubscript{2}. The anthropogenic CO\textsubscript{2} emissions upset the natural carbon cycle leading to an increased atmospheric CO\textsubscript{2} concentration. No one can deny that there is an urgent need to develop methods for CO\textsubscript{2} mitigation.

Currently, a large focus is devoted on capturing CO\textsubscript{2} from coal-fired power plant flue streams, which continues to be the major CO\textsubscript{2} contributor [2]. There are three main technological approaches for CO\textsubscript{2} capture—post-, oxy- and pre-combustion. Post-combustion systems capture CO\textsubscript{2} from N\textsubscript{2}-rich flue gas stream, produced by burning fossil fuel in air. Oxy-combustion uses pure stream of O\textsubscript{2}, instead of air, to combust coal and thus produces CO\textsubscript{2}-rich gas stream. On the other hand, pre-combustion systems are designed mainly to remove CO\textsubscript{2} from the syngas (CO + H\textsubscript{2}) prior to its combustion for power production [3]. Under pre-combustion conditions, after the water–gas shift reactor (CO + H\textsubscript{2}O \rightarrow CO\textsubscript{2} + H\textsubscript{2}), the gas stream mainly consists of CO\textsubscript{2}, H\textsubscript{2}O and H\textsubscript{2}. The partial CO\textsubscript{2} pressure for pre-combustion capture conditions is around 20–30 bar and the temperature is between 250 and 450 °C [4].

Both the physical and chemical solvents can be used for pre-combustion CO\textsubscript{2} capture. Unlike chemical solvent, physical solvent (such as Selexol and Rectisol) selectively absorbs CO\textsubscript{2} without forming any chemical bonds. Thus, the physical solvent requires relatively less regeneration energy as compared to that of chemical solvent [5]. However, these physical solvent-based processes suffer severe disadvantages: (1) lose pressure during regeneration step, and (2) require a low operating temperature. Therefore, syngas needs to be cooled prior to CO\textsubscript{2} absorption step to attain a relatively low operating temperature. After CO\textsubscript{2} absorption, the remaining hydrogen gas stream requires to be reheated to the gas turbine inlet temperature. However, chemical solvents have the advantage of high mass transfer driving force into solution and better acid gas selectivity. Also, chemical solvents can be used in processes that utilize thermal swing regeneration and generate the CO\textsubscript{2} at elevated pressure [6]. But chemical solvents increase the energy and cost penalty and thus are downgraded as a future CO\textsubscript{2} sorbent [7].
Currently, the focus is to develop advanced physical and chemical solvent systems that have the potential to provide significant improvements in both cost and performance as compared to the Selexol and Rectisol for pre-combustion CO₂ capture. The challenges are to modify regeneration conditions to recover the CO₂ at a higher pressure, improve selectivity to reduce H₂ losses, and develop a solvent that has a high CO₂ loading at a higher temperature, which would increase integrated gasification combined cycle (IGCC) efficiency. In the same line, another vital task is to develop a new sorbent which could be highly efficient for pre-combustion capture conditions. Consequently, the US Department of Energy (DOE) performed the thermodynamic modeling activities that included screening analyses for a number of different metal oxides, zirconates, silicates and titanates under various operating conditions to identify new solid sorbents for pre-combustion CO₂ capture. About 18 sorbents were modeled and finally seven candidates are chosen—magnesium oxide (MgO), calcium oxide (CaO), lithium zirconate (Li₂ZrO₃), calcium zirconate (CaZrO₃), barium zirconate (BaZrO₃), barium titanate (BaTiO₃) and barium silicate (BaSiO₃) for further investigations [8]. Among these solid sorbents, MgO and CaO are attractive because of their easy accessibility and favorable thermodynamic properties [9].

The alkaline earth metal oxides (such as CaO and MgO) combine with CO₂ to form thermodynamically stable carbonates. Metal carbonates, when heated, liberate pure stream of CO₂ gas and regenerate the oxides. Eventually, the generated pure CO₂ gas can either be sequestered underground or used for enhanced oil recovery [10]. CaO are abundant and thus relatively easily accessible than MgO. However, CaO as CO₂ sorbent suffers severe major drawbacks and a detailed discussion can be found elsewhere [11]. It is a fact that regeneration of oxides needs a lot of energy [12]. MgO and Mg(OH)₂ are known to be better candidates than CaO for CO₂ capture applications due to their low regeneration energy requirement and low operating temperature [13]. Thus, MgO (periclase), although occurs only rarely as an oxide, we choose it here for our study. One should note that MgO when recycled between naturally occurring magnesite or dolomite can cause relatively lesser energy or carbon emission penalty [14].

The CO₂ absorption capacity of MgO was studied as a function of particle size, surface area, temperature, pressure, support and concentration of water vapor. The CO₂ uptake capacity on different MgO sorbents at different conditions is listed in Table 1. Most of the experiments are restricted to low temperature and ambient or low pressure condition. Hence, the reported sorption capacities of CO₂ on MgO are not very high. However, the sorbents which can operate in the range of 300–350 °C would be ideal for the use in IGCC applications. Therefore, we study the CO₂ capture capacity of MgO in the relatively high temperature and pressure condition of 300–375 °C and 10–50 bars, respectively.

### Experimental procedure

The reaction

\[ \text{MgO(s)} + \text{CO}_2(g) \rightarrow \text{MgCO}_3(s) \]

was studied experimentally. The carbonation reaction was performed in a closed system which permits us to efficiently maintain high temperature and pressure for a long period of time. The decomposition reaction of carbonate was examined using Thermo gravimetric Analysis (TGA) technique.

About 0.2 g of 325-mesh-sized magnesium oxide (delivered by Alfa Aesar) was put inside a closed cylindrical vessel (1.25” long and 0.35” internal diameter). About 0.1 ml of water was also introduced on the vessel walls. Chemically pure CO₂ gas (Airgas) was passed into this system. Before experiments, CO₂ gas was flushed three times to ensure a pure CO₂ atmosphere inside the reactor. The reaction was performed for 30 min at a desired temperature and pressure condition. Once the reaction completed, the system was air-cooled. The product was then grind using mortar and pestle. The powder particle was again put back for another reaction at the same experimental condition. This cycle was repeated until no increment in weight of the product was observed.

Thermo gravimetric analysis (TGA) of the product was done using TGA 2950 Thermo gravimetric analyzer. The samples (10 mg) were heated under argon purge, at a heating rate of 10 °C/min to a final temperature of 800 °C. The TGA provides continuous measurements of the sample weight as a function of time and temperature. The amount of formation of MgCO₃ was analyzed by the percent loss in weight of the sample while heating up to 800 °C in an argon atmosphere.

The product characterization was performed using X-ray powder diffraction method. Bruker GADDS/D8 is equipped with Apex Smart CCD Detector and direct-drive rotating anode. The MacSci rotating anode (Molybdenum) operates with a 50 kV generator and 20 mA current. X-ray beam size can vary from 50 to 300 μm. The usual collection time is 1200 s.

An isothermal gas adsorption was employed to measure internal surface areas of the powder particles. Micromeritics Tristar II 3020 (surface area and porosimetry analyzer instrument) was used with N₂ as adsorptive gas at 77 K (liquid nitrogen bath). The samples were first degassed under 300 °C with a N₂ gas flow for 1 h to remove
Table 1  CO₂ uptake capacity of MgO obtained from the literature

| Sorbent | Gas stream | Carbonation temperature (°C) | Pressure (bar) or, flow rate | Particle Size | Regeneration temperature (°C) | CO₂ capture capacity (mmol/g) | Conversion, % | References |
|---------|------------|-----------------------------|-----------------------------|---------------|-------------------------------|-----------------------------|--------------|------------|
| 1 MgO   | Pure CO₂   | 50–1000                     | 100 mL/min (flow rate)      | –             | –                            | 0.99                        | –            | [15]       |
| 2 MgO/Al₂O₃ (10 wt. % MgO) | (13 v % H₂O, 13 v % CO₂) | 30, 150 | 1 | (20–40) mesh size | 350 | 1.36 | [16] |
| 3 MgO   |             | 50–100                      | 0.01                        | –             | 150–400                      | 1.05                        | –            | [17]       |
| 4 K₂CO₃/MgO | (11 v % H₂O, 1 v % CO₂) | 50–100 | – | – | 150–400 | 2.98 | – | [17] |
| 5 MgO   |             | 330 ppm in air              | 0.01                        | –             | –                            | 0.64, 0.43                  | –            | [18, 19]  |
| 6 MgO/MCM-41 | Pure CO₂ | 25 | 1 | – | – | 1.06 | – | [20] |
| 7 Mesoporous MgO | Pure CO₂ | 25, 100 | 1 | – | – | 1.82, 2.27 | – | [21] |
| 8 Nonporous MgO | Pure CO₂ | 25 | 1 | – | – | 0.45 | – | [21] |
| 9 MgO–ZrO₂ | Pure CO₂ | 30, 150 | 1 | – | – | 1.15, 1.01 | – | [22, 23] |
| 10 MgO (31.7 wt%)/Al₂O₃ (22.4 wt%) | Pure CO₂ | 20, 200, 300 | 1 | – | – | 0.13, 0.24, 0.5 | – | [24] |
| 11 MgO (33.8 wt%)/Al₂O₃ (20.8 wt%) | Pure CO₂ | 20, 200, 300 | 1 | – | – | 0.08, 0.12, 0.5 | – | [24] |
| 12 K₂CO₃/MgO/Al₂O₃ | Flue gas | 60 | 1 | – | 480 | 2.49 | – | [25] |
| 13 K₂CO₃/MgO | (9 v % H₂O, 1 v % CO₂) | 60 | 40 mL/min (flow rate) | – | 400 | 2.7 | – | [26] |
| 14 MgO nanocrystal | Flue gas | 60 | 25 sccm (flow rate) | 5 nm | 60–600 | 6.4 | – | [27] |
| 15 MgO | Pure CO₂ | 350 | 1.33, 3.33 | – | – | 0.089, 0.091 | – | [28] |
| 16 MgO | Pure CO₂ | 300–500 | 9–36 | <44 μm | – | 70–80 % (~ 200 min) | – | [29] |
| 17 MgO | Pure CO₂ | – | 20–40 | <44 μm | – | 100 % (~ 120 min) | – | [30] |
the moisture and other adsorbed gases before analysis. The internal surface area was calculated using the Brunauer–Emmett–Teller (BET) method. The pore volume was also calculated from the adsorbed nitrogen after complete pore condensation ($P/P_0 = 0.9925$) using the ratio of the densities of liquid and gaseous nitrogen. The pore size was calculated using the Barrett–Joyner–Halenda (BJH) method.

Results and discussion

Figure 1 confirms the formation of MgCO$_3$ at different temperatures and CO$_2$ pressures. MgO, Mg(OH)$_2$, MgO·2MgCO$_3$ and MgCO$_3$ were identified conventionally by their corresponding Joint Committee Powder Diffraction Standard (JCPDS) card number 79-0612,82-2345,31-0804 and 86-2345, respectively.

One of the vital factors in gas–solid carbonation reaction is the presence of water and there have been numerous observations where water acts as a catalyst [31–34]. Therefore, we have also used water (0.1 ml) for the MgO–CO$_2$ reaction. In absence of water, no CO$_2$ was absorbed at these conditions due to the kinetic limitations. The CO$_2$ sorption capacity of MgO increases significantly in the presence of water vapor. Under humid condition, MgO rapidly locks CO$_2$ in the form of MgCO$_3$. Recently, Fagerlund et al. [29] proposed the reaction mechanism for MgO carbonation in the presence of steam:

$$\text{MgO} + \text{H}_2\text{O} \rightarrow \text{MgO} \cdot \text{H}_2\text{O}$$

$$\text{MgO} \cdot \text{H}_2\text{O}^+ + \text{CO}_2 \rightarrow \text{MgCO}_3 + \text{H}_2\text{O}$$

$$\text{MgO} + \text{CO}_2 \rightarrow \text{MgCO}_3$$

Figure 2 depicts the adsorption/absorption model for MgO–CO$_2$–H$_2$O reaction. Based on the previous work, it could be reasonable to corroborate that water vapor surrounds MgO particles where CO$_2$ reacts to form CO$_3^{2-}$ ions and H$^+$ ions [34–36]. Free Mg$^{2+}$ ions could further react with the CO$_3^{2-}$ ions to form MgCO$_3$. However, MgCO$_3$ forms an impervious layer around unreacted MgO particles and hinders the further diffusion of CO$_2$ molecules.

Here, we used mortar and pestle to grind the product. As mentioned earlier, we conducted each experiment for 30 min and ground the sample after that. Grinding helps in scrubbing off the outer nonporous layer of MgCO$_3$. And we performed grinding until we noticed no change in product weight after subsequent experiments. In general, after 3–4 cycles, we observed no change in the weight of product. It is certain that such intermittent grinding step is limited to the laboratory and cannot be seen as an industrial operation. Therefore, it is recommended to have an aggregative fluidization regime for a fluidized bed reactor while scaling up MgO–CO$_2$ reaction. Also, increasing the amount of water vapor cannot lead to the complete carbonate conversion of MgO. Thus, in addition to the amount of steam, surface properties of MgO (such as surface area, particle size, porosity) are also very crucial parameters for the carbonation process.

The thermal analysis curve does not show any significant differences in amounts of carbonate in the high-pressure (50 bars) experiments. We obtained almost similar TGA plots and XRD patterns for different temperatures 300–375 °C and 50 bars CO$_2$ pressure. However, the experiment performed at 300 °C and 10 bars did indicate that the product was not simply MgCO$_3$. The X-ray diffraction

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Fig. 1 XRD patterns for MgCO$_3$ formation after reaction at various temperatures and CO$_2$ pressure of a 10 bars and b 50 bars
pattern in Fig. 1a confirms the presence of Mg (OH)₂ and relatively high amount of MgO-2MgCO₃ at 300 °C and 10 bars. DTG curve (Fig. 3a) also evidences the similar situation. The two peaks around 350 and 475 °C were attributed to the losses of water of crystallization and hydroxyl water, respectively. However, another peak at 550 °C corresponds to the complete decomposition of MgCO₃. But at a temperature and pressure of 350 °C and 10 bars, respectively, only MgCO₃ was formed. This is evidenced by both DTG (Fig. 3b) and X-ray diffraction analysis (Fig. 1a).

The steps to calculate % conversion of MgO to MgCO₃ is explained in supplementary section [S1]. A conversion of 30.54 and 96.96 % for MgO to MgCO₃ was observed at 300 and 350 °C, respectively. Here, we observed the formation of relatively high amount of oxymagnesite (MgO-2MgCO₃) at 300 °C and 10 bars. It can be easily calculated that the CO₂ capture capacity of oxymagnesite is about two-third that of MgCO₃. Hence, formation of oxymagnesite can markedly reduce the overall uptake of CO₂ by MgO particles. However, it can be observed from X-ray patterns (Fig. 1) that at higher
temperature or pressure, the formation of oxymagnesite is suppressed.

In the same line, recent study reveals that oxymagnesite forms as an intermediate during the thermal decomposition of hydrated magnesium carbonate [37]. Moreover, oxymagnesite can also be formed as a product of reaction between Mg(OH)₂ and CO₂ in anhydrous synthesis [29] and/or solid-state reaction of MgO and CO₂ using steam [38]. In this regard, Duan et al. [13] calculated the phase diagram of MgO–Mg(OH)₂–MgCO₃, which suggests that the transition temperature for direct conversion of MgCO₃ to Mg(OH)₂ increases with increase in P_H₂O. Here, when temperature increases from 300 to 350 °C, partial pressure of water also increases and consequently transition temperature also increased. At 300 °C, a relatively less amount of MgO transforms to its carbonates. This can be attributed to a possible high conversion of MgCO₃ to Mg(OH)₂, which is in agreement with the phase diagram. Moreover, a significant formation of oxymagnesite at 300 °C and 10 bars also leads to a less direct conversion of MgO to MgCO₃. Thus, it can be deduced that a high amount of oxymagnesite forms at a low partial pressure of water. Therefore, the reaction mechanism is changed to:

\[ \text{MgO} + \text{CO}_2 \rightleftharpoons \text{MgCO}_3 \]

\[ \text{MgO} + 2\text{MgCO}_3 \rightleftharpoons \text{MgO} \cdot 2\text{MgCO}_3 \]

\[ \text{MgO} \cdot 2\text{MgCO}_3 + \text{CO}_2 \rightleftharpoons 3\text{MgCO}_3 \]

Moreover, at 50 bars of CO₂ pressure and a temperature of 300 °C, MgO has the highest yield of 98.54 %. At high temperatures (300–375 °C) but constant pressure (50 bars), we observe a slight but continuous decrease in the absorption of CO₂, which is in congruence with previously reported results [30]. It is well known that at low temperature, the physisorption process dominates but at elevated temperature CO₂ chemisorbs on MgO and thus CO₂ uptake capacity gradually starts decreasing.

CO₂ uptake capacity of a metal oxide is primarily dominated by the factors such as surface area, pore volume, pore functionality and pore size [28]. BET surface area measurement, pore volume and average pore sizes presented in Table 2 indicates the significant role of CO₂ to influence the particle structure. As received, 325 mesh size mesoporous MgO has a pore size of 9.09 nm and a high surface area (95.08 ± 1.5 m²/g). Figure 4 illustrates that the N₂ adsorption–desorption isotherm curves for as-received MgO follows Type IV isotherms (as per IUPAC classification); typical for mesoporous substances [39]. Also, the hysteresis pattern is H3 (following IUPAC classification) indicating the presence of slit-like pores. After capture of CO₂ at 350 °C and 10 bars, surface area dramatically decreased to (4.15 ± 0.05) m²/g. The ratio of surface area to pore volume is also reduced by almost half and results in high diffusion paths. The significant decrease in surface area attributes to the basicity of mesoporous MgO. The mesoporous MgO is highly basic with well-ordered pores to hold high CO₂ at both lower and higher temperatures. It is well known that porous materials allow molecules to pass through their pore aperture for storage, separation or conversion [36]. MgO with a fine particle size (<44 μm) has a high content of mesopores, which leads to good mass transfer properties during the absorption process. The mechanism for metal oxide reaction with CO₂ gas has been discussed a lot [40–43]. CO₂ molecules diffuse through the pores of mesoporous MgO and the present large active sites hold these CO₂ molecules [19]. The trapped CO₂ molecules further react to form MgCO₃.

It can be observed from Table 2 that at 350 °C and 10 bars, 96.96 % of MgO was converted to MgCO₃ and almost all the pores have been utilized after reaction with CO₂ molecules. Henceforth, almost no CO₂ molecules could have diffused further in the pores. Noticeably, pore size increased to 16.25 nm. Thus, it is apparent that the porosity of MgO particles plays a very vital role for CO₂ uptake.

### Table 2

| Sample                  | Surface area (m²/g) | Pore volume (cm³/g) | Pore size (nm) | SA/PV(10⁶ m⁻¹) |
|-------------------------|---------------------|---------------------|----------------|-----------------|
| As-received MgO         | 95.08 ± 1.5         | 0.22                | 9.09           | 439.59          |
| After reaction at (350 °C, 10 bars) | 4.15 ± 0.05 | 0.01                | 16.25          | 246.15          |

![Surface area = 95.08m²/g](image.png)

**Fig. 4** N₂ adsorption–desorption isotherm of as-received MgO
Previously, Beruto et al. has reported a very low CO₂ absorption capacity of MgO in absence of water vapor [27]. An uptake capacity of 0.089 and 0.091 mmol CO₂/g MgO was observed at 350 °C in 1.33 and 3.33 bars of CO₂, respectively. Thus, it was concluded that there is a high activation energy barrier to MgO recarbonation. On the contrary, Feng et al. [14] heated the sorbent (MgO) to 1000 °C in pure CO₂ and noted a low but almost constant (for 8 cycles) absorption capacity of 0.99 mmol CO₂/g MgO. Thus, unlike CaO-based sorbents, MgO does not show a fast decline in their CO₂ capture capacity over a large number of carbonation–calcination cycles. Bhagiyalakshmi et al. [20] synthesized basic mesoporous MgO (surface area of 250 m²/g) using mesoporous carbon obtained from SBA-15 and obtained a maximum CO₂ adsorption of 2.27 mmol CO₂/g at 100 °C and nearly 1.81 mmol CO₂/g at 25 °C for a feed flow rate of 30 ml/min CO₂ gas (99.9 % purity). Therefore, it is evident that even for a relatively lower surface area (95 m²/g compared to 250 m²/g), MgO can have a better CO₂ uptake at a higher temperature (here, 300–375 °C) and higher CO₂ pressure (10–50 bars). Recently, Fagerlund et al. [28] reported a very high % conversion of MgO to MgCO₃ under high temperature and pressure conditions. However, MgO showed a slow conversion rate (50 % for ~7 h) for the condition [P CO₂ (~18–19 bar), P H₂O (~1–2 bar) and 300–350 °C] which can be suitable for pre-combustion capture process. The reason could be the deposition of impervious carbonate layer on the surface of MgO which does not allow CO₂ molecules to further diffuse in the pores. Therefore, continuous removal of these nonporous layers of carbonates is needed. As mentioned earlier, we recommend to have an aggregative fluidization regime for a fluidized bed reactor while scaling up MgO–CO₂ reaction.

In summary, we demonstrate the increase in uptake of CO₂ by mesoporous MgO at high temperature and pressure in the humid environment. We observed the significant role of temperature, pressure, water vapor and porosity of the sorbent on the improvement of the CO₂ absorption capacity of mesoporous MgO.

**Conclusion**

MgO is a promising candidate for pre-combustion CO₂ capture due to their stable absorption capacity of CO₂, adequate mechanical strength, high attrition resistance and good regenerability. We observed a 96.96 % of MgO conversion to MgCO₃ at 350 °C and 10 bars CO₂ pressure. Moreover, CO₂ uptake capacity of MgO can be increased by performing the reaction in the presence of slight humid environment and scrubbing off the outer non-porous layer of MgCO₃. We also demonstrate that MgO has a low regeneration temperature (550 °C) and stable CO₂ capture capacity over wide range of temperature and pressure. Interestingly, the present study also illustrate that a high amount of oxymagnesite forms at a low partial pressure of water.

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