



The Amine-Functionalized MCM-41 for Hydration and Utilization of CO₂

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Article Info

Article type:

Research Article

Article history:

Received: 14 July 2023

Revised: 20 October 2023

Accepted: 25 December 2023

Keywords:*CO₂ adsorption MCM-41**Amine group**CaCO₃*

ABSTRACT

Carbon dioxide, as a great part of greenhouse gases, stands as a major contributor to climate change; hence, various techniques have been presented for controlling and decreasing CO₂ emissions. The studies show that the adsorption and conversion into environmentally benign substances are the most practical and efficient strategies for this purpose. As amines are active in CO₂ adsorption, N-(2-aminoethyl)-3-aminopropyltrimethoxysilane was chosen as an amine group to functionalize MCM-41 mesoporous in order to hydrate CO₂ and convert to calcium carbonate in this study. The synthesis of the mesoporous materials and amine functionalization were carried out using the wet impregnation method, and the materials were characterized by XRD, FT-IR, SEM, and BET analysis. The results demonstrated a high adsorption capacity (165 mg CaCO₃) due to the presence of the -NH₂ group in the catalysts. Moreover, the results have been compared with similar mesoporous materials functionalized with metals for CO₂ capture and hydration. The adsorption-desorption isotherm corresponded to type IV, as defined in the IUPAC classification.

Cite this article: Asadi, M., Azordeg Molkabadi, S., & Engameh, S. (2024). The Amine-Functionalized MCM-41 for Hydration and Utilization of CO₂. *Pollution*, 10 (1), 374-382.

<https://doi.org/10.22059/POLL.2023.362270.1992>



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Publisher: The University of Tehran Press.

DOI: <https://doi.org/10.22059/POLL.2023.362270.1992>

INTRODUCTION

The adverse effects of greenhouse gases on the environment are inevitable and carbon dioxide (CO₂) plays a major role here. Indeed, CO₂ is a permanent factor and forms 94% of GHGs, consequently, is the primary cause of global warming and climate change (Pirouzmand et al., 2018; Wnag et al., 2018). According to the limitation of fossil fuel resources and rising CO₂ emissions, researchers and scientists are exploring ways to reduce the concentration of CO₂ in the air (Zhang et al., 2018). There are suggested various techniques for CO₂ reduction, including 1) the use of renewable e.g., solar) and green (e.g., hydrogen) energy, 2) CO₂ capture and storage (CCS), and 3) CO₂ utilization (Roy et al., 2018). Of these strategies, CO₂ utilization is considered the most practical and efficient. Techniques for CO₂ utilization include CO₂ conversion (Wang et al., 2018; Singh et al., 2018), hydration (Pirouzmand et al., 2018; 2016), catalytic capture (Pirouzmand et al., 2016; Kim et al., 2018), photocatalytic reduction (Wang et al., 2023; Liu et al. 2020; Nosrati et al., 2023), hydrogenation (Li and Chen, 2019; Li et al., 2018; Liu et al., 2023), and direct air capture (DAC) (Brethome et al., 2018; Abdullatif et al., 2023; Sodiq et al., 2023).

Recently, CO₂ capturing by solid materials has been growing because of eliminating the need for solvents and the associated disposal processes. Typically, CO₂ adsorption on solids occurs at room temperature, and the captured CO₂ is released by regenerating the materials at temperatures ranging from 80 to 120 °C, which requires a significant amount of energy.

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Solid adsorbents are employed in two main capturing concepts: DAC, which directly extracts CO₂ from the atmosphere and capturing CO₂ from the emissions of industrial facilities like power plants and factories for chemical synthesis. DAC is often known as a “negative emission technology” because it goals to capture CO₂ already present in the atmosphere (around 417 ppm). However, it is less cost-effective compared to capturing CO₂ from industrial exhaust streams due to the lower concentration of CO₂ in the air, which presents a higher thermodynamic barrier (Hack et al., 2022).

The selection of a suitable support material is influenced by multiple factors. Two crucial attributes for this material include its ability to withstand high temperatures without decomposing and its resistance to chemical reactions with functional groups during the functionalization process. This ensures that the functional groups remain available for CO₂ chemisorption. Additionally, other properties come into play when fine-tuning the performance of adsorption and desorption. Key considerations include the specific surface area, porosity, and pore volume, all of which are interconnected with adsorption performance. The specific surface area is particularly significant as it facilitates the even distribution of functional groups on the support material. Support materials with higher surface areas can accommodate a larger quantity of functional groups, resulting in increased adsorption capacity (Hack et al., 2022). MCM-41 mesoporous is one of the support materials that fulfill these two criteria.

Pirouzmand et al. prepared Zn-containing mesoporous using direct synthesis (DS), wet impregnation (WI), and template ion-exchange (TIE) methods in order to catalyze the capture and hydration of CO₂. In this study, the functionalization of the mesoporous with Zn²⁺ was inspired by the carbonic anhydrase enzyme. The evaluations illustrated that zinc-based mesoporous materials are not only efficient in CO₂ capture and hydration to bicarbonate but can also be easily reused without requiring activation (Pirouzmand et al., 2016). In a similar study, we also reported on the activity of Mg and Ni/MCM-41, which were inspired by the RuBisCo enzyme and sea urchin, and were synthesized using DS and WI methods. Furthermore, the efficiency of prepared catalysts compared with MgO and NiO nanoparticles in hydration ability. The results demonstrated that the synthesized template-containing mesoporous by the DS method is more active in comparison with calcinated mesoporous and even nanoparticles (Pirouzmand et al., 2018). The achievements of these studies have shown the superiority of DS-synthesized mesoporous because of existing active basic sites to capturing and hydrating of CO₂.

Gao et al. synthesized a composite known as MCM-41@AMPD (2-amino-2-methyl-1,3-propanediol), as a new sterically hindered amine adsorbent to CO₂ capture, adsorption selectivity, and reusing stability. The results obtained from the modification of mesoporous materials with 7 mmol/g mesoporous of AMPD show that it is more active in CO₂ capturing and can be efficiently reused. Additionally, the adsorption selectivity of this composite is 35 times higher than that of pure MCM-41. The improvement of adsorption features by rising the content of AMPD up to 7 mmol/g mesoporous results in increasing the pore volume and surface area of mesoporous and ultimately results in conducting adsorption types from the chemisorption of -NH₂ groups with CO₂ molecules to physisorption (Gao et al., 2022).

In another study, secondary alkanolamine of 2-(ethylamino) ethanol (EAE), multi-alkylamines such as ethylenediamine (EDA), and tetraethylenepentamine (TEPA)/MCM-41 were prepared through WI so as to investigate CO₂ adsorption/desorption performance by *Muchan et al.* The evaluations illustrated that both CO₂ adsorption capacity and rate are affected by the type and content of amines. In this study, the highest adsorption capacity and rate, along with a 98% desorption efficiency, were observed in the case of 60 wt% EAE/MCM-4 at all stages (Muchan et al., 2022).

Studies have shown that MCM-41 is not very selective for CO₂ adsorption through physical adsorption driven by van der Waals force, unless it undergoes functionalization with active

groups such as amines. This modification enhances the interaction between the active sites and CO₂ molecules during adsorption. Because the existence of surface silanol groups has a vital role in the chemical adsorption of CO₂ on the surface of the mesoporous silica; in addition, selective adsorption of CO₂ molecules increases through the chemical adsorption of CO₂ into the active sites, forming carbamates or carbamic acids (Mukherjee et al., 2019). Amine-modified MCM-41 provides a solution to address problems often seen in liquid amine scrubbing, including the evaporation-related loss of volatile amines. Support materials with significant surface area and porosity are chemically modified with amines, ensuring even dispersion of the amines. Consequently, these modified materials combine the beneficial characteristics of both amine compounds and support structures. In this study, MCM-41 and NH₂-modification types of MCM-41 have been evaluated for CO₂ adsorption. In this method, the process of absorbing CO₂ and converting it to calcium carbonate is a simple system. First carbon dioxide enters the water, which contains a mesoporous modified by the amine group. CO₂ is adsorbed easily and then carbon dioxide is converted into calcium carbonate in the presence of calcium resource (CaCl₂·H₂O).

MATERIAL AND METHODS

Cetyltrimethylammonium bromide $\geq 98\%$ (CTAB), sodium hydroxide (NaOH), tetraethyl orthosilicate $> 98\%$ (TEOS) used for MCM-41 synthesis, n-Hexane, N-(2-aminoethyl)-3-aminopropyltrimethoxysilane (APTMS) used for amine-modification of MCM-41 (NH₂-MCM-41) synthesis, hydrochloric acid (HCl), sodium bicarbonate (NaHCO₃), tris(hydroxymethyl) aminomethane buffer ($\geq 99\%$), calcium chloride (CaCl₂·H₂O) used for CO₂ hydration to bicarbonate and utilization for calcium carbonate precipitation. The materials are supplied by Merck.

X-Ray diffraction (XRD) measurements were performed on a Philips-PW17C diffractometer with Cu K α radiation. The Scanning electron microscopy (SEM) images of the samples were determined by SEM AIS 2300C Seron Technology. Fourier transform infrared (FTIR) spectra were recorded on a Tensor 27 Bruker instrument using KBr pressed powder discs. The specific surface area of the materials was determined by a Micromeritics ASAP 2460 instrument.

MCM-41 synthesis

In a typical non-hydrothermal synthesis, 0.20 g (0.52 mmol) of CTAB was dissolved in 96 mL of NaOH aqueous solution (15.0 mmol/l), followed by the dropwise addition of 1 mL (4.48 mmol) of TEOS and stirred for 3 hours. In order to remove the cationic surfactant, the obtained powder was calcined in the oven at 550°C for 6 h (Asadi and Azordeg, 2020; Anbia et al., 2013).

Preparation of amine-modification mesoporous (NH₂-MCM-41)

To prepare NH₂-MCM-41 composites using WI method, add 20 ml of n-hexane to calcinated MCM-41 in the presence of N₂ gas, followed by dropwise 1.15 mmol APTMS as an amine precursor into the mixture, which then concentrates at 80°C in the oven for 24 hours. Ultimate hot product is filtered, washed with n-hexane and deionized water, and is dried at ambient temperature for a full day (Pirouzmand et al., 2018; 2016).

CO₂ hydration and utilization

The method described in the literature (Pirouzmand et al., 2018; 2016) was used to carry out the hydration and precipitation experiments. CO₂ hydration was performed at ambient temperature in the presence of 100 mg of MCM-41 and NH₂-MCM-41, which were dispersed in 10 mL of 1.0 M Tris-HCl buffer (pH 8.0), followed by the catalyst suspension was then added to 100 mL of a saturated CO₂ solution (130 mL CO₂; measured using soda lime). CO₂ hydration

was determined to be complete within 10 min as the pH stabilized at a constant value. The hydrated CO_2 solution was filtered to recover the MCM-41 and NH_2 -MCM-41 catalysts for the next cycle. The filtrate was then treated with 20 mL of 4% CaCl_2 solution at pH 10.0. After 120 min, precipitated CaCO_3 was obtained (Pirouzmand et al., 2018; 2016).

RESULTS AND DISCUSSION

The XRD patterns of MCM-41 and NH_2 -MCM-41 in Figure 1 indicated an intense reflection peak at $2\theta = 2.21^\circ$ and three weak peaks in the 2θ range of 3.93, 4.53, and 6.03° , assigned to the d_{100} , d_{110} , d_{200} , and d_{210} planes, respectively. That indicates the formation of well-ordered mesoporous materials with hexagonal regularity. As depicted, the structure of the mesoporous has been maintained because of the retention of the major reflection (100) after modification with the amine group ($-\text{NH}_2$). Additionally, the high-intensity of NH_2 -MCM-41 diffraction peaks is related to the well-scattered ratio between the channel wall and silicon framework (Miricioiu et al., 2019; Xu et al., 2023).

Figure 2 shows the FT-IR spectra of MCM-41 and NH_2 -MCM-41 functionalized using the

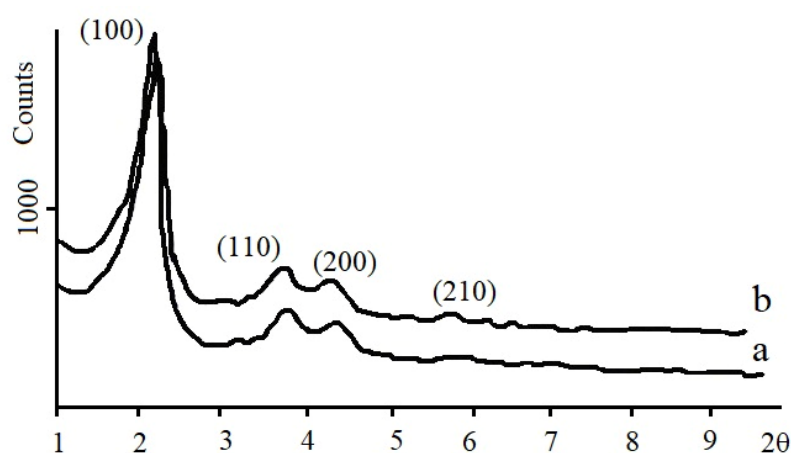


Fig. 1. XRD patterns of a) MCM-41 and b) NH_2 -MCM-41

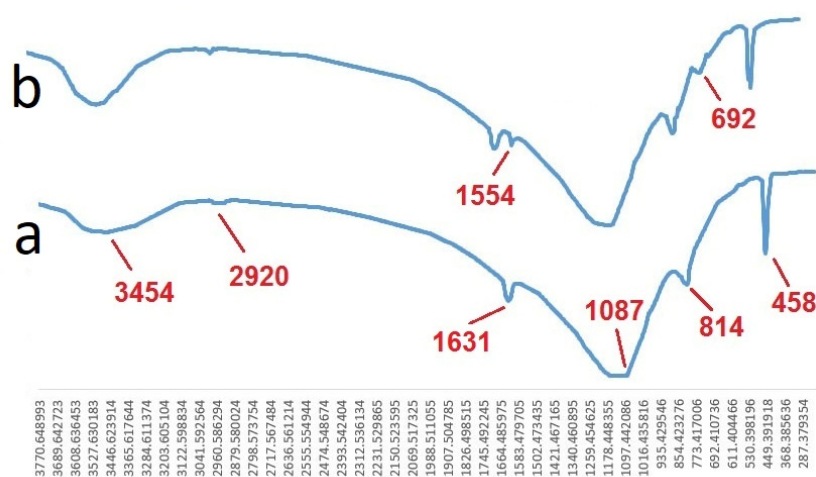


Fig. 2. FT-IR spectra of a) MCM-41 and b) NH_2 -MCM-41

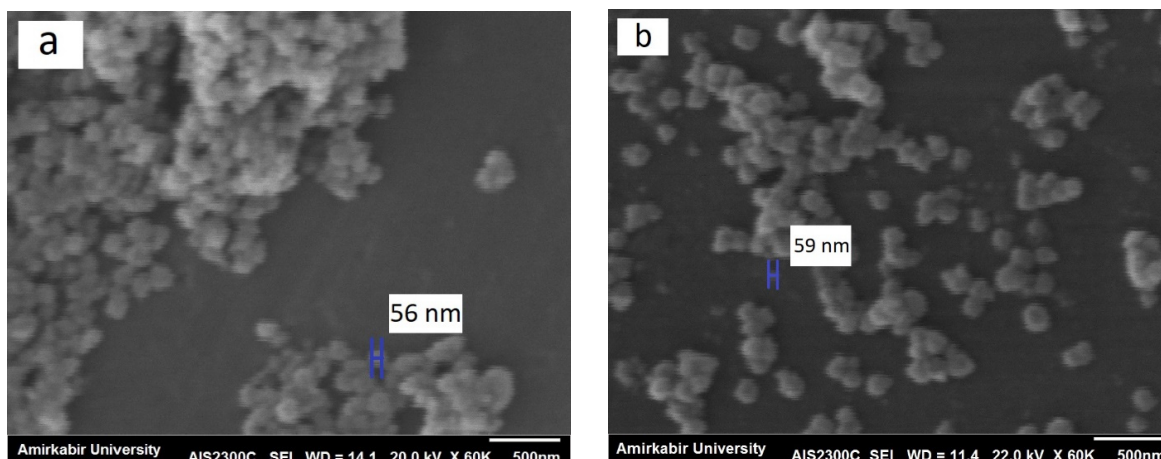


Fig. 3. SEM images of a) MCM-41 and b) NH_2 -MMC-41

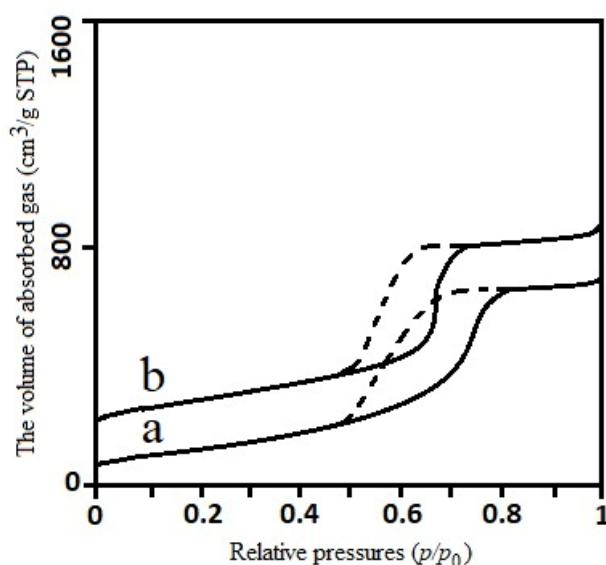


Fig. 4. The curves of N_2 adsorption-desorption isotherms; a) MCM-41 and b) NH_2 -MCM-41

WI method. The OH groups from the silanol surface or the physically adsorbed water on the surface can be seen at 3454cm^{-1} . The bands at 1087cm^{-1} , 814cm^{-1} , and 458cm^{-1} are assigned to Si-O-Si and Si-O vibrations. The identified peak at 1631cm^{-1} can be attributed to stretching vibrations of adsorbed water molecules. A weak absorption band at 2920cm^{-1} related to the C-H stretch vibration of the cationic template (CTAB), which is barely visible and is hidden here because the template has been removed by the calcination. As depicted in Figure 2 (a), after modification with the amine group, two medium-intensity novel peaks emerged that were not observable in the spectrum of MCM-41 (Figure 2 b). These peaks at regions 692cm^{-1} and 1554cm^{-1} are attributed to the bending vibration and symmetric stretching modes of $-\text{NH}_2$, respectively. Based on the FT-IR spectra, it can be concluded that the modification with the $-\text{NH}_2$ group has no effect on the pore walls (Miricioiu et al., 2019; Xu et al., 2023).

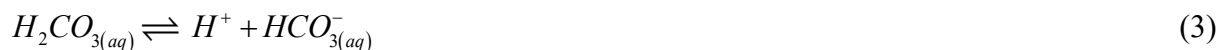
SEM characterization was carried out in order to examine the prepared mesoporous morphology (Figure 3). SEM images exhibit sphere-regular particles with average diameters of 56 nm for MCM-41 and 59 nm for NH_2 -MCM-41.

The curves of N_2 adsorption-desorption isotherms (Figure 4) correspond to a type IV isotherm as defined by IUPAC. There is a linear increase in the volume of absorbed nitrogen at relative

pressures (p/p_o) up to 0.6. The volume-enhancing effect is much greater at 0.68- 0.72 p/p_o , and it is related to the existence of capillary pores.

CO₂ hydration and utilization

The possible chemical reactions of CO₂ hydration to bicarbonate and the precipitation of calcium carbonate are as follows (Siqueira et al., 2017):



The second equation is the rate-determining step, and the catalyst plays a significant role. Other carbonates with alkaline metals, such as Mg²⁺, Na⁺, Ba²⁺, dawsonite (NaAlCO₃(OH)₂) are suggested as possible products of CO₂ hydration.

CO₂ adsorption capacity would improve in the presence of amine groups because of their strong adsorption properties and the basic-acid interaction between the amine group and acidic CO₂ via chemical adsorption (Pirouzmand et al., 2018). Besides, reports showed that due to enhanced amine's efficiency in the presence of humidity, the formation of ammonium bicarbonate/carbonate occurs in the presence of moisture.

According to the formation of bicarbonate/carbonate carried out after ammonium carbamate formation in Figure 5, the emergence of bicarbonate/carbonate from carbamate hydrolysis in CO₂ adsorption in the presence of primary and second amines performed through a concerted one-step mechanism. The transition state is characterized by nucleophilic attack of the CO₂

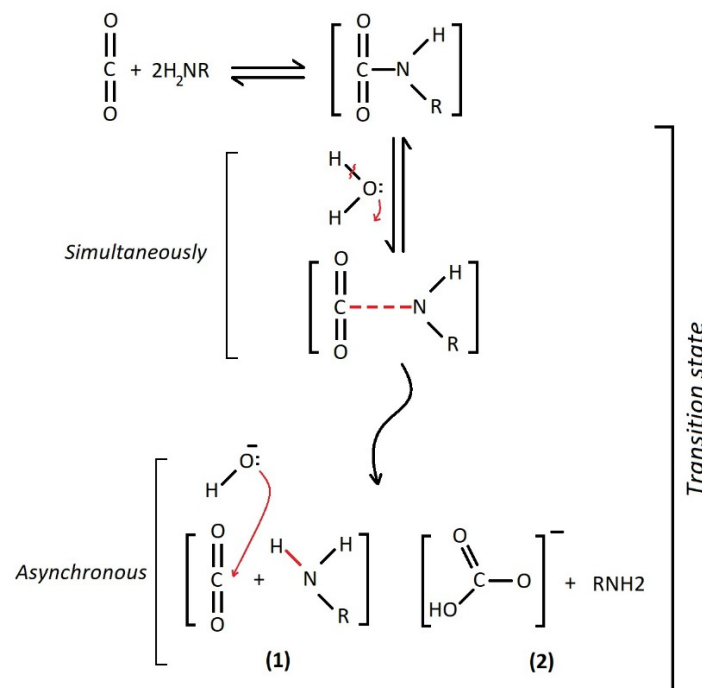


Fig. 5. Transition state; formation of bicarbonate and ammonium carbamate

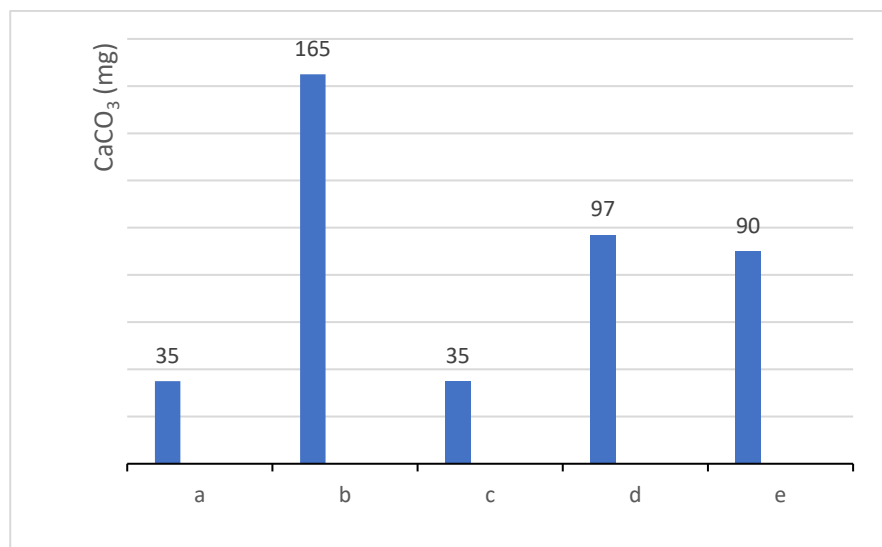


Fig. 6. Adsorption capacity of synthesized mesoporous: a) MCM-41, B) NH₂-MCM-41, c) Zn-MCM-41 (Pirouzman et al., 2016), d) Mg-MCM-41 (Pirouzman et al., 2018), e) Ni-MCM-41 (Pirouzman et al., 2018)

carbon by water oxygen, elongation of the carbamic acid C–N bond, and breakage of the O–H bond, all of which are performed simultaneously. Notably, the formation of carbonic acid occurs through a concerted but asynchronous process (in two steps), in other words, the proton from H₂O attaches to nitrogen before oxygen binds to the CO₂ carbon (Ben-Said et al., 2020). Finally, the carbonate solution was treated with 20 mL of 4% CaCl₂ solution according to the equations 4 and 5. After 120 min, precipitated CaCO₃ was obtained.

Figure 6 demonstrates the adsorption capacity of synthesized mesoporous materials. The experiments of hydration and precipitation were performed four times and the average content of CaCO₃ generation was reported in Figure 6. A comparison of adsorption capacity among amine-functionalized mesoporous and metal-modified mesoporous, which is inspired by Rubisco and CA enzymes, can be observed. In this work, calcinated MCM-41 (WI) illustrates the same adsorption capacity as previous work (35 mg CaCO₃) that was reported by *Pirouzman et al.* in 2016. Furthermore, the amine group increased the adsorption capacity of the catalyst by almost 71% which is 165 mg more than that of Mg and Ni/MCM-41 (WI), as reported in our former work (Pirouzman et al., 2018). As discussed, the adsorption route in non-modified MCM-41 is physical adsorption driven by van der Waals forces and is not very active in CO₂ adsorption; in contrast, the formation of carbamates or carbamic acids in the active sites by amine groups increases the chemical adsorption of CO₂.

CONCLUSION

CO₂ hydration process includes a series of chemical reactions, causing the conversion of CO₂ into bicarbonate, which ultimately results in the precipitation of calcium carbonate. These reactions are considerably affected by the presence of catalysts. Besides, the study underscores the crucial contribution of amine groups in enhancing the capacity for CO₂ adsorption. This improvement is attributed to the robust adsorption characteristics of amines and the chemical interplay between amines and the acidic CO₂, facilitated through chemical adsorption. In the present work, the mesoporous was synthesized by WI method as well as functionalized by the -NH₂ group in order to create active sites for CO₂ hydration and finally precipitation of calcium carbonate. The formation of bicarbonate/carbonate during the hydrolysis of carbamate

in the context of CO₂ adsorption. This process occurs through a one-step mechanism, involving nucleophilic attack, bond elongation, and bond breakage simultaneously. The formation of carbonic acid is noted to be a two-step process, where the proton attaches to nitrogen before oxygen binds to the CO₂ carbon. The adsorption-desorption isotherm type of the samples was determined as type IV based on the curve of N₂ adsorption-desorption, and the volume-enhancing is related to the existence of capillary pores. Meanwhile, the evaluations demonstrated the results of the study correspond to previous literature, and CO₂ adsorption is improved by amine modification. Likewise, the average content of CaCO₃ in precipitation is 165 mg, which is a little less than two times the generation capacity of metal-modified mesoporous in our former work.

ACKNOWLEDGMENT

The authors gratefully acknowledge the financial support of this study by Mr. M, Asadi.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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