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Enhanced CO_2 Adsorption Performance of Amine-Functionalized MCM-41 Mesoporous Silica

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ABSTRACT

Global carbon dioxide (CO_2) emissions continue to rise, driving an urgent demand for advanced carbon capture technologies. Mesoporous silica—specifically Mobil Composition of Matter No. 41 (MCM-41)—offers considerable potential owing to its ordered pore structure and high surface area. In this study, MCM-41 was synthesized using a sol–gel method and functionalized with two commercial amine-based surface modifiers, Hydrosil 2775 (S1) and Hydrosil 2999 (S2). Structural characterization by Fourier-transform infrared spectroscopy confirmed the successful grafting of amine groups onto the silica framework. Brunauer–Emmett-Teller analysis demonstrated a specific surface area of 334.42 m^2/g, confirming the retention of mesoporosity. CO_2 adsorption capacity was evaluated by direct dynamic mass-response measurements. The results demonstrated a marked mass increase, corresponding to a net uptake of approximately 22.8 mg/g. These findings highlight the effectiveness of amine-functionalized MCM-41 as a promising and readily scalable adsorbent for industrial carbon capture applications.

Keywords: MCM-41; CO_2 capture; amine functionalization; mesoporous silica; Hydrosil modifiers; gas adsorption

1. INTRODUCTION

The continuous increase in atmospheric carbon dioxide (CO_2) emissions from industrial activities and fossil fuel combustion has intensified the global need for effective carbon capture technologies. Carbon capture and storage represents a critical strategy to mitigate climate change by trapping emissions at their source. Among various approaches, adsorption onto solid porous materials has gained considerable attention owing to its energy efficiency and ease of regeneration[1,2]. Mesoporous silica materials, particularly Mobil Composition of Matter No. 41 (MCM-41), are highly promising supports for CO_2 capture owing to their high specific surface area, large pore volume, and well-ordered hexagonal pore structure[3]. However, pristine silica surfaces exhibit weak physical interactions with CO_2 molecules[4]. To overcome this limitation, chemical modification via amine functionalization is commonly employed. The grafting of amine groups onto the silica framework enhances the chemical affinity for CO_2, leading to markedly improved adsorption capacity and selectivity[5,6,13]. Previous studies have shown that the performance of amine-functionalized MCM-41 depends heavily on the type of amine used and the loading conditions[7,8]. In this study, two commercial amine-based modifiers—Hydrosil 2775 and Hydrosil 2999—were used to functionalize synthesized MCM-41. These modifiers offer a scalable and cost-effective approach for developing high-performance adsorbents. The primary objective of this study is to characterize the structural properties of the synthesized materials and evaluate their CO_2 adsorption performance through dynamic mass-response measurements. By analyzing the interaction between the amine-modified surface and CO_2 gas, this research provides insight into the potential of these materials for industrial-scale carbon capture applications. The structure of the remaining paper is as follows. Section 2 presents the materials and methods used in this study, Section 3 discusses the results and their interpretation, and Section 4 concludes the study.

2. MATERIALS AND METHODS

2.1. Materials and Synthesis Procedure. The synthesis and functionalization of the adsorbents were carried out using high-purity chemical reagents. MCM-41, a mesoporous silica with an ordered hexagonal structure, was used as the primary support framework[3]. Cetyltrimethylammonium bromide (CTAB, 98%, Sigma-Aldrich) was used as the structure-directing agent, while tetraethyl orthosilicate (TEOS, 99%, Sigma-Aldrich) served as the silica precursor. Sodium hydroxide (NaOH, Sigma-Aldrich) was used to establish the alkaline conditions required for hydrolysis and condensation reactions. MCM-41 was synthesized by dissolving CTAB in deionized water under continuous stirring, followed by the addition of NaOH and dropwise addition of TEOS at 80 °C. The mixture was maintained for 2 h to promote the formation of the silica network. The resulting precipitate was filtered, washed, dried at 60 °C, and calcined at 550 °C for 5 h to remove the surfactant template and obtain

Figure 1. Drying setup with stirring and heating

the ordered mesoporous structure[2]. For surface functionalization, the calcined MCM-41 was dispersed in deionized water, and the pH was adjusted to approximately 10. Hydrosil 2775 (S1) and Hydrosil 2999 (S2) were added separately under continuous stirring at 70 °C and allowed to react overnight to ensure effective grafting of amine groups onto the silica surface[8]. The modified samples were subsequently washed, centrifuged, and dried prior to characterization and CO_2 adsorption testing. The synthesis setup and the final product are illustrated in Figures 1 and 2.

Material properties are summarized in Table 1, highlighting key physical characteristics and functions under identical testing conditions.

2.2. Characterization Techniques. The structural and chemical properties of the samples were analyzed using FTIR spectroscopy and BET analysis. FTIR spectroscopy was used to verify the presence of the silica framework and confirm the successful incorporation of amine groups following functionalization. Characteristic peaks corresponding to Si-O-Si stretching and amine-related bonds were examined. BET analysis was performed to determine the specific surface area, pore volume, and pore size distribution of the samples. These analyses were conducted under standard conditions to evaluate the effect of functionalization on the structural and adsorption properties of MCM-41.

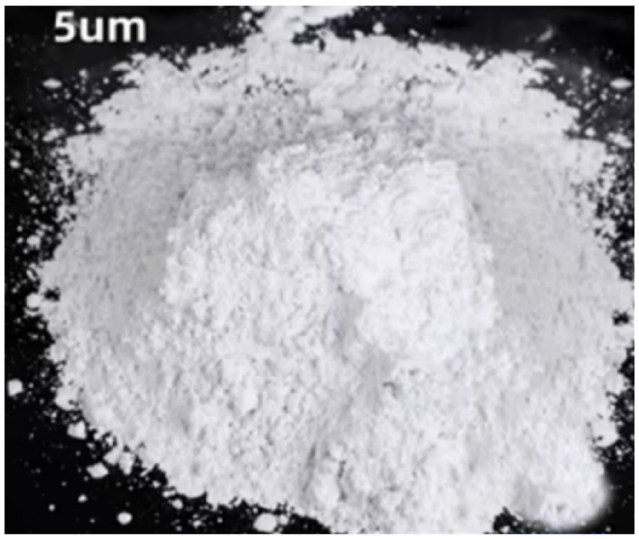

Figure 2. MCM-41 white powder after drying

2.3. Experimental Procedure. CO_2 adsorption experiments were conducted using a high-precision microbalance system to monitor changes in sample mass during gas exposure. Approximately 5-6 mg of each sample was placed on the sample holder inside a sealed chamber connected to a controlled CO_2 supply. Prior to gas introduction, the baseline mass was recorded under ambient conditions to ensure stability. CO_2 was introduced at a constant flow rate, and the mass variation was continuously monitored until equilibrium was reached. Following the adsorption stage, the gas flow was stopped to observe desorption behavior and evaluate the reversibility of the adsorption process. All experiments were conducted under identical environmental and operational conditions to ensure accurate comparison between unmodified and functionalized samples. A summary of the experimental matrix and specimen identifiers is provided in Table 2.

CO_2 adsorption experiments were conducted at a constant flow rate of 50 mL/min under controlled laboratory conditions.

2.3.1. Test setup. The experimental setup consisted of a high-precision analytical microbalance used to monitor mass changes during CO_2 adsorption. Approximately 5-6 mg of each sample was placed in a sealed chamber connected to a controlled CO_2 supply. The initial mass was recorded prior to gas exposure, followed by monitoring until equilibrium was reached. The gas flow was then stopped to observe desorption behavior and evaluate adsorption reversibility. All tests were conducted under identical conditions to ensure reliable comparison.

2.3.2. Instrumentation. The instrumentation used in this study included a high-resolution analytical microbalance (Mettler Toledo) capable of detecting mass variations at the milligram level, enabling real-time recording of adsorption and desorption profiles. Structural and chemical analyses were carried out using an FTIR spectrometer (Thermo Scientific) equipped with an ATR detector to confirm the presence of silica framework peaks and identify amine-related bands following surface modification. BET surface area and pore structure parameters—including specific surface area, pore volume, and average pore diameter—were determined from nitrogen adsorption-desorption isotherms at 77 K using a surface area analyzer (Micromeritics). Instrument calibration was performed according to manufacturer specifications to ensure measurement accuracy and repeatability. The instrumentation used in this study is shown in Figures 3–6.

3. RESULTS AND DISCUSSION

This section presents the key findings obtained from the characterization and CO_2 adsorption testing of MCM-41 before and after surface functionalization. The results demonstrate the impact of incorporating amine-based functional groups on structural properties, chemical composition, and adsorption performance. A combination of FTIR, BET surface analysis, and real-time CO_2 uptake measurements was used to evaluate changes

Table 1. *Summary of material characteristics*

Sample	Condition	Form	Color	Purpose
MCM-41	Unmodified	Powder	White	CO_2 adsorption baseline
Hydrosil 2775	Amine modifier	Liquid	Light yellow	Introduces additional active sites
Hydrosil 2999	Ammonium modifier	Liquid	Yellow-brown	Enhances CO_2 affinity
MCM-41-S1	Functionalized	Powder	Off-white	Evaluates S1 modification effect
MCM-41-S2	Functionalized	Powder	Pale yellow	Evaluates S2 modification effect

Table 2. *CO_2 adsorption-desorption test matrix*

No.	ID	Sample	Functionalization Type	Mass (mg)	Adsorption/Desorption Time (min)	Temperature
1	A-1	MCM-41 (unmodified)	None	5.70	15/10	25 °C
2	A-2	MCM-41 (unmodified)	None	5.72	15/10	25 °C
3	B-1	MCM-41-S1	S1	5.83	15/10	25 °C
4	B-2	MCM-41-S2	S2	5.80	15/10	25 °C
5	C-1	MCM-41-Mix	S1 + S2	5.85	15/10	25 °C
6	Blank	System baseline check	—	0.00	—	25 °C
7	Rep-1	MCM-41 (unmodified)	None	5.71	15/10	25 °C
8	Stab-A	MCM-41 (unmodified)	None	5.70	Continuous	25 °C
9	Stab-B	MCM-41 (functionalized)	S1 + S2	5.83	Continuous	25 °C
10	Calib	Reference mass (standard)	—	5.00	—	25 °C
11	Final	MCM-41-Mix	S1 + S2	5.85	15 cycles	25 °C

in framework stability, organic loading, textural properties, and adsorption behavior.

3.1. FTIR Characterization. FTIR characterization was conducted to confirm the structural integrity of the mesoporous silica framework before and after functionalization. Figure 7 shows the FTIR spectrum of unmodified MCM-41, where peaks at ~1100 cm^{-1} , ~800 cm^{-1}, and ~500 cm^{-1} correspond to vibrations of the Si-O-Si network, respectively. The absence of absorption bands in the 2800-3000 cm^{-1} region confirms the effective removal of the surfactant template[7]. Following functionalization, new bands at 2850-2950 cm^{-1} and ~1550-1650 cm^{-1} are attributed to C-H stretching and N-H bending vibrations, respectively, confirming successful grafting of amine groups onto the silica surface[8].

Following the FTIR analysis of the unmodified MCM-41 sample, the corresponding spectrum after functionalization is presented in Figure 8.

3.2. BET Surface Area and Pore Structure. BET analysis was conducted to evaluate the surface area and pore structure

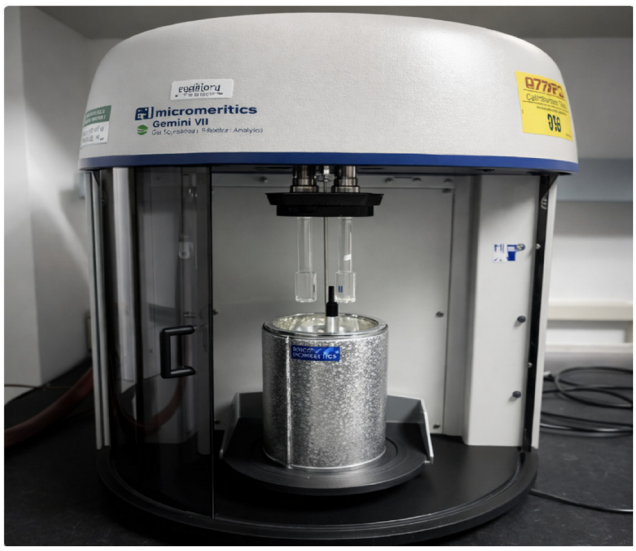

Figure 3. BET surface area analyzer used for nitrogen adsorption-desorption measurements

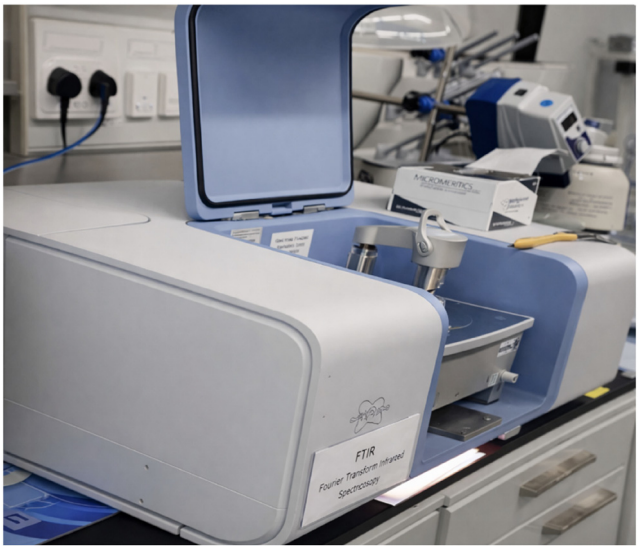

Figure 4. FTIR spectrometer used for functional group analysis

Figure 5. Vacuum filtration setup used for powder separation and collection

characteristics of the synthesized materials. Unmodified MCM-41 exhibited a specific surface area of 334.42 m^2/g and an average pore diameter of 6.53 nm, confirming the formation of a well-defined mesoporous structure. These values fall within the typical range reported for MCM-41 materials in previous studies[9]. As shown in Figure 9, the nitrogen adsorption–desorption isotherm measured at 77 K extends over the complete relative pressure range (P/P0 = 0–1.0). The isotherm exhibits characteristic Type IV behavior with a significant hysteresis loop at high relative pressure, consistent with mesoporous materials. The sharp increase in adsorption at high P/P0 values is attributed to capillary condensation within the mesopores, further confirming the ordered mesoporous structure of MCM-41. Following surface functionalization, a slight decrease in surface area and pore volume was observed due to partial pore occupation by grafted amine groups, consistent with previous studies on amine-functionalized mesoporous silica systems[10]. These results confirm that chemical

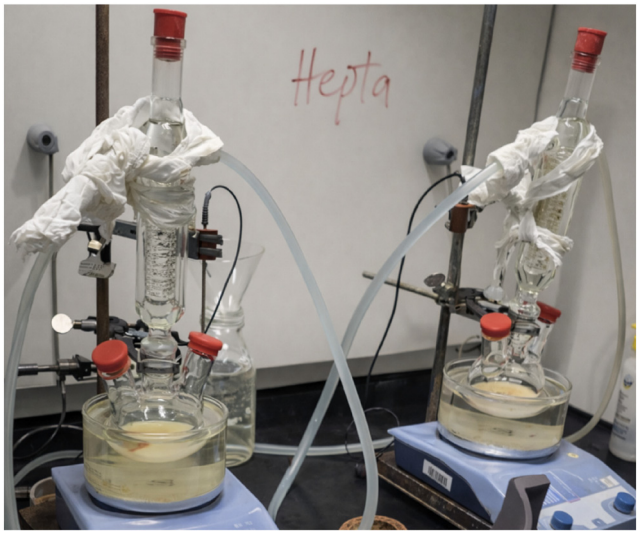

Figure 6. Experimental setup for MCM-41 synthesis under controlled stirring and reflux conditions

modification primarily affected internal surface sites while preserving the overall pore architecture, which is essential for gas adsorption applications.

3.3. CO_2 Adsorption Performance. The CO_2 adsorption–desorption performance of functionalized MCM-41 was evaluated through dynamic mass monitoring under controlled ambient conditions. As shown in Figure 10 , the sample mass remained stable at approximately 5.70 mg prior to CO_2 exposure, confirming baseline stability and measurement accuracy. Upon introduction of CO_2, a rapid increase in mass was observed, indicating fast diffusion of gas molecules into the mesoporous channels and immediate interaction with available adsorption sites. The mass gradually increased until equilibrium was reached at approximately 5.83 mg, corresponding to a net uptake of 0.13 mg (approximately 22.8 mg/g). This enhanced adsorption capacity is attributed to the presence of amine functional groups introduced during surface modification, which provide active sites for interaction with CO_2 molecules[11]. The adsorption process involves both physisorption and chemisorption mechanisms. The presence of amine groups enables chemical interaction with CO_2 through reversible carbamate formation, while physical adsorption also contributes within the mesoporous structure. This combined mechanism explains the enhanced adsorption performance and the observed reversible behavior. During the desorption phase, initiated by terminating the CO_2 flow, a marked decrease in mass was recorded in Figure 10, indicating that the adsorption process is largely reversible. This behavior suggests that the material can be regenerated under mild conditions, which is advantageous for cyclic carbon capture applications. Similar adsorption-desorption behavior has been reported in previous studies[12].

3.4. Influence of Key Experimental Parameters. This subsection discusses how the selected experimental parameters influenced the CO_2 adsorption performance of the synthesized MCM-41 samples. The main factors considered include material type, surface functionalization treatment, mass variation response, and gas exposure duration. These parameters were selected to assess whether surface functionalization enhances CO_2 capture capacity and whether structural modification influences adsorption kinetics and reversibility.

3.4.1. Effect of material type and surface functionalization. Comparison between unmodified MCM-41 and functionalized samples demonstrated a clear improvement in CO_2 adsorption performance. Unmodified MCM-41 exhibited limited uptake owing to weak interactions between CO_2 molecules and surface silanol groups. Following functionalization with Hydrosil 2775 (S1) and Hydrosil 2999 (S2), a marked increase in adsorption capacity was observed, indicating enhanced affinity toward CO_2. The mass increased from 5.70 to 5.83 mg, corresponding to a net uptake of 0.13 mg. This improvement is attributed to the amine functional groups introduced by surface modification, which provide active binding sites and promote stronger interactions with CO_2 molecules. These results confirm that surface functionalization substantially enhances the adsorption performance of MCM-41 relative to the unmodified material.

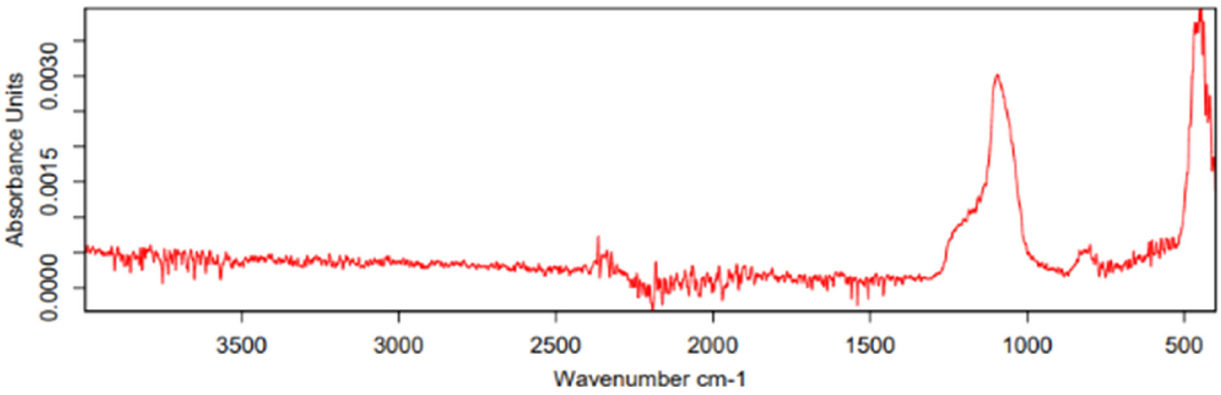

Figure 7. FTIR spectrum of MCM-41 before functionalization, showing characteristic Si–O–Si bands and absence of organic functional groups

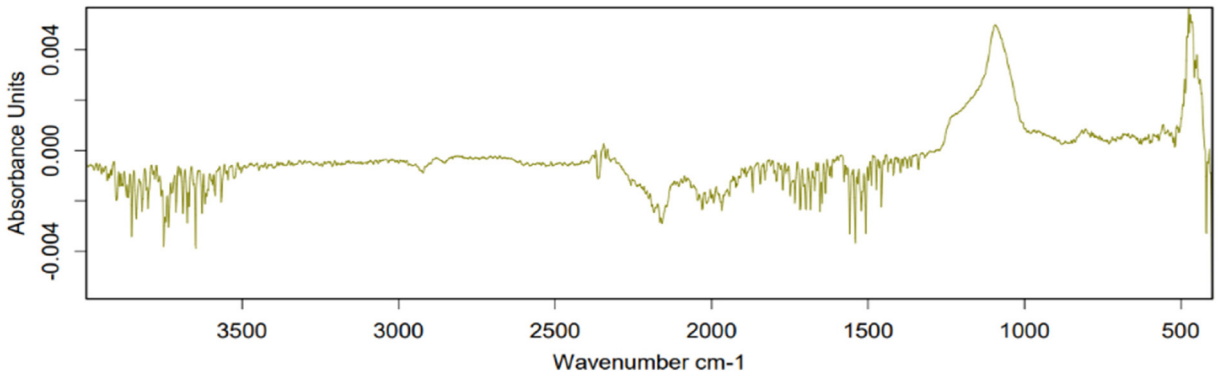

Figure 8. FTIR spectrum after amine functionalization of MCM-41, showing H–N bending and C–H stretching vibrations associated with amine groups. The retention of characteristic Si–O–Si bands confirms that the MCM-41 structural framework was preserved following surface modification

3.4.2. Influence of exposure time and adsorption–desorption behavior. Adsorption time and sample interaction duration play a significant role in the CO_2 uptake process. Both materials exhibited a rapid initial increase in mass, followed by stabilization as equilibrium was reached. After termination of the gas flow, a decrease in mass was observed, indicating that the adsorption process is largely reversible. The adsorption behavior involves both physisorption and chemisorption mechanisms. While physical adsorption occurs within the mesoporous structure, the presence of amine functional groups

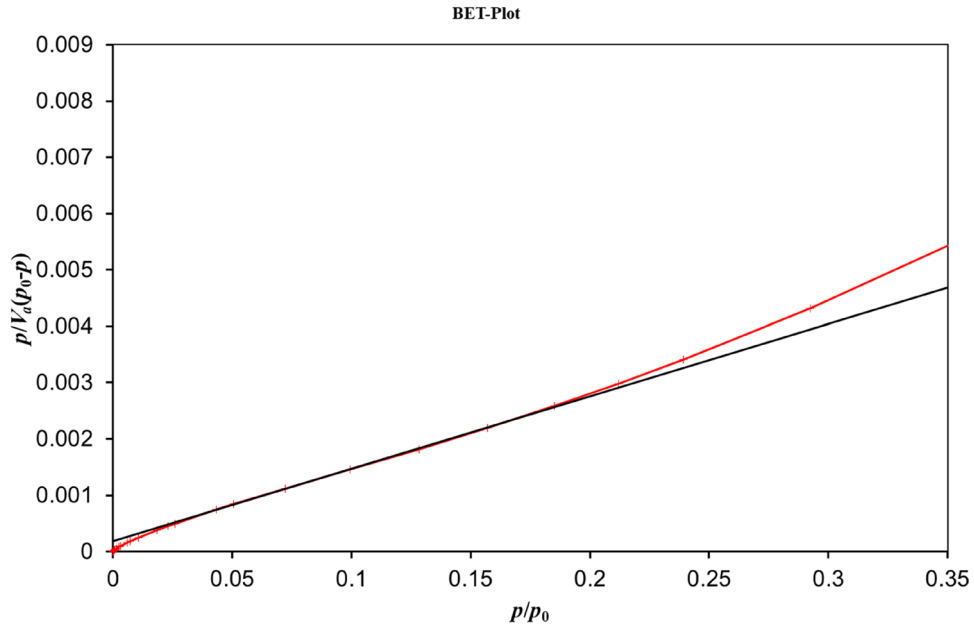

Figure 9. Nitrogen adsorption–desorption isotherm of unmodified MCM-41 at 77 K over the complete relative pressure range (P/P0 = 0–1.0), exhibiting characteristic Type IV behavior with a hysteresis loop indicative of mesoporous materials

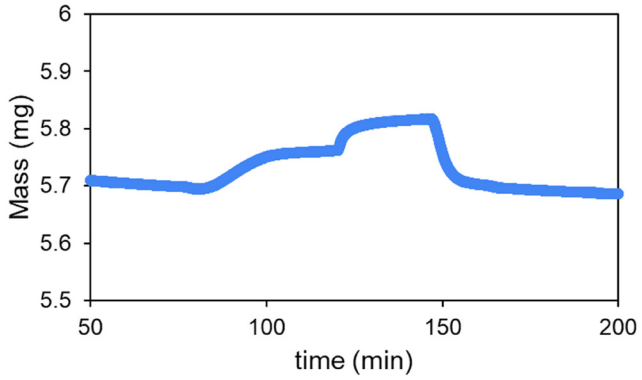

Figure 10. CO_2 adsorption-desorption profile of functionalized MCM-41 showing mass variationas a function of time. The adsorption stage demonstrates rapid CO_2 uptake followed by partial mass recovery during desorption, indicating predominantly reversible adsorption behavior

enables chemical interaction with CO_2 through reversible carbamate formation. The functionalized sample exhibited a slower desorption rate compared to unmodified MCM-41, reflecting stronger amine-CO_2 interaction forces. This behavior highlights the potential for improved capture performance through optimization of amine loading and functionalization conditions.

3.4.3. Interpretation and practical implications. The results demonstrate that functionalization of MCM-41 enhances CO_2 adsorption performance without compromising pore accessibility. Although the improvement is moderate, it confirms the effectiveness of chemical grafting in increasing the density of active adsorption sites. Further optimization—such as increasing amine concentration or adjusting reaction conditions—could significantly enhance performance for real-world carbon capture applications. Overall, the findings highlight the potential of functionalized mesoporous silica as a low-cost, reusable, and efficient solid sorbent for industrial CO_2 separation.

3.4.4. Comparison with previous studies and limitations. The obtained results are consistent with previous studies reporting enhanced CO_2 adsorption performance in amine-functionalized MCM-41 systems due to increased surface reactivity and the presence of active amine sites. The observed improvement in adsorption capacity confirms the effectiveness of surface functionalization in promoting stronger interactions between CO_2 molecules and the adsorbent surface. However, some studies have reported higher adsorption capacities under optimized synthesis and functionalization conditions, indicating that further improvement is achievable. Variations in synthesis parameters, amine loading, and experimental conditions may significantly influence adsorption performance. The present study is subject to certain limitations. The absence of advanced characterization techniques such as scanning electron microscopy (SEM) and elemental analysis (CHN) limits detailed understanding of surface morphology and nitrogen content. Additionally, optimization of functionalization parameters was not extensively explored. Future work should address these limitations and focus on improving material performance through enhanced characterization and process optimization.

4. CONCLUSIONS

In this study, the synthesis and amine functionalization of MCM-41 were successfully carried out to evaluate its potential for CO_2 capture applications. Structural characterization using FTIR spectroscopy confirmed the effective grafting of amine groups from Hydrosil 2775 and Hydrosil 2999 onto the silica framework, while BET analysis verified a high specific surface area of 334.42 m^2/g, confirming the retention of the mesoporous structure following modification. Dynamic CO_2 adsorption tests demonstrated a marked mass response, with the sample mass increasing from 5.70 mg to 5.83 mg, corresponding to a net uptake of approximately 22.8 mg/g. These results indicate that amine-functionalized MCM-41 is a promising and readily scalable adsorbent for CO_2 capture under ambient conditions. From a practical perspective, the developed material offers a cost-effective and efficient approach for industrial CO_2 separation processes. The combination of preserved pore structure and enhanced surface reactivity makes it suitable for repeated adsorption–desorption cycles. Theoretically, this study highlights the importance of surface functionalization in improving gas--solid interactions and optimizing adsorption performance in mesoporous materials. Future work should focus on multicycle stability testing and regeneration energy assessment to further evaluate the feasibility of these materials for large-scale industrial flue gas treatment.

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