Capture of CO₂ by vermiculite impregnated with CaO

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Introduction

The greenhouse effect is a natural phenomenon that can be enhanced by a large amount of carbon dioxide (CO₂) discharged into the atmosphere, mainly due to the burning of fossil fuels. Carbon storage and capture are currently considered as techniques with great potential for the necessary reduction of this gas emission [1,2]. However, there are two major challenges...
for the use of these techniques: (i) the high cost of the capture processes and (ii) the scale-up difficulties [3].

The calcination reaction of calcium carbonate is widely studied and this process occurs only at temperatures over \(\approx 900 \, ^\circ C\) [4–7]. At lower temperatures, the formation of calcium carbonate is thermodynamically favourable, i.e. calcium oxide and carbon dioxide are converted into calcium carbonate, as shown in the following chemical equation (Equation 1):

\[
\text{CaO(s)} + \text{CO}_2(g) \rightleftharpoons \text{CaCO}_3(s)
\]

The formation of calcium carbonate from calcium oxide can be used as a strategy to capture CO\(_2\) [3]. It is noteworthy that the greater the conversion of calcium oxide in calcium carbonate, the more efficient the process will be and the better use of this raw material will be made. Since the calcium oxide carbonation reaction is exothermic, lower temperatures imply higher conversion values and, on the other hand, lower reaction rates. Thus, when pursuing to obtain a high conversion, the reaction becomes slow and, consequently, long residence times become necessary. Then, in addition to the use of catalysts, the increase in the surface area available for the reaction becomes a possible alternative to accelerate the process [1,8,9].

Vermiculite is a mineral with chemical formula \((\text{Mg,Fe,Al})_3(\text{Al,Si})_4\text{O}_{10}(\text{OH})_2.4\text{H}_2\text{O}\) which has been widely used as a support for catalysts due to its large surface area when expanded [10]. Some studies show that catalysts as \(\text{NiO, TiO}_2\) and \(\text{Al}_2\text{O}_3\) become more efficient when using this mineral as support [10,11]. These studies indicate that the thermal expansion of vermiculite prevents catalysts from being sintered and, consequently, their surface area is not reduced even when the experiments are carried out at high temperatures [12].

Several studies pursue alternatives that enable the capture of carbon dioxide by CaO using different fluid–solid contact reactors, e.g. recycling reactors [12,13], fixed [5] or fluidized bed reactors [14,15] and catalysts bed reactors [16,17]. Many of these studies show that the sintering of calcium oxide is a key factor related directly to the number of cycles or residence time supported by this material inside the furnace [18]. Therefore, calcium oxide can be impregnated in vermiculite in order to increase its surface area and, consequently, increase the efficiency of this reactor [12]. Some of us have been contributing to the study of techniques to capture CO\(_2\), either by modifying the Solvay process or using carbonation of CaO [19–21]. However, to the best of our knowledge, reports on dealing with the capture of CO\(_2\) by calcium oxide supported by vermiculite are scanty.

**Methodology**

Carbonation experiments were carried out using a sample of CaO P.A. (Synth). This sample was ground, calcined at 950 °C and then stored in a desiccator. Commercial vermiculite (Dimy) was used for all experiments. The impregnation of calcium oxide in vermiculite was performed using a thermal heating technique, i.e. mixtures with different CaO/vermiculite mass ratios were made and homogenized. Then, 5 ml of distilled water was added to 2 g of the mixture, all material was dried in a furnace at a temperature of 105 °C for 12 h. The dried samples were heated to 950 °C for 30 min, in a muffle oven. This procedure aimed to ensure the removal of all water present in the sample, to dehydrate the oxides that were eventually hydrated and to expand the vermiculite.

**Thermogravimetric analysis**

The thermal analysis experiments were performed on a Mettler Toledo equipment, model: Star System. Carbonation curves were performed using CO\(_2\) (99.8% - White Martins) at a flow rate of 100 ml/min and the heating rates used were 5 °C/min, 10 °C/min, 15 °C/min and 20 °C/min.

**Scanning electron microscopy (SEM) – energy dispersive spectrometry (EDS)**

MEV-EDS analyses were carried out using a Tescan equipment model VEGA 3 LMH coupled to an EDS INCA x-act model 51-ADD0007. All the samples were metallized with gold using the sputtering technique.

**Textural characteristics**

The liquid N\(_2\) desorption and adsorption isotherms were obtained on a Quantachrome Autosorb IQ2 after outgassing approximately 0.2 g of sample in vacuum at 423 K for 12 h (6 mm Cell Type, without rod). The specific surface area was obtained by the Brunauer-Emmett-Teller (BET) method and the pore size distribution by the Barrett-Joyner-Halenda (BJH) model following the desorption curve of the isotherm [22].
Isoconversional kinetic

Isoconversional methods stand out because they allow the apparent activation energy of a thermal process to be calculated without prior knowledge of the kinetic model \([21,23–29]\). In addition, using this method is possible to assess the dependence of activation energy on conversion. The origin of this method is the analysis of the kinetic equation 2 for a single reaction step:

\[
\frac{\partial \alpha}{\partial T} = \frac{A}{\beta} \exp \left( -\frac{E_a}{RT} \right) f(\alpha) \quad (2)
\]

Where \(A\) and \(E_a\) are the pre-exponential factor and the activation energy, respectively, \(f(\alpha)\) is the conversion function, \(R\) is the gas constant, \(\beta\) is the heating rate, \(T\) is the temperature and \(\alpha\) is the extent of conversion of the reagents into products.

The isoconversional principle establishes that, to constant conversion, the rate of the reaction is a function only of the temperature and, rearranging the equation 2, equation 3 is obtained:

\[
\ln \left( \frac{\beta}{d\alpha/dT} \right) = \ln \left( A f(\alpha) \right) - \frac{E_a}{RT\alpha} \quad (3)
\]

Equation 3 is the base of the differential Friedman method [30]. This method requires the numerical differentiation of experimental \(\alpha vs T\) curves. However, this procedure usually produces a lot of noise which leads to a spread in the \(E_a\) values. This can be avoided using the integral method, represented by equation 4, which is obtained through the integration of equation 2:

\[
g(\alpha) = \frac{A}{\beta} \int_0^{T_\alpha} \exp \left( -\frac{E_a}{RT} \right) dt = \frac{A}{\beta} \int l(E, T) \quad (4)
\]

The integral \(I(E,T)\) has no analytical solution and is normally solved applying numerical methods. Two expressions can be employed as approximate solutions for the integral \(l(E,T)\), the Osaka-Flynn-Wall (OFW) solution (equation 5) [31,32] and the Kissinger-Akahira-Sunose (KAS) solution (equation 6) [33]:

\[
-ln(\beta) = 1.0516 \left( \frac{E_a}{RT_\alpha} \right) - A' \quad (5)
\]

\[
-ln \left( \frac{\beta}{T_p^2} \right) = \frac{E_a}{RT_p} - \ln \left( \frac{A''R}{E_a} \right) \quad (6)
\]

Where \(A'\) and \(A''\) are constants. These equations show that a graph of \(-\ln(\beta)\) as a function of \(1/T_p\) or \(-\ln(\beta/T_p^2)\) as a function of \(1/T_p\) will be a straight line and the values of activation energy for the process can be calculated from the slope of these lines [27].

**Figure 1.** The thermogravimetric curve for the sample of CaO and the 1:1 mixture of CaO and Vermiculite (100 ml/min of CO\(_2\) and 10 °C/min).

**Thermodynamic data**

All the thermodynamic data were obtained from the HSC Chemistry 6.0 software.

**Results**

**Capture of CO\(_2\)**

Thermal analysis curves recorded with vermiculite, using a flow rate of 100 ml/min of CO\(_2\), showed that this material cannot capture this gas. Figure 1 shows the results of thermal analysis for tests performed with pure CaO and a 1:1 mixture of CaO:vermiculite (mass ratio). It is possible to observe that the capture of CO\(_2\) has increased considerably when vermiculite is used as a support. It is worth noting that when all calcium oxide is converted to calcium carbonate (equation 1) the expected increase in mass is 78.6%. The results show that the mass gain obtained for the sample of pure calcium oxide was 23.2% at a temperature of \(\approx 900 \degree\)C, this means that the conversion was \(\approx 0.30\), which is equivalent to \(\approx 13\) g of CO\(_2\) per mol of CaO. For the 1:1 mixture of vermiculite and CaO the results show a mass gain of 15% at a temperature of 888.7 °C which is equivalent to a conversion of 0.38 (16.8 g of CO\(_2\) per mol of CaO). It is noteworthy that for the vermiculite sample impregnated with CaO, the amount of calcium oxide used was half of that used for pure calcium oxide, therefore, it can be concluded that there was a much greater conversion when vermiculite was used as a support.

Equation 1 shows that the carbonation reaction is the reverse reaction of the calcination reaction (equation 7):
CaCO₃(s) = CaO(s) + CO₂(g) \quad \Delta G^0
\quad = -147,6T + 172070 \text{ (J/mol)} \quad (7)

For the activity of solids equal to the unit and reagents in the standard state, the values of \( \Delta G^0 \) (equation 7) show that the reaction will be spontaneous above 1165.8 K (892.6 °C). Thermodynamic data is in agreement with the mass loss observed for temperatures above 847.3 °C and 888.7 °C on the carbonation curves of impregnated vermiculite and calcium oxide, respectively. In other words, it can be said that the carbonation reaction is thermodynamically favourable at low temperatures and, therefore, for the capture of CO₂ one must work at low temperatures. On the other hand, it is known that chemical reactions are slow at low temperatures and when the reaction system cannot be subjected to high temperatures a catalyst must be used. In this work, using a heterogeneous reaction, the researchers chose to change the contact surface of the CaO sample in order to increase the reaction rate. Finally, the reduction in the calcination reaction temperature, when CaO is impregnated on the vermiculite surface, is one evidence of the increase in surface area available for the reaction promoted by the support, i.e. equation 7 shows that carbonation is favoured by an increase in the external pressure of CO₂. Thus, for a material with a smaller surface area, the average particle size will be large and, consequently, the layer of solid product (CaO) on the surface of the particles will be thicker, making it difficult for CO₂ to escape. Studies show that the CO₂ pressure at the CaCO₃/CaO interface can reach values as high as 6.3 atm [34,35] and, thus significantly increase the temperature required for calcination to occur.

SEM-EDS images of the vermiculite sample impregnated with CaO after the CO₂ capture test is shown in Figure 2. The images show that calcium oxide is dispersed over the vermiculite surface which makes it more exposed to the carbonation reaction as shown by the TG results.

Experiments were carried out with samples of vermiculite impregnated with different amounts of CaO. The conversion of CaO to CaCO₃ is shown in Figure 3.

The results show that it was possible to convert 73% of the calcium oxide into carbonate using a 3:1 mixture (CaO:vermiculite – 75% of CaO). When this value is compared with the values obtained for pure calcium oxide, it can be concluded that
calcium oxide capture capacity was increased from 13 g of CO₂ per mol of CaO to 32.2 g of CO₂ per mol of CaO which is equivalent to an increase of ≈ 250%. The conversion value obtained was as high as that found in studies using recycled reactors, which require a longer residence time and have a better solid-fluid contact design than that used in this work [3,13]. Further, the results also show that the capture capacity decrease when using vermiculite impregnated with 80% of CaO. In order to improve the capture capacity of calcium oxide, experiments were performed by impregnating calcium oxide in vermiculites with different particle sizes (Figure 3). A mixture of 2:1 (CaO:vermiculite – 66% of CaO) was used in these experiments since this ratio showed good capture results and uses a lower amount of CaO than the ratio with the best capture values (Figure 4).

Figure 5 shows the Nitrogen adsorption-desorption isotherms using liquid N₂ as adsorbate molecule for all the materials and the textural parameter of the samples are exhibited in Table 1. In a general approach, the isotherms of the solids are similar to II-type, existing, predominantly, macropores [36]. There is also the presence of mesopores due to the hysteresis loop originating from the capillary condensation. The shape of the hysteresis loop corresponds to the filling of the mesopores H3-type, observed for aggregates of plate-like particles with slit-shaped pores.[37]

Among the porous materials, the solid corresponding to 106 < φ < 300 μm presents the higher specific surface area value (11 m²·g⁻¹). The hysteresis profiles are similar, suggesting a linear tendency between the specific surface area and size particle. On the other hand, occurs a change in the hysteresis profile by the ruination of the lamellar structure and reduction of the availability of CaO within the porous structures. In other words, there is a rupture of the lamellar structure for size particles 0 < 106 μm reflecting the reduction of the capture of CO₂. Thus, there is a restricted relation between the specific surface area, particle size, and capture of CO₂ efficiency.

The best capture values were obtained for the impregnation made in the vermiculite with a particle size fraction of 106 μm < φ < 300 μm and it was possible to capture 31 g of CO₂ per mol of CaO. The results suggest that for smaller particle sizes there is an increase in the surface of the material (Table 1) which explains the improvement in CO₂ capture values. On the other hand, the lamellar structure of vermiculite is destroyed when the sample with φ < 160 μm is used as a support, Figure 6. The results show that due to the destruction of the lamellar structure of vermiculite, the lowest CO₂ capture values were obtained when using the fraction of smaller granulometry as support.

**Kinetic of CO₂ capture (isoconversional integral method)**

The isoconversional method was used to study the kinetics of CO₂ capture by vermiculite (particle size 300 μm < φ < 1180 μm) impregnated with CaO (2:1). Figure 7 shows the conversion curves for the experiments carried out with different heating rates.

The isoconversional integral methods of Ozawa-Flynn-Wall (OFW) and Kissinger-Akahira-Sunose were used to determine the apparent activation energy of the CO₂ capture process. Figures 8 and 9 show the results obtained when using equations 4
and 5, respectively (only a few values of \( \alpha \) are shown in the figures).

Table 2 shows the values for the correlation coefficients and the apparent activation energy calculated when using both integral methods, the results show good correlation coefficients when both methods are used.

Figure 10 shows the apparent activation energy values calculated for the CO2 capture process as a function of the conversion. As can be seen, for low conversions (\( \alpha < 0.3 \)), the apparent activation energy values decrease, suggesting a mixed controlling step (20 kJ < \( E_a \) < 40 kJ), i.e., chemical and diffusion steps occur at close rates so that the process kinetics depends on both. For conversion values greater than 0.3, the \( E_a \) values increase considerably, suggesting a chemical controlling step (\( E_a > 40 \text{ kJ} \)). The authors believe that at low temperatures the vermiculite is not fully expanded and, therefore, the diffusion of gas-phase into its lamellar structure is slower. As the temperature increases, the vermiculite expands and facilitates the diffusion of CO2 into the pores. As a consequence, the slow step in the process becomes just the chemical reaction between calcium oxide and CO2, which lead to higher activation energy values.

### Conclusions

In this work we have observed that the use of vermiculite impregnated with CaO enhanced the conversion of this oxide into CaCO3 from \( \approx 0.3 \) to \( \approx 0.7 \), employing a mixture of 75% CaO and 25% vermiculite. In addition, the greater conversion occurred for particle size in the range of 106 \( \mu \text{m} < \phi < 300 \mu \text{m} \). Our results suggest that the lamellar...
structure of vermiculite seems to collapse when the experiments are carried out with particles with granulometry smaller than 106 μm, diminishing the ability of the material to capture CO$_2$. The application of the kinetic methods of Kissinger-Akahira-Sunnose and Ozawa-Flynn-Wall provided reliable correlation coefficients. The apparent activation energy values showed that for low conversions ($\alpha < 0.3$) the controlling step of the process is a mixed step, depending on the chemical reaction and the diffusion of the reagents into the vermiculite, displaying rates of the same order of magnitude ($20 \text{kJ} < E_a < 40 \text{kJ}$). When the conversions values, $\alpha$, is greater than 0.3, the apparent
activation energy values suggest that the diffusion process no longer affects the slow step, being a chemical-dependent step ($E_a > 40$ kJ).

**Table 2. Apparent activation energy and correlation coefficients for the OSW and KAS methods.**

| Conversion | OFW method | KAS method |
|------------|------------|------------|
|            | $E_a$ (kJ mol$^{-1}$) | $R^2$ | $E_a$ (kJ mol$^{-1}$) | $R^2$ |
| 0.15       | 67.72      | 0.9383     | 43.86      | 0.9266     |
| 0.20       | 49.54      | 0.9857     | 23.67      | 0.9786     |
| 0.25       | 28.72      | 0.9873     | 39.82      | 0.9461     |
| 0.30       | 48.33      | 0.9626     | 66.45      | 0.9943     |
| 0.35       | 69.03      | 0.9954     | 71.10      | 0.9999     |
| 0.40       | 79.43      | 0.9999     | 73.56      | 0.9908     |
| 0.45       | 82.63      | 0.9934     | 90.85      | 0.9969     |
| 0.50       | 100.17     | 0.9975     | 89.49      | 0.9932     |
| 0.55       | 98.84      | 0.9927     | 88.71      | 0.9932     |

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**Data availability statement**

https://osf.io/xkr9p/?view_only=646236823cf84207bbf4ecc5a7d78d14
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**Disclosure statement**

No potential conflict of interest was reported by the authors.
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