

Optimizing Lithium-Ion-Exchanged Zeolite 13X for Enhanced CO₂ Adsorption: A Systematic Analysis

Intivenuka Mohana Rao ^{1, 2, ✉}, Rekha Krishnan G ³, Deepthi L Sivadas ³, K Prabhakaran ²

1. Aerospace Ordnance Entity, Vikram Sarabhai Space Centre, Thiruvananthapuram, IND

2. Department of Chemistry, Indian Institute of Space Science and Technology, Thiruvananthapuram, IND

3. ASD/PCM, Vikram Sarabhai Space Centre, Thiruvananthapuram, IND

Received: August 22, 2025 | Review began: September 10, 2025 | Review ended: July 04, 2026 | Published: August 05, 2026

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Abstract

The lithium modification of zeolite 13X is systematically performed with lithium ion solutions of various concentrations with a view to using it as an adsorbent for carbon dioxide (CO₂) to maintain a safe atmosphere in the crew cabin of human spaceflight missions. The effect of lithium ion solution concentration on the elemental composition, crystal structure, morphology and textural properties of the lithium modified zeolite is characterized by Inductively Coupled Plasma-Atomic Emission Spectrometry, X-Ray Diffraction, Scanning Electron Microscope, and N₂ adsorption-desorption analysis, respectively. Treatment with lithium ion solution does not alter the Si/Al ratio, crystal structure, and morphology of the zeolite; however, it results in partial substitution of Na with Li leading to an improvement in surface area from 557 to 640 m²/g, pore volume from 0.298 to 0.350 cm³/g, and an increase in basic character. This eventually resulted in a considerable improvement in CO₂ adsorption capacity from 4.7 to 5.9 mmol/g at 298 K. The maximum surface area, pore volume, and CO₂ adsorption capacity are observed at a minimum Li ion solution concentration of 0.1 M in a single treatment step. The lithium modified zeolite 13X retains majority of the CO₂ adsorption capacity over a large number of adsorption-desorption cycle indicating its potential as a CO₂ capture material. The CO₂ adsorption capacity of 5.9 mmol/g obtained at 0.1 M lithium ion solution in a single step treatment is significantly higher than the adsorption capacity of 5.71 mmol/g reported in the literature by three cycles of treatment with 1 M lithium ion solution demonstrating a considerable saving of costly and scarce lithium resource, time, and energy.

Categories: Advanced Materials, Materials Engineering, Nano Materials

Keywords: zeolite 13x, lithium ion, regeneration, ion-exchange modification, co2 adsorption

Introduction

Human activities are releasing massive amounts of carbon dioxide (CO₂) into the atmosphere, leading to a global average temperature increase of over 1°C since the late 19th century [1,2]. The burning of fossil fuels and deforestation is driving global warming and associated climate change [3]. The consequences of this trend are being felt by communities worldwide, from more frequent natural disasters and heat-related illnesses to food and water scarcity. As the world's population continues to grow, our carbon footprint is expanding, exacerbating the global warming crisis [4]. From melting glaciers and polar ice caps to altered ecosystems and disrupted food chains [5], the effects of global warming are being felt across the globe [6]. This necessitates the development of CO₂ absorption technologies both from the source

How to cite this article:

Mohana Rao I, G R, Sivadas D, et al. (August 05, 2026) Optimizing Lithium-Ion-Exchanged Zeolite 13X for Enhanced CO₂ Adsorption: A Systematic Analysis. Cureus J Eng 3 : es44388-025-00083-2. DOI <https://doi.org/10.7759/s44388-025-00083-2> Page 1 of 20

of production and from air. Apart from this, closed environments, such as crew cabins in spacecraft and submarines, require precise control of atmospheric conditions to ensure the health and safety of occupants. One critical aspect of maintaining a safe and healthy environment is the control of CO₂ levels [5]. The permissible CO₂ concentration is 1.01 kPa for short duration and 0.40 kPa for long missions [7,8]. Elevated CO₂ concentrations can lead to respiratory problems, fatigue, and decreased cognitive function, ultimately compromising the crew's ability to perform critical tasks [9]. Effective CO₂ adsorption systems are essential to remove CO₂ from the air and maintain a safe and healthy atmosphere [10].

The major CO₂ capture technologies currently employed to manage CO₂ concentration in spacecraft atmosphere can be divided into two categories: (1) physical adsorption using molecular sieves; and (2) chemical adsorption using metal hydroxides/metal oxides and solid/liquid amines. For short-duration missions, lithium hydroxide (LiOH) is the proven candidate for CO₂ removal, with theoretical CO₂ adsorption efficiency of 92%. LiOH is a hygroscopic alkali metal hydroxide that removes CO₂ from the crew cabin in the presence of water by a phenomenon of surface adsorption followed by chemical reaction, producing lithium carbonate [6]. The exothermic reaction generates water as a byproduct, thereby escalating the temperature and humidity inside the crew cabin. For long-duration missions or deep space human exploration journey, LiOH cannot be employed due to the non-regenerability of the material. Unlike the natural life support system existing on earth which facilitates an intrinsic way to manage O₂-CO₂ concentrations, the long duration manned space journey explicitly entails a highly efficient, regenerable and cost-effective CO₂ mitigation system for providing the essential human comfort inside crew cabin. Molecular sieves/zeolites and solid amines are the two classes of adsorbents generally used for removing CO₂ for long duration missions. Zeolites are exceptional candidates for CO₂ removal due to the unique combination of properties like high affinity toward CO₂, good thermal stability, chemical inertness and non-flammable characteristics [11] and their regenerable ability either by means of temperature and/or partial pressure swings methods.

Zeolites are nanoporous crystalline materials with unique thermal stability, chemical resistance, and shape-selective properties. Their frameworks, composed of [SiO₄]⁴⁻ and [AlO₄]⁵⁻ units [12-18], generate the net negative charge, which is balanced by exchangeable cations [19]. These cations play a crucial role in CO₂ capture, exerting an electrostatic attraction on CO₂ molecules and enhancing adsorption. Zeolites exhibit high CO₂ adsorption capacity, rapid kinetics, thermal stability, and good regeneration properties [20,21], making them promising CO₂ adsorbents. However, moisture adversely affects the CO₂ adsorption performance [22,23].

The adsorption of CO₂ on zeolites is a complex process that involves a dual mechanism, encompassing both physical and chemical adsorption pathways. Physical adsorption occurs through non-covalent interactions between CO₂ molecules and the zeolite surface [24], driven by van der Waals forces and the quadrupole moment of CO₂. This process is characterized by a negative enthalpy change (ΔH), indicating an exothermic reaction. In contrast, chemical adsorption involves a strong electrostatic attraction between the negative quadrupole on oxygen atoms of CO₂ molecules and the zeolite framework. This attractive force significantly enhances CO₂ adsorption. The interplay between these two mechanisms enables zeolites to effectively capture and separate CO₂ from gas mixtures [25], making them promising materials for carbon capture.

Modifications to the zeolites are attempted to improve their CO₂ adsorption performance. Modification methods employed on zeolites can be broadly categorized into two types: physical treatments that alter surface morphology, and chemical functionalization that incorporates new chemical groups or metal ions into the zeolite structure. Surface morphology modification has been explored as a potential strategy for enhancing CO₂ capture. However, this approach has several limitations. The modification of surface morphology may not provide sufficient active sites for CO₂

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adsorption and the increased surface area may not necessarily translate to improved CO₂ capture efficiency. Furthermore, the modification process can be complex and may require additional materials or energy, which can increase costs and reduce the overall sustainability of the process [26-30]. Therefore, alternative strategies that focus on optimizing material properties and adsorption mechanisms may be more effective for achieving efficient CO₂ capture.

Various modification techniques such as amine modification [24,31-34], ion exchange [24,35-37], acid treatment [38,39], composite formation [40-43], and nano-sizing [31,44] are the chemical functionalization methods used to enhance the CO₂ capture capabilities of zeolites. Table 2 provides a comprehensive overview of the various modification methods applied to different types of zeolites. The selection of the most suitable method depends on several factors, including the intended application, required CO₂ adsorption capacity, regeneration energy, economic viability, and environmental impact. Amine modification improved CO₂ adsorption [40,41] rates through chemical reactions between amine groups and CO₂ molecules. However, this method raised environmental concerns due to the use of toxic chemicals and waste generation [24,31]. Acid treatment improved mesopore and total pore volumes, increasing CO₂ adsorption capacity [39]. Nevertheless, excessive acid treatment damaged the zeolite structure and decreased its CO₂ capture performance [38,39]. Composite materials [42-45] exhibited higher costs due to complex synthesis processes and expensive surfactants, despite offering fast CO₂ adsorption rates. However, combining zeolites with MOFs (7.009 mmol/g at 298 K, 2 bar) [40], despite excellent performance, proved to be not cost-effective.

Compared to other modification techniques, ion-exchange modification is a versatile, simple, efficient and cost-effective method for tailoring the properties of zeolites for specific applications [24,35]. This method allows for the introduction of specific cations to the zeolite framework while preserving its structural integrity and thermal stability. This process enables the modification of a wide range of zeolites and its exchange sites, that facilitates the precise design of zeolite properties, such as CO₂ adsorption capacity and selectivity and kinetics. A handful of literatures are available on the ion exchange modification of zeolites. Harlick et al. studied the CO₂ adsorption capacities of different types of zeolite materials [46-48] and demonstrated that zeolite 13X with different silica-to-aluminum ratio has an exceptional CO₂ adsorption capacity of 4.6 mmol g⁻¹, due to its unique characteristics, including the good surface area (557 m² g⁻¹), pore diameter (10Å°), large pore volume and low Si/Al ratio (1.14-1.16) [49]. The preferential adsorption of CO₂ over CH₄ and N₂ on zeolite 13X makes it an ideal candidate for CO₂ capture. Walton et al. investigated the CO₂ adsorption equilibrium isotherms on alkali metal ion-exchanged X and Y zeolites. The results revealed a positive correlation between CO₂ uptake and decreasing cation size for both zeolites. Zeolite X and Y samples exchanged with small Li⁺ cations exhibited the highest CO₂ adsorption capacities, reaching 5.6 and 5.2 mmol/g, respectively, at 298 K and 1 bar. According to the authors, this enhanced CO₂ adsorption capacity can be attributed to the stronger ion-quadrupole interactions between the Li⁺ cations and CO₂ molecules, which are more pronounced than in materials containing larger cations.

Gęsikiewicz-Puchalska et al. [11] investigated CO₂ adsorption on zeolites X exchanged with alkaline (Li, Na, K) and alkaline earth metals (Mg, Ca). Nevertheless, they used a concentrated lithium ion solution and performed multiple treatment cycles, which were costly. The results showed that the highest CO₂ adsorption capacities at 273 K and 0.95 bar pressure were achieved with alkaline metal-exchanged zeolites, following the order Li > Na > K, with capacities of 5.94, 5.55, and 4.54 mmol/g, respectively. The enhanced CO₂ adsorption was attributed to the favorable porous parameters of the ion-exchanged samples. In contrast, exchanging with alkaline earth metals (Mg, Ca) led to a significant decrease in specific surface area and micropore volume, resulting in lower CO₂ adsorption capacities. Temperature-programmed desorption analysis was employed to investigate the adsorption of CO₂ on alkaline-modified X zeolites. The parent X zeolite (in its sodium form) was treated with various sodium and cesium aqueous solutions, utilizing both carbonates and hydroxides as precursors. The treated zeolites were then saturated with CO₂ at 50, 100, and 200°C, followed by multiple

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adsorption-desorption cycles to assess their regenerability. Notably, cesium-treated zeolites exhibited higher carbon dioxide retention capacities compared to their sodium-treated counterparts and the parent material. Furthermore, the retention capacity of Cs-treated zeolites demonstrated a more pronounced positive correlation with temperature, particularly when carbonate was employed as the precursor.

Even though a few studies have been conducted on the ion exchange of zeolite 13X, there is no systematic investigation of the lithium modification of zeolite 13X for improved CO₂ adsorption. They used concentrated lithium ion solution and multiple treatment cycles without any optimization. Lithium, being a costly and scarce resource, requires that maximum CO₂ adsorption capacity be achieved through a single-step treatment using the minimum concentration of lithium ion solution possible. The objectives of this study are (1) to prepare and characterize Li-ion modified zeolite 13X, (2) to optimize the initial Li ion solution concentration for the highest CO₂ adsorption, (3) to study the effect of Li modification on the physical and chemical properties, and (4) to assess the stability and reusability of the Li-modified zeolites 13X for multiple CO₂ adsorption-desorption cycles.

Materials And Methods

Lithium nitrate with a purity >99%, supplied by Merck Life Sciences Pvt. Ltd, India, was employed for the ion exchange of the modified zeolite preparation. Zeolite 13X powder with an average particle size of 2 μm was supplied by Sigma-Aldrich, USA. Hydrofluoric acid (40%) and perchloric acid (70%), supplied by Merck Life Sciences, India, were used for dissolving the zeolite samples for composition analysis. Distilled water was used for the preparation of all sample solutions used in all analyses.

Lithium modification of Zeolite by ion exchange

A series of lithium-modified zeolite 13X materials were prepared by a ion exchange process. In a typical procedure, 10 g of zeolite 13X was added to a round-bottom flask containing 400 mL of lithium nitrate solutions of various concentrations (0.01, 0.02, 0.05, 0.07, 0.1, 0.5, and 1 M). The reaction mixture was heated at 80°C under stirring for 4 hours to facilitate efficient ion exchange. After cooling to room temperature, the resulting slurry was centrifuged at 7000 RPM for 10 minutes to separate the solid product from the liquid supernatant. The obtained solid was washed with distilled water to remove residual impurities and then heated at 60°C in an air oven for 2 hours to remove moisture and promote crystal structure ordering. To enhance stability and crystallinity, the dried product was further calcined in a muffle furnace at 350°C for 4 hours. The treatment cycle was repeated using the optimized lithium ion solution concentration to achieve the maximum CO₂ adsorption capacity of zeolite 13X. The resulting lithium-modified zeolite 13X materials were denoted as 13X/C/Li X, where X represents the lithium ion solution concentration (0.01, 0.02, 0.05, 0.07, 0.1, 0.5, and 1 M), and C represents the cycle number.

Characterization

The prepared materials were thoroughly characterized by a range of advanced characterization techniques. The elemental composition of the zeolite samples was accurately determined using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) (Thermo Scientific iCAP 7000 series). The solution for ICP-AES analysis was prepared as follows. A total of 0.05 g of the modified zeolite powder was placed in a platinum crucible, to which 3 mL of distilled water, 5 mL of hydrofluoric acid, and 5 mL of 70% perchloric acid were added. The mixture was heated at 200°C until the sample was completely dissolved. This solution, after being made up to 100 mL, was used for the ICP-AES analysis. The crystal structure of the modified zeolite material was investigated using X-ray diffraction (XRD) analysis. XRD measurements were performed on a Bruker D8-Discover X-ray diffractometer, equipped with a Cu anode operating at 40 kV and 40 mA. The diffractograms were recorded using a LynxEye solid-state detector, scanning from 10° to 90° with a step size of 0.02° and a counting time of 0.5 s per step. The surface morphology of the samples was examined using a Zeiss Gemini 500 field emission scanning electron microscope (FESEM). To enhance the conductivity of the sample surface, a thin layer of gold/palladium (80:20) was deposited using sputter coating prior to imaging. FESEM imaging was

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performed at an accelerating voltage of 2 kV, with the SE2 detector used to capture high-resolution images of the sample surface. The textural properties of the ion-exchanged samples were characterized by nitrogen adsorption/desorption isotherm measurements at 77 K, utilizing a TriStar II 3020 instrument. To ensure accurate analysis, the materials were subjected to degassing under vacuum at 623 K for 3 hours prior to measurement. The specific surface area and pore volume of the samples were subsequently determined using the Brunauer-Emmett-Teller (BET) method, providing valuable insights into their porous structure. CO₂ adsorption experiments were conducted using a volumetric TriStar II 3020 instrument to evaluate the CO₂ uptake capacity of the samples. Prior to the measurement, the samples were subjected to outgassing at 623 K under vacuum for 3 hours to remove any impurities and moisture. The CO₂ adsorption isotherms were subsequently determined at two temperatures, 273 K and 298 K, up to a final pressure of 1 bar, providing valuable insights into the adsorption behavior and capacity of the materials.

Results And Discussion

Chemical composition of lithium-modified zeolite 13X

To achieve the substitution of sodium cations with lithium ions within the zeolite framework through ion exchange, equilibrium between the solution and the solid must be reached. To assess the efficiency of this exchange, ICP-AES measurements were conducted. The chemical composition of untreated zeolite 13X and of the zeolite 13X treated with Li ion solutions of various concentrations from 0.01 to 1 M is given in Table 1. As evident from Table 1, the lithium content in zeolite 13X increases concomitantly with the concentration of the initial lithium ion solution, while the sodium content decreases, indicating a partial replacement of sodium ions by lithium ions through the ion-exchange process. Notably, the increase in lithium content is incremental up to a concentration of 0.1 M, with lower ion-exchange degrees, likely attributed to the strong hydration properties of Li⁺ [13]. The maximum lithium incorporation achieved is 0.96% at a concentration of 0.1 M. Moreover, the Si/Al ratio of all lithium-modified zeolite samples remains within a narrow range (1.14-1.16), suggesting that the ion-exchange process does not compromise the structural integrity of the zeolite.

Materials	Si/Al ratio	Na, Wt%	Li, Wt %
Zeolite 13X	1.16	14.7	<0.01
13X/C1/Li 0.01	1.14	14.1	0.17
13X/C1/Li 0.02	1.16	12.6	0.25
13X/C1/Li 0.05	1.15	10.4	0.45
13X/C1/Li 0.07	1.16	8.49	0.89
13X/C1/Li 0.1	1.15	8.04	0.96
13X/C1/Li 0.5	1.14	8.02	0.98
13X/C1/Li 1	1.15	8.01	0.98

TABLE 1: The sodium and lithium content of lithium modified zeolite 13X samples

XRD

The XRD patterns of Li-modified zeolite 13X retain all characteristic diffraction peaks of the parent zeolite, indicating that the framework structure is preserved after Li⁺ ion exchange (Figure 1). The absence of new diffraction peaks or significant peak shifts confirms that lithium incorporation does not alter the crystal structure, demonstrating the excellent structural stability of the zeolite and the retention of its face-centered cubic framework.

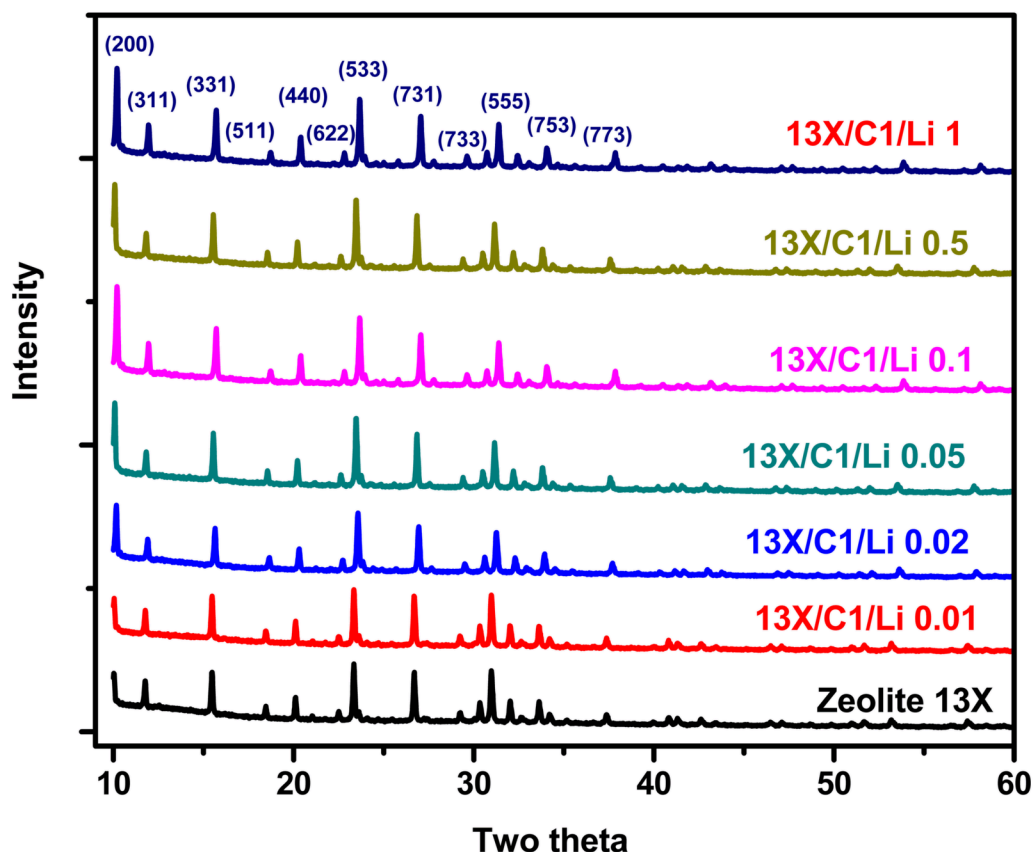


FIGURE 1: X-ray diffraction patterns of lithium-modified zeolite 13X samples

Morphology of Li-modified zeolite 13X

Scanning electron microscopy (SEM) was employed to examine the effect of Li⁺ ion exchange on the morphology of zeolite 13X. As shown in Figure 2, both the zeolite 13X and Li-modified samples retain the characteristic well-defined polyhedral crystal morphology of zeolite 13X, with no evidence of particle fracture, surface erosion, or agglomeration following ion exchange. Furthermore, no noticeable changes in particle size or external surface texture are observed, even with increasing lithium solution concentration. These results indicate that the ion-exchange treatment is sufficiently mild to preserve the crystal morphology and structural integrity of the zeolite particles.

The absence of significant morphological changes suggests that the Li⁺ ions are primarily incorporated through the replacement of exchangeable Na⁺ ions within the internal pore network rather than by modifying the external crystal surface. This observation is consistent with the XRD results, which confirm that the crystalline framework remains intact after ion exchange. The preservation of the original particle morphology is advantageous because it maintains the

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accessibility of the microporous network while improving the internal adsorption characteristics. Consequently, the enhanced CO₂ adsorption performance of the Li-modified zeolite is attributed to changes in the internal cation distribution and pore environment rather than to alterations in particle morphology or crystal structure.

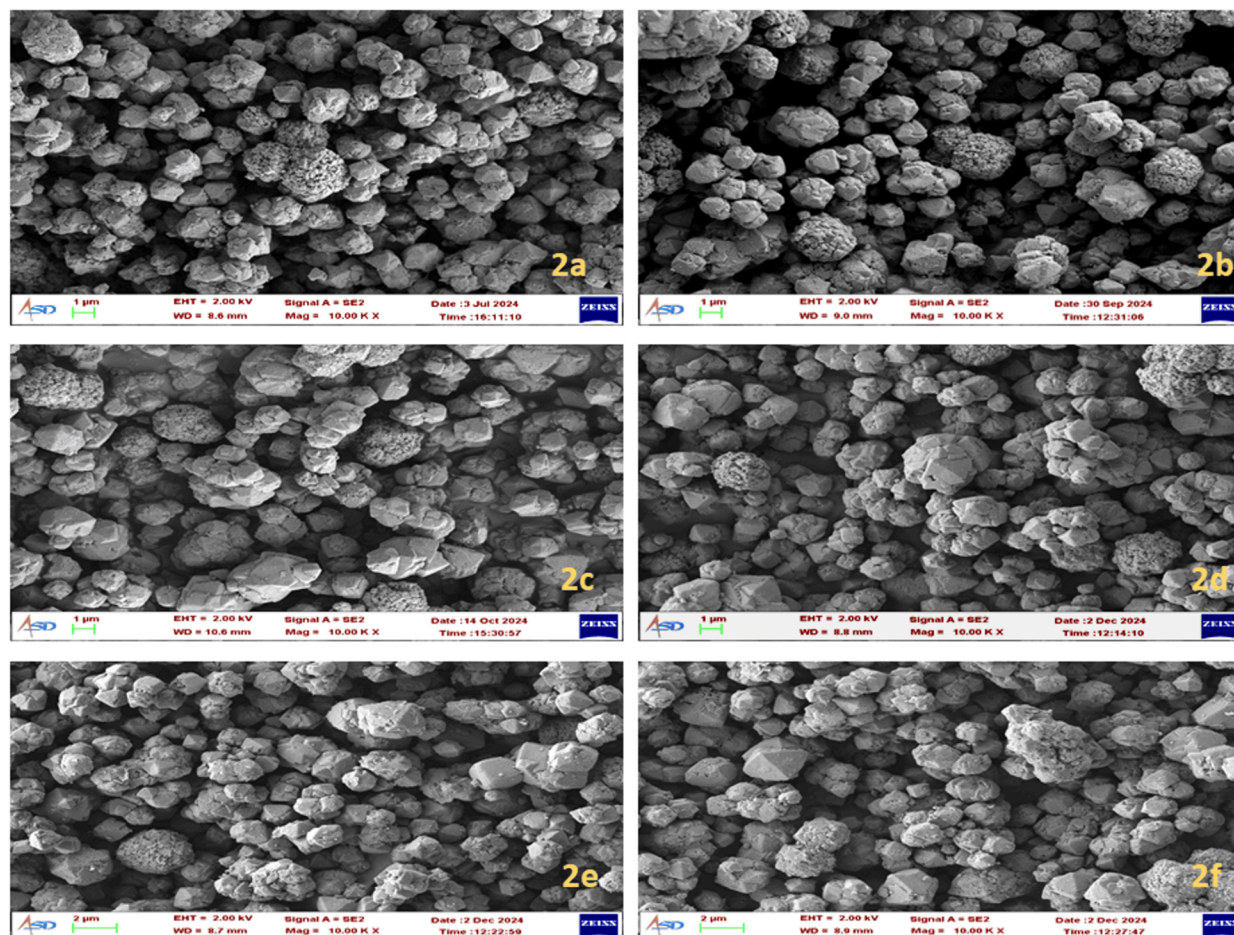


FIGURE 2: SEM images of lithium-modified zeolite 13X

2a-Zeolite 13X, 2b-13X/C1/Li 0.01, 2c-13X/C1/Li 0.05, 2d-13X/C1/Li 0.1, 2e-13X/C1/Li 0.5, 2f-13X/C1/Li 1. SEM, Scanning electron microscopy

Thermal stability of lithium-modified zeolite 13X

To evaluate the thermal stability of both zeolite 13X and modified zeolite 13X, thermo gravimetric analysis (TGA) was performed in an inert atmosphere. The TGA profiles, illustrated in Figure 3, reveal the weight loss of the materials as a function of temperature. The results indicate that both samples exhibit an initial weight loss at lower temperatures up to 200°C, attributed to the desorption of physically adsorbed water. Beyond this temperature, both zeolite 13X and lithium-modified zeolite 13X demonstrate excellent thermal stability, with no significant weight loss observed up to 800°C. This finding suggests that the modification process does not compromise the thermal stability of the zeolite material. Nearly 1.6% additional weight loss is observed in the lithium-modified sample at temperature of 300°C due to more hydration power of lithium ions or the presence of a trace amount of LiOH, which decompose on heating.

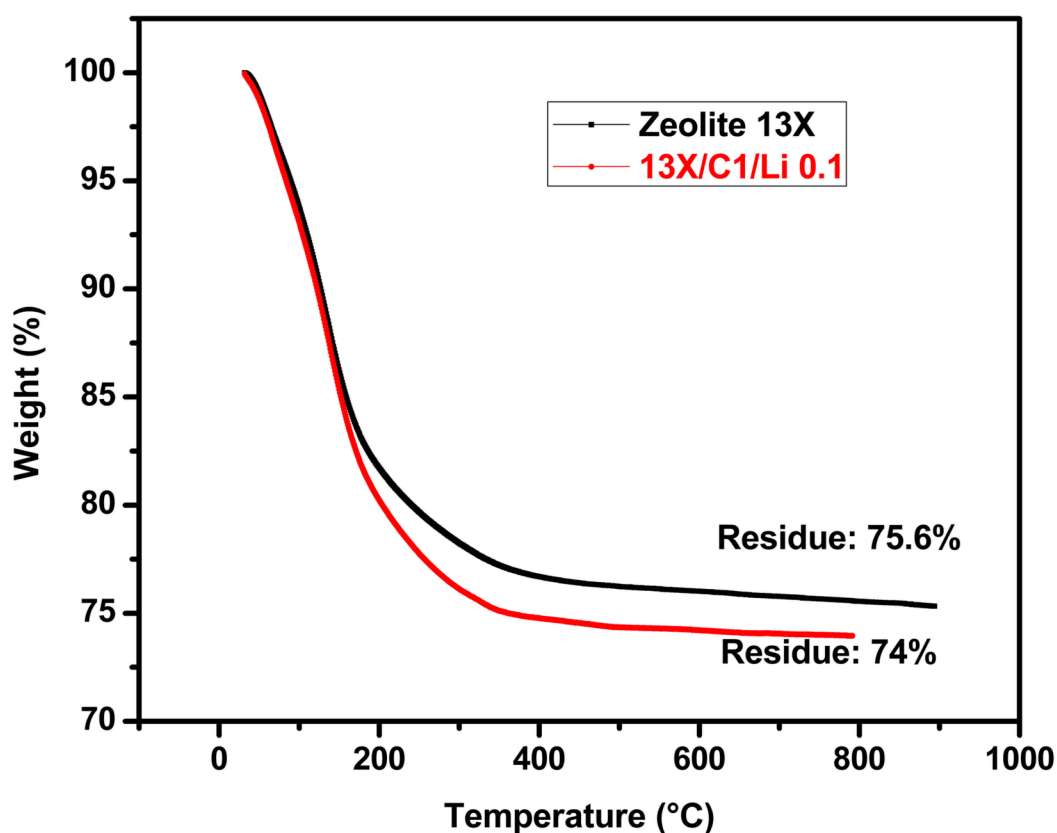


FIGURE 3: TGA of zeolite 13X and 13X/C1/Li 0.1 samples

TGA, thermogravimetric analysis

Textural property

The textural properties of pristine and Li-modified zeolite 13X are summarized in Table 2. The BET surface area increased from 557 m² g⁻¹ for the parent zeolite to a maximum of 640 m² g⁻¹ after ion exchange with 0.1 M Li⁺, followed by a slight decrease at higher Li concentrations. A similar trend was observed for the total pore volume, which increased from 0.298 to 0.350 cm³ g⁻¹. The enhancement in textural properties is attributed to the replacement of larger Na⁺ ions by smaller Li⁺ ions, creating additional free volume within the zeolite framework.

The N₂ adsorption-desorption isotherms (Figure 4) exhibit combined Type Ib/IVa characteristics, indicating the presence of both micropores and a small fraction of mesopores. The density functional theory pore size distribution (Figure 5) shows that Li-exchanged samples possess a dominant microporous structure with pore sizes mainly in the 11–14 Å range and negligible mesoporosity. The increased surface area and pore volume of the 0.1 M Li-modified zeolite provide more accessible adsorption sites and facilitate CO₂ diffusion into the microporous network, thereby enhancing the adsorption capacity.

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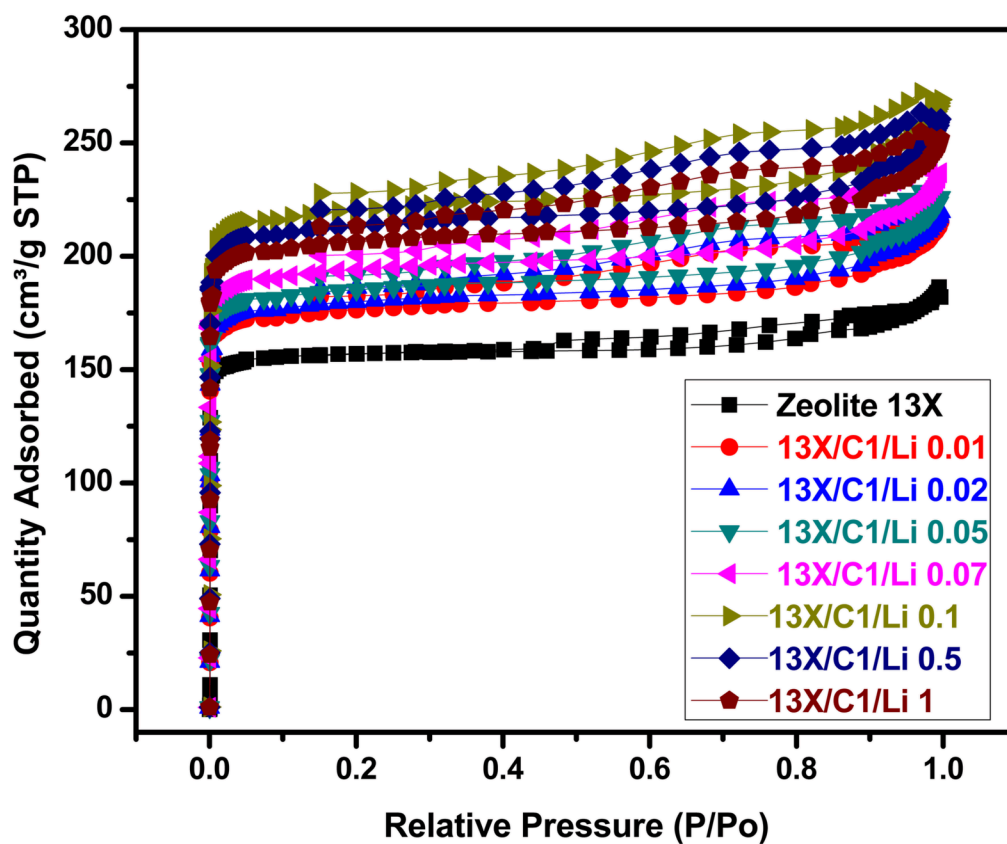


FIGURE 4: Nitrogen adsorption-desorption isotherms of Li-modified zeolite 13X samples

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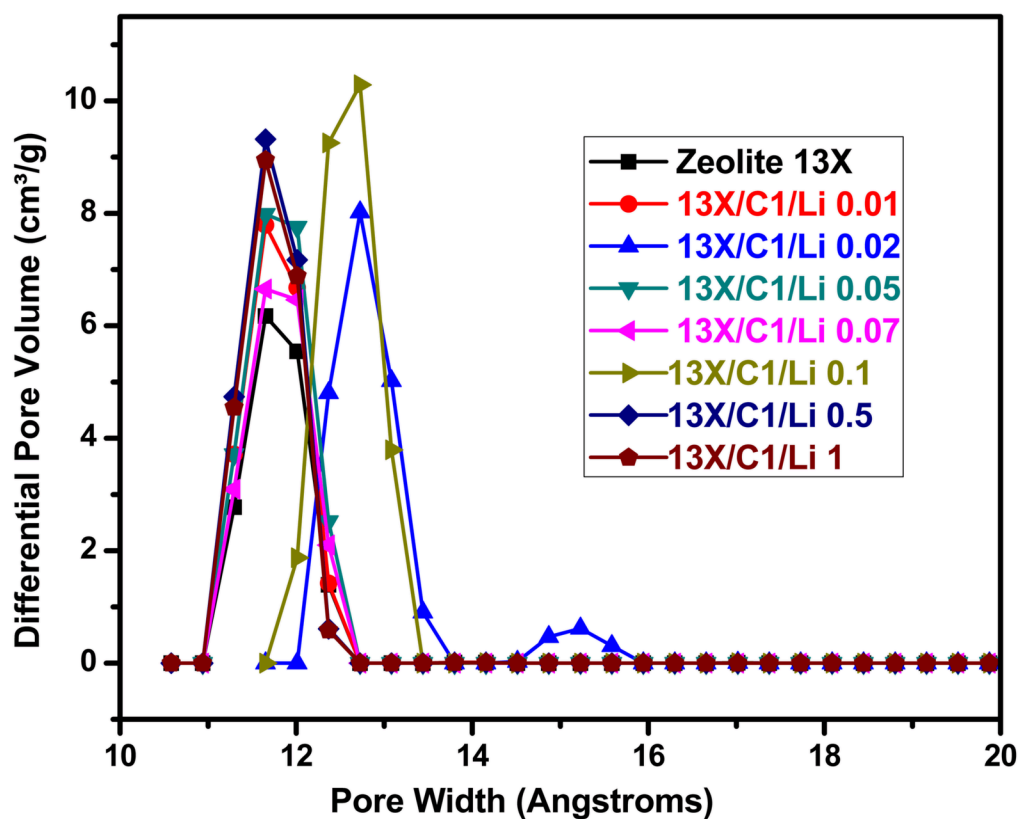


FIGURE 5: Pore size distribution curves of Li-modified zeolite 13X samples

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Mohana Rao I, G R, Sivadas D, et al. (August 05, 2026) Optimizing Lithium-Ion-Exchanged Zeolite 13X for Enhanced CO₂ Adsorption: A Systematic Analysis. Cureus J Eng 3 : es44388-025-00083-2. DOI <https://doi.org/10.7759/s44388-025-00083-2>

Materials	Surface area (m ² g ⁻¹)	Micropore volume (cm ³ gm ⁻¹)	Total pore volume (cm ³ gm ⁻¹)
Zeolite 13X	557	0.196	0.298
13X/C1/Li 0.01	564	0.204	0.301
13X/C1/Li 0.02	586	0.219	0.308
13X/C1/Li 0.05	612	0.224	0.319
13X/C1/Li 0.07	629	0.237	0.331
13X/C1/Li 0.1	640	0.245	0.350
13X/C1/Li 0.5	634	0.244	0.341
13X/C1/Li 1	630	0.243	0.336

TABLE 2: Textural properties of lithium-modified zeolite 13X samples

CO₂ adsorption efficiency evaluation

The adsorption of CO₂ in zeolite involves a complex interplay of physical and chemical mechanisms. Physical adsorption occurs through van der Waals forces, while chemical adsorption involves acid-base reactions [16]. The effectiveness of zeolite modification with ion exchange depends on various factors, including the concentration and type of exchanged cations, reaction time, and temperature [11]. The CO₂ adsorption efficiency of zeolite 13X and lithium-modified zeolite 13X was evaluated at room temperature (298 K) using the BET method, with the results presented in Figure 6 and Table 3. The data reveal that lithium-exchanged zeolite 13X exhibits a significantly higher carbon dioxide adsorption capacity at 1 bar and 298K compared to the virgin zeolite 13X. The maximum CO₂ adsorption achieved is 26 wt% (5.9 mmol/g) for the 0.1 M lithium-modified sample, corresponding to a lithium content of 0.96%. The initial uptake of carbon dioxide increases with increasing lithium concentration, as shown in Figure 6, indicating a strong initial affinity between the adsorbate and adsorbent. The enhanced CO₂ adsorption capacity of lithium-modified zeolite 13X can be attributed to the unique properties of Li⁺ ions, including their small atomic radius, high polarizing power, high basicity, and charge density [13]. The presence of lithium induces a higher electric field within the zeolite cavities, while the framework itself possesses a more basic character compared to the virgin 13X. The small atomic radius of Li⁺ ions enables them to form closer associations with the zeolite framework, enhancing the affinity of the zeolite for CO₂. Consequently, materials containing Li⁺ cations exhibit the highest CO₂ capacities due to stronger ion-quadrupole interactions between CO₂ and Li⁺ compared to larger cations such as Na⁺. Gęsikiewicz-Puchalska et al. [13] achieved a CO₂ adsorption capacity of 5.71 mmol/g at 298K in a zeolite 13X sample by three cycles of treatment with 1 M Li ion solution which otherwise shows a CO₂ adsorption capacity of 5.45 mmol/g. Considering the original CO₂ adsorption capacity of 4.27 mmol/g in our zeolite 13X sample, the adsorption capacity of 5.48 mmol/g obtained by treatment with 1 M lithium ion solution is comparable

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to value reported by Gęsikiewicz-Puchalska et al. [13]. It is interesting to note that our systematic study revealed a high CO₂ adsorption capacity of 5.9 mmol/g at a low lithium ion concentration of 0.1 M in a single step treatment, saving costly and scarce lithium resource as well as time and energy.

The influence of moisture is an important consideration for the practical application of Li-modified zeolites in CO₂ capture, as industrial flue gases typically contain significant amounts of water vapor. Water molecules possess a strong affinity for the polar adsorption sites and exchangeable Li⁺ cations present in the zeolite framework. Consequently, H₂O molecules can compete with CO₂ for the available adsorption sites, leading to a reduction in CO₂ adsorption capacity under humid conditions. In addition, the adsorption of water within the microporous structure may partially block pore channels, thereby limiting the diffusion of CO₂ molecules into the internal adsorption sites.

Despite this competitive adsorption, the predominantly physisorption-based interaction between CO₂ and the Li-modified zeolite facilitates effective regeneration. Water molecules are generally desorbed under moderate heating together with CO₂, allowing the adsorbent to recover its adsorption capacity over repeated adsorption-desorption cycles. Therefore, although humidity may reduce the instantaneous CO₂ uptake, the reversible nature of the adsorption process contributes to the long-term stability and reusability of the Li-modified zeolite.

Material	CO ₂ adsorption capacity at RT	
	mmol/gm	Wt%
Zeolite 13X	4.27	18.8
13X/C1/Li 0.01	5.43	23.9
13X/C1/Li 0.02	5.50	24.2
13X/C1/Li 0.05	5.66	24.9
13X/C1/Li 0.07	5.72	25.2
13X/C1/Li 0.1	5.90	26.0
13X/C1/Li 0.5	5.57	24.5
13X/C1/Li 1	5.48	24.1

TABLE 3: CO₂ adsorption capacities at 298 K of the lithium-modified zeolite 13X samples

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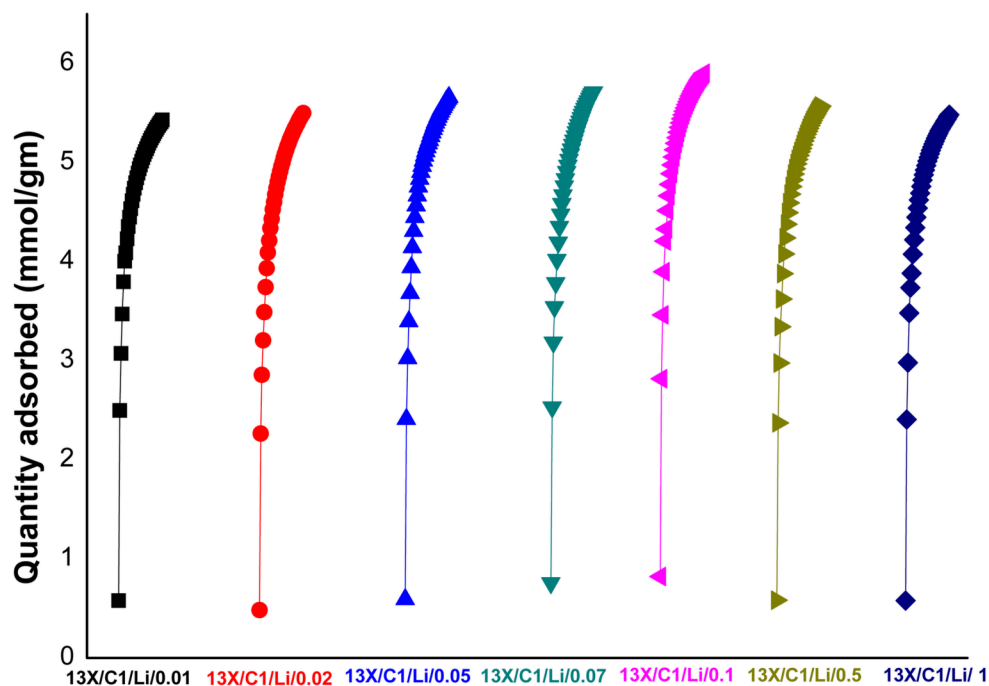


FIGURE 6: CO₂ adsorption isotherms at 298 K of the lithium-modified zeolite 13X samples

To further investigate the optimal lithium loading for enhanced CO₂ adsorption, three additional cycles were conducted using a 0.1 M lithium solution. The results presented in Table 4 reveals that no significant improvements in lithium loading and CO₂ adsorption capacities are achieved beyond the initial cycle. This suggests that the exchange of Na with lithium in zeolite 13X reaches its maximum at 0.1 M lithium ion solution concentration in a single step treatment. Consequently, 13X/C1/Li 0.1 is identified as the optimized material for maximum CO₂ adsorption.

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Material	Si/Al ratio	Li, wt%	Surface area (m ² /g)	Total pore volume (cm ³ /gm)	CO ₂ adsorption capacity at RT	
					mmol/g	wt%
13X/C1/Li 0.1	1.15	0.96	640	0.350	5.9	26.00
13X/C2/Li 0.1	1.14	0.96	634.1	0.351	5.87	25.89
13X/C3/Li 0.1	1.16	0.97	634.9	0.352	5.91	26.00

TABLE 4: Effect of number of treatment cycle on lithium loading and CO₂ adsorption capacity

Effect of temperature on CO₂ adsorption capacity

The CO₂ adsorption isotherms obtained at 273 K and 298 K are presented in Figure 7, with the corresponding adsorption capacity values listed in Table 5. Lithium-modified zeolite 13X exhibited CO₂ adsorption capacities of 6.13 mmol/g at 273 K and 5.90 mmol/g at 298 K. This increase in adsorption at lower temperatures is attributed to the exothermic nature of the adsorption process. As the temperature decreases, the kinetic energy of CO₂ molecules is reduced, enabling more effective interactions with the active sites within the zeolite's porous framework. Furthermore, the strong electrostatic interactions between the polarizable CO₂ molecules and the charged zeolite structure become more significant at lower temperatures, thereby enhancing CO₂ uptake.

Sample	CO ₂ adsorption capacity (mmol/g) at 1 bar pressure	
	273 K	298 K
13X/C1/Li 0.1	6.13	5.90

TABLE 5: CO₂ adsorption capacities of Li-modified zeolite 13X at various temperature

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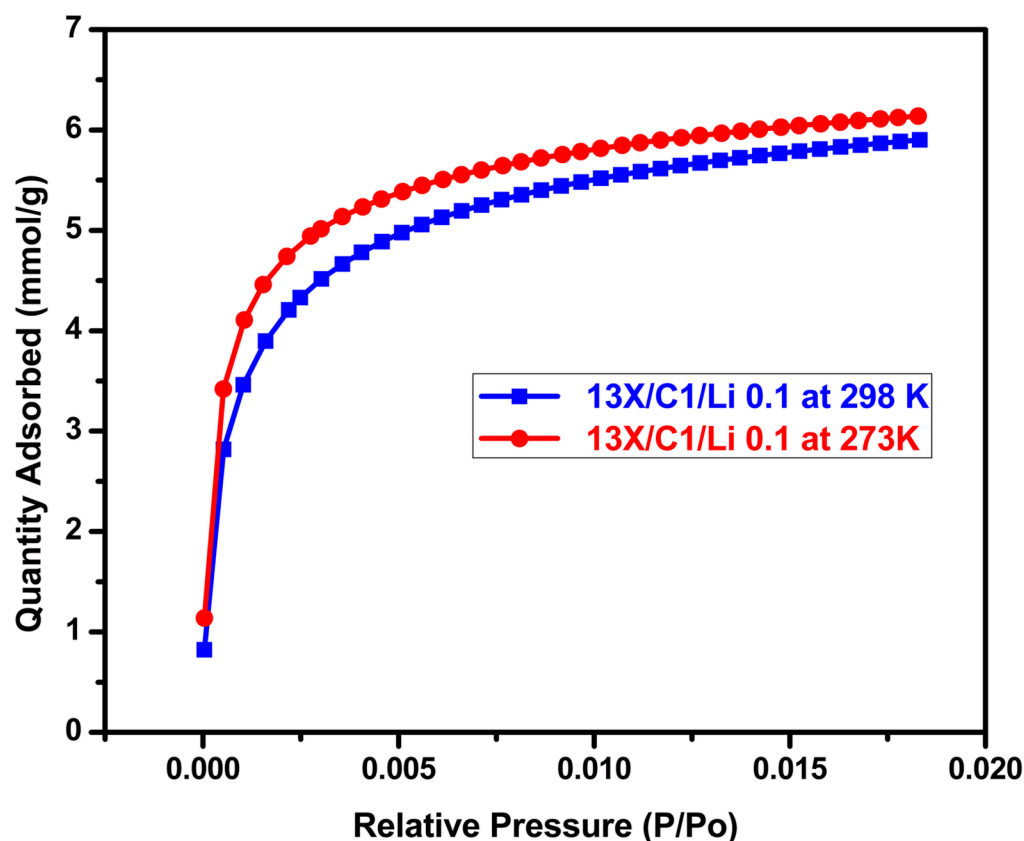


FIGURE 7: CO₂ adsorption isotherms of 13X/C1/Li 0.1 sample at different temperatures

Regeneration

The regenerability depends on the nature of adsorbent-adsorbate interactions. In physisorption involving weak adsorbent-adsorbate interactions, the adsorbent can be completely regenerated by easily desorbing the adsorbed CO₂ either by increasing the temperature or by decreasing the pressure. On the other hand, chemisorption involving strong adsorbent-adsorbate bonding is irreversible. Differential Scanning Calorimetry (DSC) analysis was performed on the CO₂-adsorbed Li-modified zeolite to evaluate the adsorption behavior and estimate the heat of adsorption. Based on the DSC results, the heat of adsorption was calculated to be 38 kJ mol⁻¹. This value falls within the typical range associated with physisorption, indicating that CO₂ adsorption on the Li-modified zeolite is predominantly governed by weak adsorbent-adsorbate interactions rather than the formation of strong chemical bonds. In contrast, chemisorption generally exhibits significantly higher heats of adsorption (>80 kJ mol⁻¹).

To assess the regenerability and stability of the 0.1 M lithium-modified zeolite 13X, a series of five consecutive CO₂ adsorption-desorption cycles were performed. Each adsorption cycle was followed by desorption at 350°C. A slight decrease in adsorption capacity from 5.9 to 5.76 mmol/g is observed from the first to the second adsorption-desorption cycle. However, this adsorption capacity is preserved in the subsequent adsorption-desorption cycles. The results (Table 6, Figure 8) demonstrate excellent consistency and reproducibility with a CO₂ adsorption capacity of 25.3 wt%

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maintained across all five cycles. The small decrease in adsorption capacity (~2.37 wt% decrease) observed in the second cycle is attributed to the presence of slight amount of LiOH in the lithium-modified zeolites. This LiOH irreversibly absorb CO₂ by chemical reaction. This finding indicates that the 0.1 M lithium-modified zeolite 13X can be effectively regenerated and reused without significant loss of adsorption capacity. Based on these results, the 0.1 M concentration is identified as the optimal condition for achieving the highest CO₂ adsorption capacity.

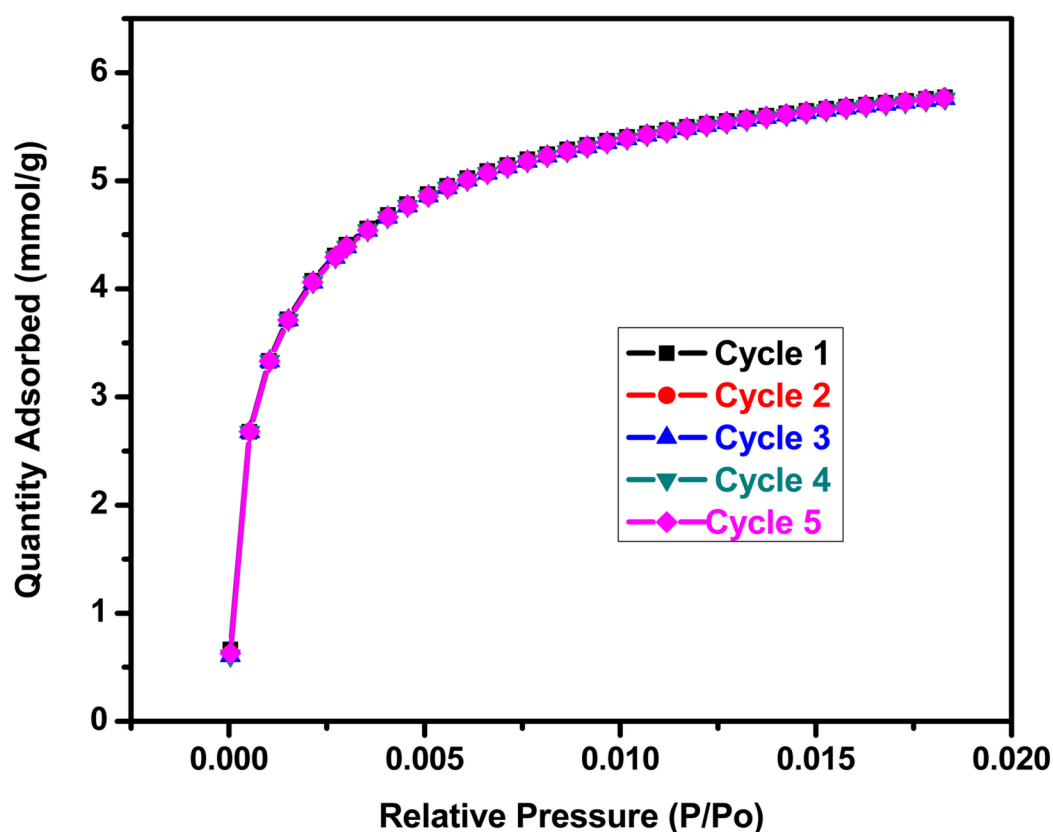


FIGURE 8: CO₂ adsorption isotherms at 298 K of 13X/C1/Li 0.1 showing the cycling stability

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Cycles	CO ₂ adsorption capacity	
	mmol/g	Wt%
1	5.90	26
2	5.76	25.36
3	5.75	25.32
4	5.75	25.32
5	5.76	25.36

TABLE 6: CO₂ adsorption capacity at 298 K of 13X/C1/Li 0.1 sample as function of adsorption-desorption cycle

Conclusions

The treatment of zeolite 13X with lithium ion solutions of various concentrations is performed to achieve maximum CO₂ adsorption capacity at a minimum solution concentration in a single treatment step. The lithium ion solution treatment does not alter the Si/Al ratio, crystal structure, and morphology of the zeolite 13X. The surface area, pore volume, and lithium content of the zeolite 13X increase from 557 m²/g, 0.298 to 0.350 cm³/g, and <0.01 to 0.96 wt%, respectively, by a single-step treatment with 0.1 M lithium ion solution. This increase in surface area and pore volume, coupled with the increase in basicity due to lithium ion exchange, results in a considerable increase in CO₂ adsorption capacity from 4.27 to 5.9 mmol/g at 298 K. The increase in CO₂ adsorption capacity achieved by this single-step treatment with 0.1 M lithium ion solution is much higher than the increase reported (5.45 to 5.71 mmol/g) in the literature by three cycles of treatment with 1 M lithium ion solution. The lithium-modified zeolite 13X exhibits and retains the majority of its CO₂ adsorption capacity over a large number of adsorption-desorption cycles. This optimized treatment results in a remarkable saving of the costly and scarce lithium resource, time, and energy.

The enhanced textural properties, high CO₂ adsorption capacity, and reversible adsorption behavior of the Li-modified zeolite 13X make it a promising adsorbent for a wide range of carbon capture applications. The moderate heat of adsorption (~38 kJ mol⁻¹) indicates that CO₂ is predominantly adsorbed through physisorption, enabling efficient regeneration with relatively low energy consumption and supporting repeated adsorption-desorption cycles.

One important application is the removal of CO₂ from closed environments, such as crew cabins in spacecraft, where maintaining safe CO₂ concentrations is essential for astronaut health. The high microporosity, rapid adsorption kinetics, and regenerability of Li-modified zeolite 13X make it a suitable candidate for use in regenerative air revitalization systems, where compact, lightweight, and reusable adsorbents are required.

In addition, Li-modified zeolite 13X has significant potential for commercial CO₂ capture processes, including post-combustion carbon capture from power plants and industrial flue gases, natural gas purification, biogas upgrading, and pressure swing adsorption or temperature swing adsorption systems. Its high adsorption capacity, structural stability

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after ion exchange, and low regeneration energy requirements contribute to improved process efficiency and reduced operating costs. These characteristics make Li-modified zeolite 13X a promising adsorbent for sustainable carbon capture and gas separation technologies.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

Concept and design: Intivenuka Mohana Rao, Rekha Krishnan G, Deepthi L Sivadas, K Prabhakaran

Acquisition, analysis, or interpretation of data: Intivenuka Mohana Rao, Rekha Krishnan G, K Prabhakaran

Drafting of the manuscript: Intivenuka Mohana Rao, Rekha Krishnan G, Deepthi L Sivadas

Critical review of the manuscript for important intellectual content: Intivenuka Mohana Rao, Rekha Krishnan G, Deepthi L Sivadas, K Prabhakaran

Supervision: Rekha Krishnan G, Deepthi L Sivadas, K Prabhakaran

Disclosures

Human subjects: All authors have confirmed that this study did not involve human participants or tissue. **Animal subjects:** All authors have confirmed that this study did not involve animal subjects or tissue. **Conflicts of interest:** In compliance with the ICMJE uniform disclosure form, all authors declare the following: **Payment/services info:** All authors have declared that no financial support was received from any organization for the submitted work. **Financial relationships:** All authors have declared that they have no financial relationships at present or within the previous three years with any organizations that might have an interest in the submitted work. **Other relationships:** All authors have declared that there are no other relationships or activities that could appear to have influenced the submitted work.

Acknowledgements

This work was presented in the ICAMST-2025 conference held at M.S. Ramaiah University of Applied Sciences, Bengaluru.

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