

Optimization of mechanical milling process to obtain MgO modified with γ -Al₂O₃ and improve its CO₂ adsorption capacity

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Abstract: Mesoporous MgO modified with γ -Al₂O₃ samples were prepared via mechanical milling and evaluated for CO₂ capture under different temperature (30-200 °C) and pressure (2-20 bar) adsorption conditions. The effects of methanol as a process control agent and the effects of milling time over microstructure, textural and CO₂ adsorption properties of the samples were also evaluated. MgO modified with γ -Al₂O₃ obtained for 4h, with methanol, exhibited a maximum CO₂ adsorption capacity of 20.06 wt.%(4.55 mmol CO₂/g) at 30 °C and 20 bar, in comparison with sample obtained without methanol under the same conditions, which had an uptake capacity of 6.44 wt.%(1.46 mmol CO₂/g). This considerable uptake capacity of the sample obtained in the presence of methanol is due to the defects created in its crystalline lattice, which allow an increase in its textural properties, such as high specific surface area, pore area and pore volume.

Key Words: CO₂ adsorption; MgO modified with γ -Al₂O₃; mechanical milling; textural properties; structural properties.

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I. Introduction

In recent years, the CO₂ capture and storage technologies of fossil fuel-based power plants have garnered a great deal of attention, since they are an efficient measure to prevent global warming [1-2]. In this context, in order to mitigate the harmful effects of global warming on ecosystems and living beings, global strategies have been proposed to reduce the high atmospheric concentrations of CO₂ [3]. Consequently, many methods for CO₂ capture have been recently explored, such as amine scrubbing, separation membranes, cryogenic fractionation, adsorption systems, among others [4]. The adsorption method is particularly promising, owing to its superior advantages, such as no liquid waste, low energy consumption and a wide range of operating temperatures [3,5,6]. Specifically, for CO₂ adsorption technologies, a great variety of solid sorbents, such as zeolites [7], carbon-based materials [8-10], hydrotalcites [11], organic-inorganic hybrids, metal organic frameworks [12], metal oxides [5,13-15] and oxide composites [16,17] have been widely studied. Among the aforementioned adsorbents, metal oxides such as MgO are promising candidates for CO₂ capture, due to its abundant source and low cost. MgO can adsorb CO₂ and can be regenerated at relatively low temperatures (<200 °C), in comparison with CaO, for example [18,19]. Additionally, it is recognized that water vapor in flue gas is favorable for the adsorption of CO₂ by MgO [20]. Pure MgO presents drawbacks, such as low surface area-to-volume ratio and formation of an undesired carbonate layer on its surface, which is an impediment for further interaction between MgO and CO₂ [21]. Thus, the CO₂ adsorption capacity of MgO is correlated with its textural and structural properties, allowing a more efficient contact between the active sites of MgO and CO₂ [22,23]. It has been observed that porosity, high surface area, small particle size and the crystalline structure of MgO enhance CO₂ capture capacity [24]. Introducing Mg species into porous supports such as alumina, silica, active carbon, among others, promote the exposure of more basic active sites and improve adsorption towards acidic CO₂ molecules. Previously, it has been reported that MgO/Al₂O₃ nanocomposites used as CO₂ adsorbents are ideal for low-temperature CO₂ capture in presence of water vapor [3]. The supported MgO/ γ -Al₂O₃ composite, with its advantages, can enhance interactions with natural acidic CO₂, increasing its adsorption capacity and selectivity. For this purpose, high-energy ball milling (known as mechanical milling) has been considered as a viable method for preparation of MgO modified with γ -Al₂O₃ with reduced particle grain size, on a nanometric scale (<100 nm), with homogeneous distribution, and supported on a metal matrix. Additionally, it allows production of bulk quantities of solid-state materials using simple equipment at room temperature [25-27].

In this work, mesoporous MgO modified with γ -Al₂O₃ powders were obtained via the high-energy mechanical milling from MgO and γ -Al₂O₃ powders used as raw materials. The textural and structural changes of these materials during the ball milling with and without process control agent were studied in detail. Samples were also tested for CO₂ uptake at different conditions of temperature and pressure. The results obtained were analyzed and widely discussed.

II. Material And Methods

2.1 Raw materials

Magnesium and alumina oxides were utilized as raw materials for the preparation of MgO modified with γ -Al₂O₃. MgO powder was obtained by oxidation of nanocrystalline Mg at 600 °C for 10 minutes, according to the report by Sánchez [28]. In a typical synthesis, 3g of Al(NO₃)₃·9H₂O (Merck; 99.9% purity) and 3g CO(NH₂)₂ (Merck; 99.9% purity), in a molar ratio of 1:1, were transferred directly into a 50 mL crucible and mixed with 1 mL distilled water to obtain a solution, which was afterwards heated at 110 °C in order to evaporate the water. After, the powder was calcined at 800 °C for 10 min in a muffle furnace. Alumina powder was previously obtained via the chemical-combustion method as reported by Granados et al. [29].

2.2 Preparation of MgO modified with γ -Al₂O₃

MgO modified with γ -Al₂O₃ were obtained under different conditions of mechanical milling. Separately, measured quantities of MgO and γ -Al₂O₃ powders, in a molar ratio of 3:1, were placed in a 50 mL stainless-steel container with stainless steel balls as milling media. The mixtures were mechanically milled for 2 and 4 h, at ball-to-powder ratio of 10:1, in a high-energy Spex 8000 ball mill. These samples were obtained and denoted as MA1 and MA2, respectively. For MgO modified with γ -Al₂O₃ obtained at the same milling time with methanol as process control agent (PCA), these were denoted as MA3 and MA4, respectively.

2.3 Characterization

The alumina, MgO and MgO modified with γ -Al₂O₃ powders were characterized by using different analytical methods. X-ray diffraction (XRD) patterns for the as synthesized samples were recorded using a Bruker D8 Discover instrument coupled to CuK α ($\lambda=1.5406$ Å) radiation in the 2 θ range (from 20° to 90°) at 35 kV and 25 mA, with a step of 0.035°/6 seconds. The average crystallite size was estimated by the method of broadening of X-ray diffraction peaks using the Debye-Scherrer equation. The sample morphology was analyzed by scanning electron microscopy (SEM), using a Jeol JSM-6610LV coupled to an energy dispersive spectroscopy (EDX) system. Sample textural properties were examined by N₂ adsorption-desorption measurements at 77 K using a Belsorp Max (BEL Japan INC) instrument. The Brunauer-Emmett-Teller (BET) method was applied in order to obtain the nitrogen adsorption-desorption isotherms and to calculate the specific surface area, average pore diameter and pore total volume of the samples. The pore volume and pore area from the desorption branch of the isotherm were analyzed via the Barrett-Joyner-Halenda (BJH) method. It must be said that all samples were degassed at 350 °C for 2 h in a high-purity N₂ atmosphere before the N₂ adsorption measurements.

2.4 CO₂ adsorption experiments

MgO modified with γ -Al₂O₃ obtained at 4 h with and without PCA (MA2 and MA4) were tested for CO₂ adsorption. Prior to CO₂ adsorption experiments, the samples were previously heated at 500 °C for 20 min in a muffle to eliminate the pre-adsorbed species (H₂O and CO₂) present in the atmosphere. About 50 mg of the powder for each sample was carried out by using a 50 mL-capacity Parr 4592 stainless-steel microreactor, by exposing the powder samples to extra-dry carbon dioxide (99.8% purity) for 1 h (as a constant saturation time), at different temperatures (30, 50, 100 and 150 °C), and pressures of 2, 5, 10, 15 and 20 bar. The CO₂ adsorption capacities (wt.% of adsorbent) of the as-prepared MgO modified with γ -Al₂O₃ were determined by thermogravimetric analysis with a simultaneous TGA-DSC system, using an SDT Q600 calorimeter (TA Instruments-Waters LLC), previously calibrated. The samples were heated from room temperature to 700 °C at a heating rate of 10 °C/min under a high-purity helium atmosphere, with a flow of 100 mL/min.

III. Results and Discussion

3.1 XRD analysis

The X-ray powder diffraction patterns of alumina, MgO, used as starting materials, and MgO modified with γ -Al₂O₃ prepared via mechanical milling, are shown in Figure 1. Figure 1(a) shows the diffraction pattern of the alumina obtained by solution combustion at 800 °C for 10 min, showing only Bragg reflections that exhibit a marked broadening of all of the diffraction peaks, this sample was identified, and confirms an amorphous phase that corresponds to γ -Al₂O₃ (JCPDS file 001-1307). These results are consistent with previous report of γ -

Al₂O₃ materials prepared by solution-combustión[29].Figure1(b) shows the XRD pattern of the MgO sample obtained by a direct oxidation reactionof nanocrystalline Mg at 600 °C for 10 minutes. The sharp and well defined peaks which correspond to MgO crystalline phase known as periclase(JCDPS file 009-5447).According to the literature[30], this MgO phase crystalline was obtained by solution combustion.Also, sample MgO exhibits a marked broadening of all the diffraction peaks, suggesting the existence of small crystallite sizes in the existing crystalline phase. It was estimated that their average crystallite size was of 17 nm.Figures 1(c) and 1(d), shows the XRD patterns of MA1 and MA2 samplesprepared for 2 and 4 h of millingwithout PCA. Both samples, were indexed, as main phase, to crystalline phaseMgO with a cubic structure, and another crystalline phase of low intensity, attributed to thestructure of magnesium aluminium oxide (MgAl₂O₄), this phase is also known as spinel (JCPDS file 021-1152). This is due that during the milling, without lubricating agent, the powders react by the reaction of the solid state between MgO and γ -Al₂O₃oxides to form the ternary oxide. After 4 h of milling it is observed that the intensity of the MgAl₂O₄phase increases slightly with respect to the sample prepared for 2 h.Accoding to literature the secondary phase MgAl₂O₄ appeared when the Al₂O₃ was doped with 5 wt.% MgO [30].Comparing Figures 1(c) and 1(d) with the MgO XRD pattern (Figure 1(b)), it is clear that the MgO all difraction peaks heighten and broaden; this change is due to the decrease in grain size as a result of the milling process.For these MA1 and MA2 samples, the average crystallite size for the MgO phase, were 15 and 13nm, respectively.The XRD patterns of MA3and MA4 samples obtained for 2 and 4 h of milling with PCA are shown in Figures 1(e-f).As can be observed the diffractogramsshow only the characteristics peak of MgO, whereas the peaks corresponding to the MgAl₂O₄ phase disappear, this could be due to the complete dissolution of γ -Al₂O₃ in the MgO phase. The milling processes of MgO and γ -Al₂O₃ with PCA form the solid solution and the decrease in grain size. The MgO powder produced at 2 and 4 h have crystallite sizes of 14 and 12 nm, respectively, as determined by the XRD line-broadening technique.

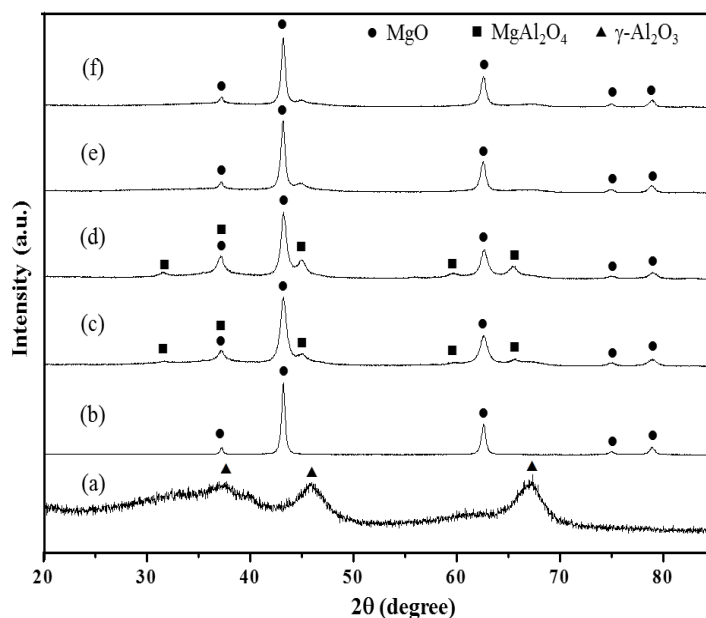


Figure 1. XRD patterns of : a) γ -Al₂O₃, b) MgO,c-d) MgO modified with γ -Al₂O₃ without PCA and e-f) MgO modified with γ -Al₂O₃ and PCA, obtained for 2 and 4 h by mechanical milling.

3.2 SEM analysis

The MgO, γ -Al₂O₃ and the MgO modified with γ -Al₂O₃at different millingtimes, with and without PCA, were analyzed by scanning electron microscopyto observe their microstructural morphologies. Figure 2(a) showsthe morphology of the γ -Al₂O₃ sample obtained by chemical-combustion at 800°C for 10 minutes,constituted by homogeneous particles of smooth and porous surface, withparticle size greater of approximately 25 μ m. The MgO powder obtained by oxidation at 600°C for 10 minutes(Figure 2(b)),exhibited homogeneous particles with irregular shape and porous structure; the particle sizewas less than 5 μ m and formed agglomerates of fine particles, characteristic of annanocrystalline material. Figures 2(c) and 2(d) showstheimages of the MA1 and MA2 samples in whichitsmorphology is different with regard to the MgO and γ -Al₂O₃startingpowders.The SEM image shows that the milled MgO modified with γ -Al₂O₃particles become smaller with sizes of less than 3 μ m and irregular shape.The SEM images of MA3 and MA4 samples (Figures 2(e-f)) shows a very similar behavior in their morphology with respect to the MA1 and MA2 samples. However, it is observed in these samples that they are more porous, with particles sizes between 1 and 5 μ m and form

agglomerates of smaller sub-particles. It must be pointed out that the milling process with a lubricant agent favour the formation of MgO modified with γ -Al₂O₃ with mesoporous structure and small grain size. To identify the dispersion of the elements, present in MA2 and MA4 samples, an elemental mapping, obtained by EDX, was performed.

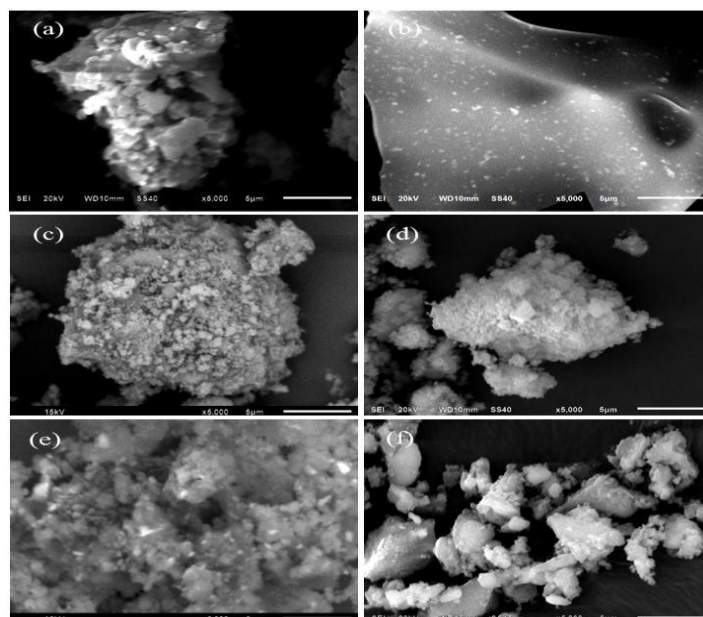


Figure 2. SEM images of: a) γ -Al₂O₃, b) MgO, c-d) MgO modified with γ -Al₂O₃ without PCA and e-f) MgO modified with γ -Al₂O₃ and PCA, obtained for 2 and 4 h by mechanical milling.

Figures 3(a) and 3(b) suggest a homogeneous dispersion for oxygen, aluminum and magnesium over the surfaces of the MgO modified with γ -Al₂O₃. Figures 3(c) and 3(d) show the EDX spectra of the MA2 and MA4 samples. Mean peaks of higher intensity correspond to O, Al and Mg, of which the composites are formed. The presence of carbon as impurity, is due to the methanol used as PCA during the mechanical milling.

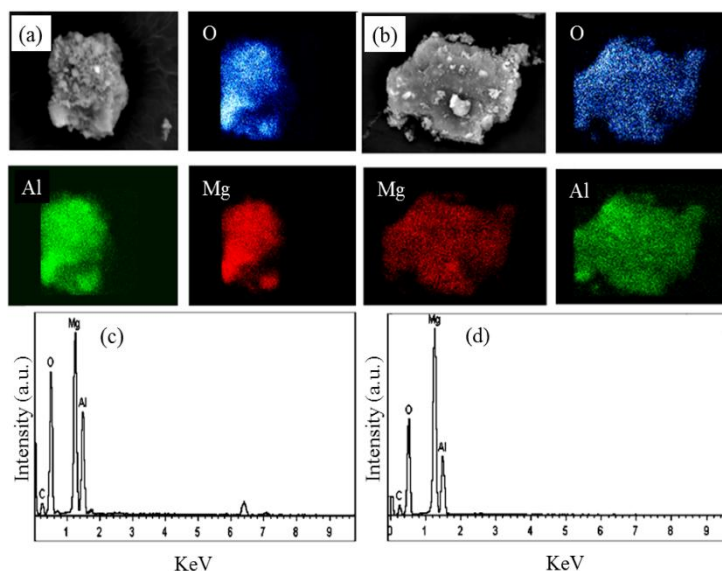


Figure 3. SEM images of elemental mapping of MgO modified with γ -Al₂O₃: a) without PCA, b) with PCA for samples obtained for 4 h by mechanical milling and c-d) their EDS spectra.

3.3 N₂ Physisorption analysis

Figure 4 shows the N₂ adsorption-desorption isotherms all the samples. The MA1 and MA2 samples obtained for 2 and 4 h of milling without process control agent, show isotherm type II, having a well-defined plateau, and exhibit the characteristics of microporous materials, however, the curves present a very small hysteresis loop. From MA3 and MA4 samples obtained at 2 and 4 h of milling with process control agent it could

be seen that the samples displayed isotherms type IV with H3 hysteresis loop according to the IUPAC classification, indicating the presence of micropores associated with mesopores. Hysteresis indicates the existence of mesopores that are characteristic of solids formed by agglomerates or aggregates of particles. The values obtained in pore size correspond to mesoporous materials with pore sizes ranging from 2 to 50 nm[31].

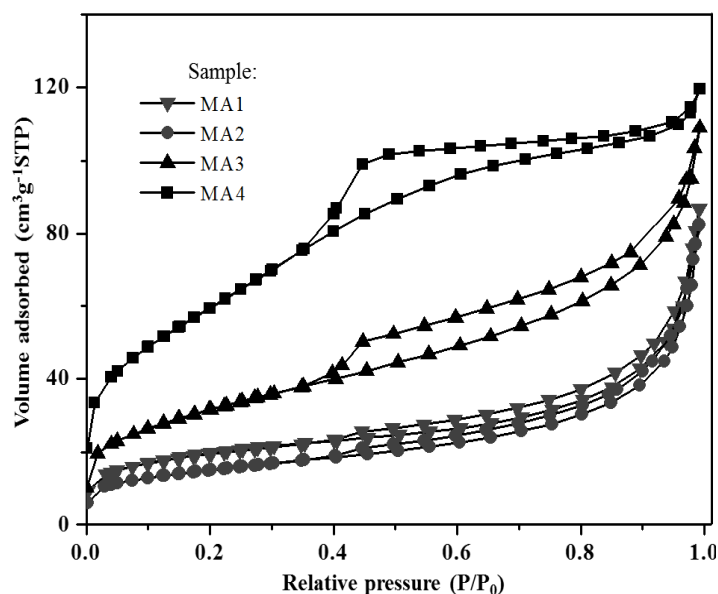


Figure 4. Nitrogen adsorption-desorption isotherms of MgO modified with γ -Al₂O₃ samples obtained at different conditions via mechanical milling.

Table 1 shows the textural properties of MA1, MA2, MA3 and MA4 samples. The results of the specific surface area, average pore diameter and total pore volume were obtained by using the BET method. For MA1 and MA2 samples, it was observed that when the milling time increased, the specific surface area and the total pore volume decreased, but its average pore diameter increased; this could be due to the fact that during the impact of the ball milling with the oxide powders, the particles were compacted and the pores of the oxides were closed. However, with the BJH method, the pore area and pore volume values were very similar. With regard to the MgO modified with γ -Al₂O₃ obtained with PCA (MA3 sample), a different behavior was observed during the milling process; in this case, the presence of the lubricant avoided agglomeration and reduced the effect of cold melting. The specific surface area and total pore volume determined by the BET method increased; however, its average pore diameter decreased. Similar results for the pore area and pore volume determined by the BJH method were observed. For MA4 sample, it was observed that the specific surface area and pore area doubled in comparison with MA3 sample. In this case, the effect of milling time is another important parameter to consider during the process of mechanical milling. Table 1 shows the results of the textural properties of MgO modified with γ -Al₂O₃ obtained with and without PCA. The mechanical milling, with the addition of methanol as a process control agent and increase in milling time, improved the milling process by reduction in grain size, creating more active sites and abundant defects, as well as increasing the specific surface area, total pore volume, pore area and pore volume.

Table 1: Textural properties of CO₂ adsorption behavior on MgO modified with γ -Al₂O₃ obtained without and with process control agent for 2 and 4 h via mechanical milling.

Sample	Milling time (h)	BET			BJH	
		Specific surface area (m ² /g)	Average pore diameter (nm)	Pore total volume (cm ³ /g)	Pore area (m ² /g)	Pore volume (cm ³ /g)
MA1	2	71.18	7.41	0.132	35.38	0.115
MA2	4	54.39	9.11	0.124	35.64	0.114
MA3	2	114.38	5.78	0.165	79.07	0.146
MA4	4	221.37	3.31	0.184	159.40	0.148

3.4 CO₂ capture performance of MgO modified with γ -Al₂O₃

3.4.1 TGA analysis

Based on the N₂ physisorption and XRD results, tests for CO₂ adsorption were performed in MA2 and MA4 samples. These samples, exposed to a CO₂ atmosphere, were denoted as MA2-CO₂ and MA4-CO₂ respectively, and were evaluated by thermogravimetric analysis. The results show that for MA2-CO₂ sample, the effect of pressure does not influence the adsorption capacity at 30 °C and 1 h of contact time, what was observed from the TGA curves (Figure 5(a)), however, it must be noted that at a pressure of 15 bar, a total weight loss of 7.04 wt.% of CO₂ (1.60 mmol CO₂/g) was achieved. As can be noticed the MA4-CO₂ sample (Figure 5(b)) had a different behavior in CO₂ weight loss. At 2, 5 and 10 bars, the total loss weight is ~ 16.4 wt.%, but when pressure was increased at 15 and 20 bars at 30 °C for 1 h of contact time, the capture capacity increased to a total loss weight of 17.20 (3.90 mmol CO₂/g) and 20.06 (4.55 mmol CO₂/g) wt.%, respectively. Figures 5(c) and 5(d) show the TGA curves of MA2-CO₂ and MA4-CO₂ samples, subjected to a different temperatures. The MA2-CO₂ sample had a maximum capture capacity of 9.84 wt.% CO₂ (2.23 mmol CO₂/g); was achieved at 50 °C, however, as temperature increased, the adsorption capacity decreased. For the MA4-CO₂ sample, the CO₂ capture temperature was increased (30-150 °C), the adsorption capacity remained constant with an average value of 16.21 wt.% (3.69 mmol CO₂/g). From TGA analysis, it must be pointed out that the total weight loss for all samples is associated with full CO₂ desorption. The samples show the same decay profile, showing two types of weight loss (wt.%), which means that the samples are independent of CO₂ sorption pressure and the temperature. This desorption behavior is associated their specific structural characteristics, and show the same chemical desorption species. The first stage of decomposition is observed from room temperature to 300 °C, which corresponds to the CO₂ physisorbed on the MgO modified with γ -Al₂O₃ surface. The second stage of decomposition is observed in the range of 300 to 700 °C, which corresponds to residual carbonate decomposition. As can be seen, the MA4-CO₂ sample has the higher efficient CO₂ capture, due that the mechanical milling process for 4 h with PCA modified its properties, such as size grain, specific area, dislocations, pore volume and pore area, which improved their CO₂ adsorption capacity with respect to MA2-CO₂ sample obtained without PCA. The MA1-CO₂ and MA3-CO₂ samples (figure not shown) showed the same weight decomposition profile by TGA, as in the case of the MA2-CO₂ and MA4-CO₂ samples. The total weight loss for these samples was 7.88 wt.% (1.79 mmol CO₂/g) and 14.7 wt.% (3.36 mmol CO₂/g) when exposed to 30°C under CO₂ pressures of 2 bar, respectively. Other authors (e.g. Chen, *et al.* [10]) obtained a Mg/carbon sphere nanocomposite via the solid-state grinding method with a CO₂ adsorption capacity of 3.08 mmol/g at 1 bar of pressure and 25 °C; Zhang, *et al.* [4] obtained MgO/ γ -Al₂O₃ with a molar ratio of 2, reaching a maximum CO₂ adsorption capacity of 1.06 mmol/g at 1 bar and 60 °C.

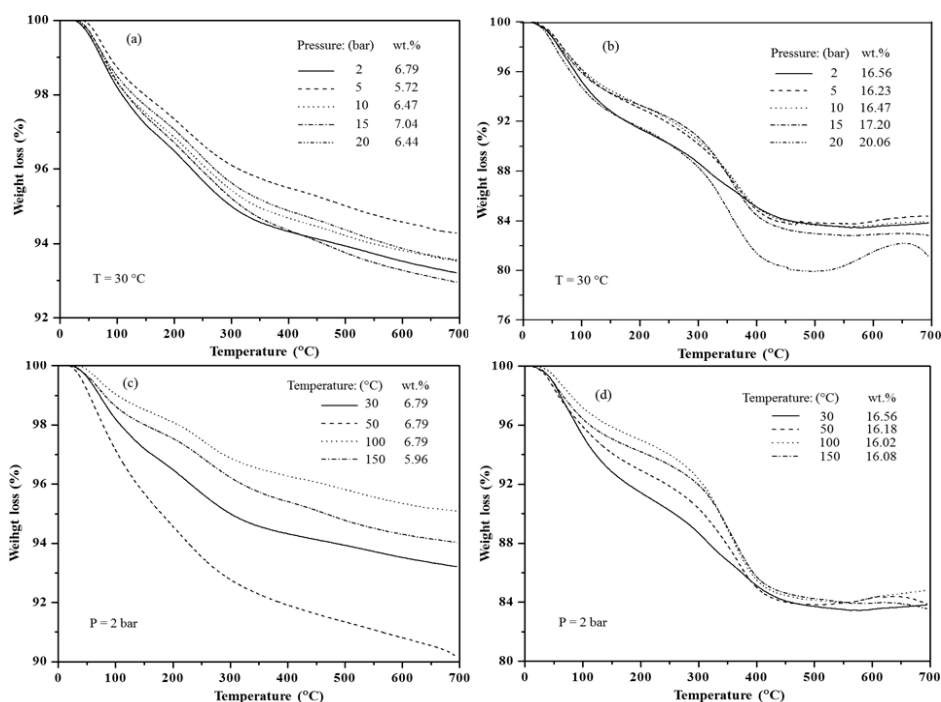


Figure 5. TGA curves for the MgO modified with γ -Al₂O₃ (MA2-CO₂ and MA4-CO₂ samples) after sorption of CO₂: a-d) at different pressures and temperatures.

3.4.2 DSC analysis

Figures 6(a-d) correspond to the heat flow curves obtained at different pressures and temperatures for MA2-CO₂ and MA4-CO₂ samples. As can be noticed, not endothermic peak was identified during the process of CO₂ desorption. It means that the release of CO₂ in the mixture of oxides is due to a process of physisorption. All the heat flow curves obtained at different pressures and temperatures show similar decay profiles of the CO₂ desorption process. For MA4-CO₂ sample an exothermic peak between 570-580 °C is observed, this can be due to the chemical reaction between MgO and γ -Al₂O₃ to form the MgAl₂O₄ during the desorption of CO₂, showed by simultaneously differential scanning calorimetry-thermogravimetric analyses and confirmed by the XRD results (Figure 7).

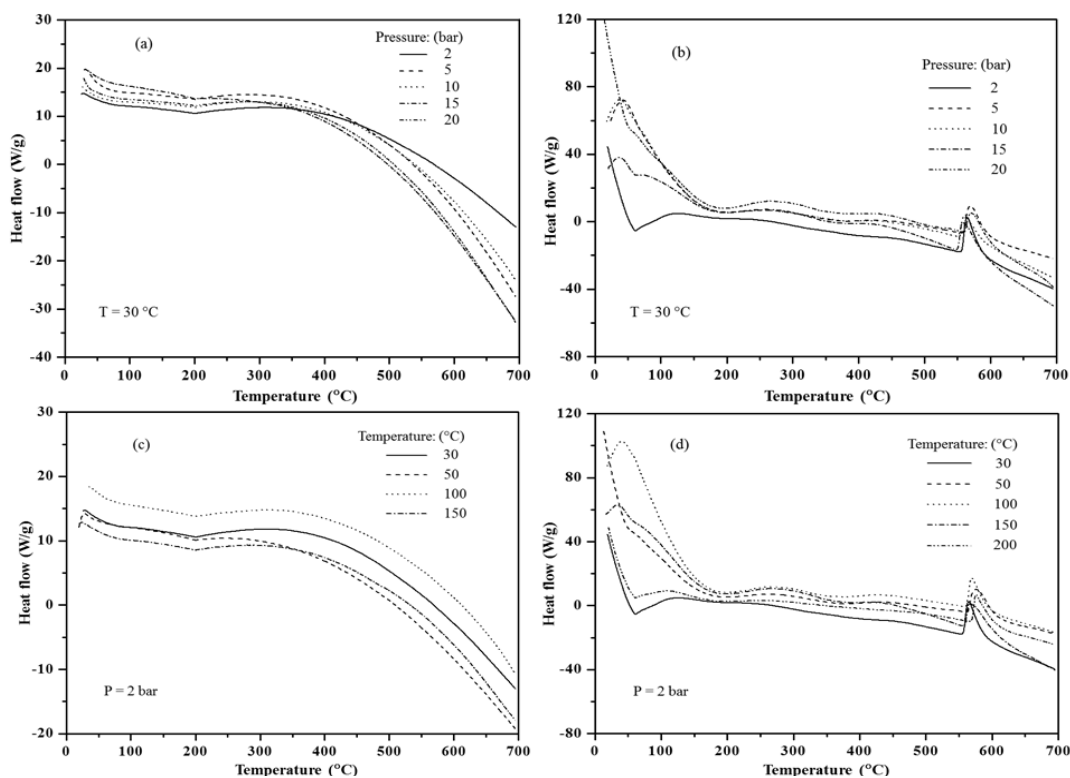


Figure 6. DSC curves for the MgO modified with γ -Al₂O₃ (MA2-CO₂ and MA4-CO₂ samples) after sorption of CO₂: a-d) at different pressures and temperatures.

3.5 Characterization of samples subjected to CO₂ desorption process

3.5.1 XRD analysis

To identify the crystalline phases present in the samples after CO₂ desorption process by TGA-DSC, (denoted as MA1D, MA2D, MA3D y MA4D) were characterized by XRD. The XRD pattern of the MA1D sample (Figure 7(a)) shows the main crystalline phase of MgO, and in less intensity, the Al₂O₃ phase. The crystallite size for the main phase of MgO was 15 nm. In the diffractogram of MA2D sample (Figure 7(b)), the crystalline phase of MgO was observed and, in less intensity, the crystalline phases of Al₂O₃, MgCO₃ and C. For this phase of MgO, the crystallite size was 14 nm. For MA3D sample (Figure 7(c)), MgO was also identified as the main phase and in lower intensities, the phases of Al₂O₃, MgCO₃ and C. The crystallite size for the MgO phase was 12 nm. The XRD pattern for MA4D sample is shown in Figure 7(d), in which the main phase corresponds to MgO; in which the other phases in minor intensities were identified as Al₂O₃ and MgAl₂O₄. The average crystallite size for the main phase of MgO was 10 nm. It must be mentioned that the crystallite size of the main phases was determined by using the Debye-Scherrer equation.

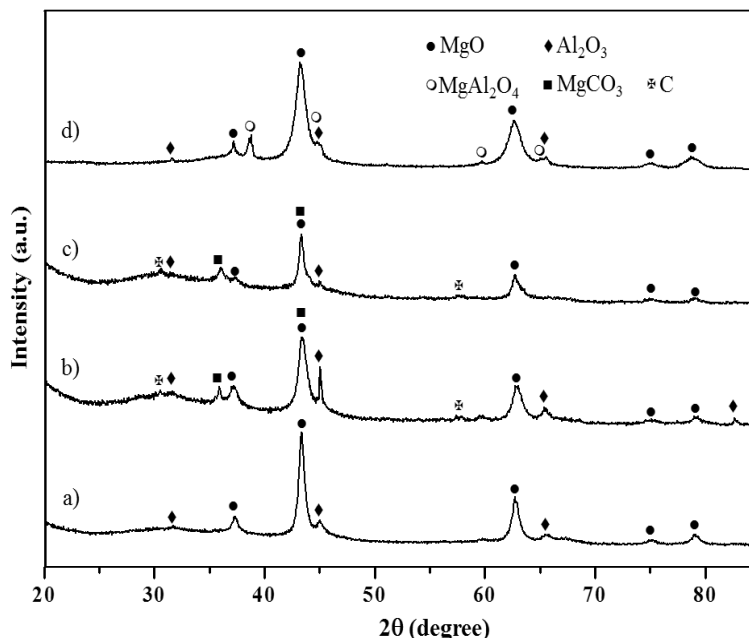


Figure 7. XRD patterns of MgO modified with γ -Al₂O₃ sample subject to CO₂ desorption process.

IV. Conclusions

MgO modified with γ -Al₂O₃ samples were obtained by high-energy mechanical milling from MgO and γ -Al₂O₃ powders. The addition of methanol as a process control agent (PCA) during the milling process increased its textural and microstructural properties such as the specific surface area, pore area and pore volume with respect to the sample obtained without PCA. The milling time favored the reduction in grain size to a nanometric scale and the creation of dislocations in the crystalline lattice, which helped to improve the adsorption processes of CO₂ in the MgO modified with γ -Al₂O₃. The main phase for the samples obtained at 2 and 4 h of mechanical milling with and without process control agent was MgO. The results of the thermogravimetric analysis showed that the samples obtained with and without PCA have great affinity and selectivity for CO₂ adsorption at low temperatures and moderate pressures. The MgO modified with γ -Al₂O₃ and obtained at 4 h of milling with methanol reached the best sorption of CO₂ with a maximum capture of 20.06 wt.% (4.55 mmol CO₂/g). MgO modified with γ -Al₂O₃ has good CO₂ adsorption capacity, excellent stability, and can be prepared easily, which make it a viable alternative as a CO₂ capture technology in industrial applications.

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J. Bonifacio-Martínez, et al. "Optimization of mechanical milling process to obtain MgO modified with γ -Al₂O₃ and improve its CO₂ adsorption capacity" *IOSR Journal of Environmental Science, Toxicology and Food Technology (IOSR-JESTFT)*, 13(3), (2020): pp59-67.