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Q2 MgO-based adsorbents for CO₂ adsorption: Influence 2 of structural and textural properties on the CO₂ 3 adsorption performance

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A B S T R A C T

A series of MgO-based adsorbents were prepared through solution–combustion synthesis and 18 ball-milling process. The prepared MgO-based powders were characterized using X-ray 19 diffraction, scanning electron microscopy, N₂ physisorption measurements, and employed as 20 potential adsorbents for CO₂ adsorption. The influence of structural and textural properties of 21 these adsorbents over the CO₂ adsorption behaviour was also investigated. The results 22 showed that MgO-based products prepared by solution–combustion and ball-milling 23 processes, were highly porous, fluffy, nanocrystalline structures in nature, which are unique 24 physico-chemical properties that significantly contribute to enhance their CO₂ adsorption. It 25 was found that the MgO synthesized by solution combustion process, using a molar ratio of 26 urea to magnesium nitrate (2:1), and treated by ball-milling during 2.5 hr (MgO-BM2.5h), 27 exhibited the maximum CO₂ adsorption capacity of 1.611 mmol/g at 25°C and 1 atm, mainly 28 via chemisorption. The CO₂ adsorption behaviour on the MgO-based adsorbents was 29 correlated to their improved specific surface area, total pore volume, pore size distribution 30 and crystallinity. The reusability of synthesized MgO-BM2.5h was confirmed by five 31 consecutive CO₂ adsorption–desorption times, without any significant loss of performance, 32 that supports the potential of MgO-based adsorbent. The results confirmed that the special 33 features of MgO prepared by solution–combustion and treated by ball-milling during 2.5 hr are 34 favorable to be used as effective MgO-based adsorbent in post-combustion CO₂ capture 35 technologies. 36

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52 Introduction

53 It is well documented that carbon dioxide (CO₂) is the most
54 significant greenhouse gas that mainly contributes to global
55 warming. In the past five decades, atmospheric CO₂ emissions
56 have been increased due to the accelerated industrial growth

and technological development, resulting in a potential threat 57
for the environment and ecosystems (Shafeeyan et al., 2015). 58
The main anthropogenic sources of CO₂ emissions are 59
attributed to the excessive fossil materials combustion such 60
as carbon, oil and natural gas in the energy production and in 61
many industrial processes (Bhagiyalakshmi et al., 2011). In 62

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this context, inevitably due to the fact that fossil fuels will be the predominantly worldwide power supply, it is necessary to take urgent measures to reduce the high CO₂ concentrations in the atmosphere at great scale (Ello et al., 2013). As a consequence, currently, there are different CO₂ capture technologies for these purposes, including adsorption process, membrane separation, cryogenic methods, and so on. Among CO₂ capture technologies, the CO₂ adsorption using porous solid adsorbents has been receiving much attention because of their advantages, such as low regeneration energy requirements, no liquid waste, low cost, both pressure swing adsorption and temperature swing processes are widely used (Harrison, 2004). Indeed, also has attracted the researchers' attention due to that can to be designed and studied a great variety of potential CO₂ adsorbents of varied nature, cheap, and renewable for use in large-scale post combustion technologies. In this scenario, many solid porous materials have been widely recognized and used as potential adsorbents for CO₂ adsorption, including activated carbons (Houshmand et al., 2012), zeolites (Siriwardane et al., 2005), amine compounds (Saiwana et al., 2014), organometallic networks (Liu et al., 2012), metal oxides (Busca and Lorenzelli, 1982), and hydrotalcites (Radosław et al., 2015), among others.

Amid these above adsorbents listed, metal oxides are promising options for suitable CO₂ adsorbents and have a great potential in the future due to their easy accessibility and favorable thermodynamic properties (Kumar et al., 2015). However, the selection criteria of potential metal oxides for a large-scale application, entail their CO₂ capture capacity, adsorption rate, thermal stability, regeneration heat, availability, cost, textural and structural properties (Kumar and Saxena, 2014). Specifically, the alkaline-earth metal oxide (MgO) is widely used in many technological applications as: catalysis, toxic waste remediation, additive in refractory, optically transparent ceramic windows, adsorbent for many pollutants, paint and superconductor products, among others (Jin et al., 2012). MgO is a white hygroscopic solid mineral that occurs naturally as periclase, is a source of magnesium, is a non-toxic and environmentally friendly material with higher surface reactivity and adsorption capacity, and is a suitable adsorbent for anions and cations from aqueous solution due to its favorable electrostatic attractive mechanism (Crittenden et al., 2005). In recent studies, MgO has been widely explored for CO₂ capture, and has been recognized as one of the most promising adsorbents for CO₂ adsorption together with CaO, because these metallic oxides combine with CO₂ present in the flue gas post-combustion and form thermodynamically stable carbonates in a reversible reaction; heating the carbonates regenerates the respective oxides and thus liberates almost pure stream of CO₂ that it can be directly transported to a storage site, and subsequently be used technologically (Kumar and Saxena, 2014). It has been reported that MgO can adsorb CO₂ below 200°C by carbonation, and can be regenerated by calcination at relatively low temperature compared with conventionally-used CaO subjected to cyclic CO₂ capture from flue gases, which makes it very desirable because it is widely accepted that there is a large scope for cost reduction and energy efficiency improvements in CO₂ capture systems (Wang et al., 2011). In general, many metal oxides can qualify for CO₂ capture application,

however, one of the key factors in gas–solid reaction is their textural, morphological and structural properties that are essential forenhancing their CO₂ adsorption. Thus, synthesis of MgO with improved specific surface area, nanocrystalline size, large total pore volume and narrow size pore distribution is of great interest as CO₂ adsorptive materials. The direct relation of these MgO physico-chemical properties with the CO₂ capture behavioris still not completely resolved and needs to be investigated.

On the other hand, extensive synthesis methods have been followed to obtain MgO products with porous structures and multiple morphologies, including the precipitation method (Janet et al., 2007), hydrothermal synthesis (Zhao et al., 2011), and sol–gel method (Kim et al., 2005). It is well known that the employed synthesis method and the chemical precursors to prepare powders have a large influence on the materials textural and structural characteristics such as porosity, specific surface area, particle size, crystallinity, between others. Particular desirable properties of compounds in their use as adsorbents are mainly extensive specific surface area that means many active sites for contaminant adsorption and a higher porous nature, which plays an important role on CO₂ capture. Therefore, it is evident from the above that there is a need for a continuous improvement and development of new and potential adsorbents for gaseous, aqueous, and non-aqueous streams. Currently, the novel solution–combustion synthesis is widely used for preparing different oxide powders with high purity, high yield, large specific surface areas, and good quality mesoporous structures at short times compared to other conventional synthesis methods. Furthermore, this method is safe, simple and instantaneous for the facile fabrication of nanopowders, indeed, the solution–combustion synthesis requires simple equipment with energy saving (Toniolo et al., 2010). For this method are required high temperatures for the exothermic redox reactions and achieve the decomposition the metal salt and the organic fuel for the formation of products. Therefore, extensive studies have been conducted to investigate solution–combustion synthesis of many metallic oxide powders using urea as the most commonly used chemical fuel, and some other fuels such as: starch, glycine and sorbitol (Bhatta et al., 2015; Bai et al., 2011; Devaraja et al., 2014; Granados-Correa and Bonifacio-Martínez, 2014; Mantilaka et al., 2014).

On the other hand, recent advances in nanotechnology have made much interest in preparing metallic oxide nanocompounds. The mechanical ball-milling is an extensive method to obtain nanostructured materials; this mechanical ball-milling treatment allows to activate dry solids and, especially increase its specific surface area as well as improve properties (Janusz et al., 2010). Nanopowders show substantially enhanced adsorption characteristics superior to those of the conventionally prepared powders (Liang et al., 2001). It is important to note that the gas adsorption efficiency can increase with a decrease in the MgO powder size and crystallites. In fact, the high gas adsorption efficiency of nanoparticles is also caused by their large specific surface area, and structural defects on their surface. Furthermore, it has been shown that metallic oxides doped with doping-metals such as Fe, Ni, Cd, Ce, Co among others, show significant changes in their properties with respect to original materials. In

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