

Enhanced CO₂ Capture Performance of Mesoporous Ca-A Zeolite Functionalized with Amines for Post-Combustion Applications

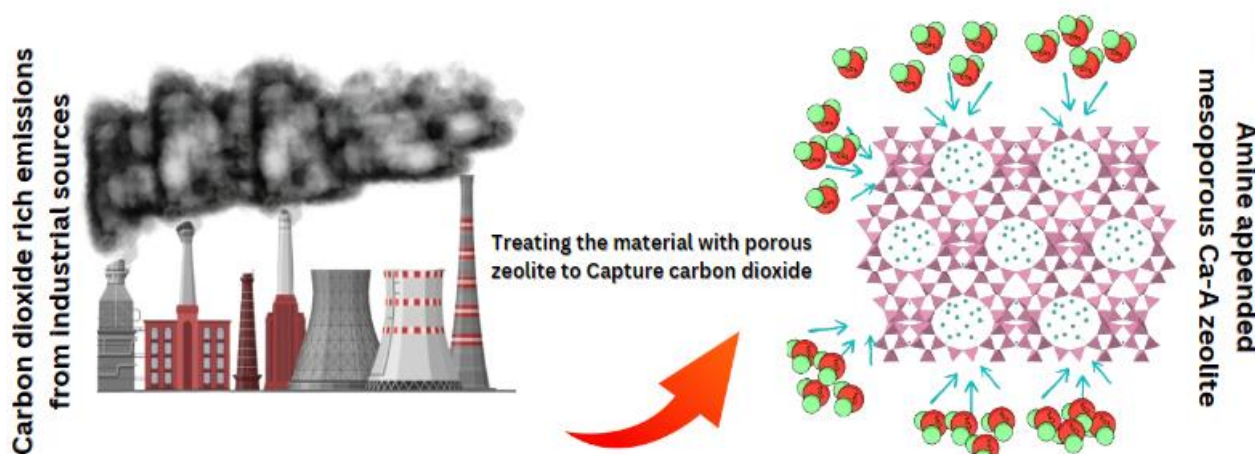
Syed Abdul Moiz Hashmi^{1*}, Muhammad Hammad Rasool², Sheraz Ahmed³

¹Department of Chemical Engineering, Universiti Teknologi Petronas, Bandar Seri Iskander, Malaysia

²Department of Petroleum and Geosciences, Universiti Teknologi Petronas, Bandar Seri Iskander, Malaysia

³Department of Chemical engineering, San Jose State University, San Jose, California

*For Corresponding author: Email address: syed_22009552@utp.edu.my



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Abstract: In this study, we explored the development of materials with enhanced CO₂ adsorption capabilities, focusing on a range of hierarchical LTA zeolites distinguished by their diverse mesopore architectures enhanced with alkylamines. Notably, the most promising CO₂ uptake performance was achieved upon grafting the hierarchical LTA zeolite with a monoamine. By harnessing both the amine groups tethered to the mesoporous surfaces and the active microporous sites within the LTA zeolites, these materials exhibited significantly improved CO₂ adsorption compared to conventional amine appended mesoporous materials. Furthermore, our adsorbent displayed commendable CO₂ uptake capacity, underscoring its potential for practical applications.

Keywords: Zeolites, Mesoporous materials, carbon capture, hierarchical zeolite, amine functionalization

1. Introduction

Carbon capture technologies have emerged as crucial tools in mitigating CO₂ emissions stemming from fossil fuel combustion, a phenomenon intertwined with the escalating threat of climate change attributed to heightened levels of atmospheric carbon dioxide (CO₂) (Hong 2022). Among these

technologies, coal-fired power stations stand out due to their substantial contribution to CO₂ emissions relative to other sources (Krishnan, Nighojkar *et al.* 2023, Ritchie and Roser 2024). Noteworthy is the versatility of carbon capture systems, applicable across various energy-related processes such as natural gas treatment, hydrogen purification via steam reforming, and methane production from biogas (Rubin, Mantripragada *et al.* 2012, Peres, Resende *et al.* 2022, Hosseinfard, Hosseinpour *et al.* 2024). Nonetheless, traditional CO₂ removal methods employing aqueous alkanolamine solutions, known as amine scrubbers, typically entail significant energy inputs for absorbent regeneration post-CO₂ saturation (Meng, Meng *et al.* 2022, Goren, Erdemir *et al.* 2024; Hasib-ur-Rahman *et al.* 2012; Zhang *et al.* 2012). Consequently, numerous studies have aimed to develop solid adsorbents capable of selectively capturing substantial CO₂ volumes, even under low-pressure conditions, while demanding reduced energy for regeneration compared to alkanolamine solutions. This focus underscores the imperative to curtail energy consumption in the realm of CO₂ capture (Rashid and Rafey 2023, Rashid and Rafey 2023, Zhang and Wang 2024).

Certainly, an array of porous solid materials has exhibited intriguing characteristics in the realm of carbon dioxide (CO₂) absorption, particularly for applications in post-combustion carbon capture and conventional coal-fired power plants. Metal-organic frameworks (MOFs) have garnered considerable attention as promising capture materials, owing to their impressive attributes such as substantial surface areas and adaptable chemical functionalities. These features synergistically contribute to high-capacity adsorption and enable selective CO₂ binding (Chen *et al.* 2015; Allangawi, Alzaimoor *et al.* 2023; Yong, Xie *et al.* 2024). Notably, several MOF frameworks featuring coordinatively unsaturated metal sites have demonstrated remarkable CO₂ adsorption capabilities, particularly under low pressures (Li, Yao *et al.* 2022). Moreover, the integration of alkylamine groups into specific metal-organic frameworks post-synthesis has been explored as a strategy to enhance CO₂ binding strength and subsequently augment CO₂ absorption, especially in post-combustion scenarios. However, it's imperative to acknowledge that the production of metal-organic frameworks is costlier compared to similar porous materials like zeolites, porous carbons, and mesoporous silicas. Additionally, the long-term stability of these materials remains subject to debate. Leveraging strong chemical interactions between CO₂ and amine groups grafted onto silica surfaces, alkylamine-modified mesoporous silicas have demonstrated impressive CO₂ adsorption capacities, particularly in post-combustion carbon capture applications (Hack, Maeda *et al.* 2022, Justin, Espín *et al.* 2024). Notably, mesoporous silicas often feature thick pore walls, which contribute to increased volume and mass of the adsorbent but don't necessarily enhance CO₂ uptake efficiency (refer to Figure 1a).

By substituting this excess material with potent CO₂-capturing compounds like zeolites, it's feasible to enhance the total CO₂ adsorption capacity and circumvent this limitation (Borchert, Carrasco *et al.* 2022, Aly, Zafanelli *et al.* 2024). Recent studies have showcased the remarkable CO₂ adsorption capabilities and strong CO₂ selectivity of Ca-A zeolite (refer to Figure 1b), especially at low CO₂ partial pressures (Bae, Hudson *et al.* 2013, Rahmah, Kadja *et al.* 2022, Rahmah, Kadja *et al.* 2022, Sun, Zhang *et al.* 2024). This underscores the potential superiority of a hierarchical Linde type A (LTA) zeolite featuring functionalized alkylamine mesopore domains over traditional aminosilicas (refer to Figure 1c). The dual CO₂ absorption ability of LTA zeolites, encompassing both inherent microporous active sites within the zeolite framework and amino groups grafted onto mesoporous regions, holds promise for enhanced CO₂ capture efficiency. The fabrication of hierarchical zeolites with tailored mesopore domains represents a pivotal advancement in CO₂-capture material development.

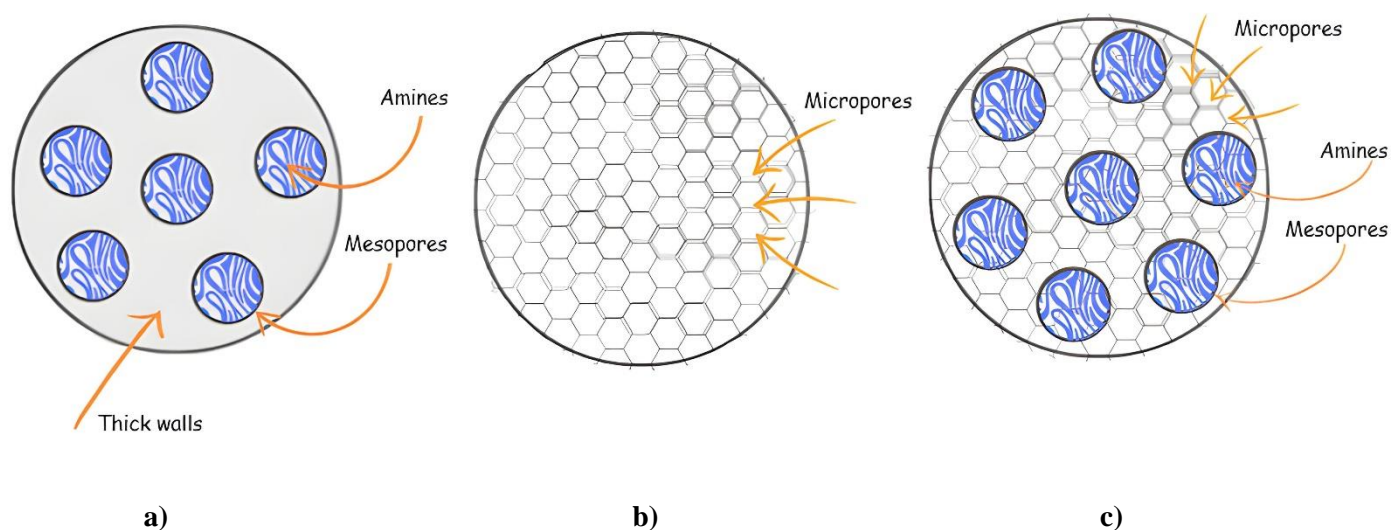


Figure 1: Schematic representation illustrating (a) amine-modified mesoporous silica, (b) LTA zeolite featuring inherent microporous domains, and (c) hierarchical zeolite with alkylamine-functionalized mesoporous domains.

The soft-templating process stands out among various methodologies for synthesizing hierarchical zeolites, offering unparalleled flexibility in tailoring mesoporous architectures (Mumtaz, Irfan *et al.* 2021, Yu and Xiao 2022, Tian, Chen *et al.* 2023, Tian, Chen *et al.* 2023, Cutad, Al-Marri *et al.* 2024, Wei, Feng *et al.* 2024). In this method, surfactant molecules modified with silane groups self-assemble into supramolecular micelle structures, serving as templates for mesopore formation. Collaborating with other mineral sources, the silane moieties actively contribute to zeolite framework crystallization. Consequently, unlike conventional mesoporous materials characterized by amorphous phases, the resulting materials boast mesopore walls composed of zeolite crystals. In this study, we engineered hierarchy within a Linde type A (LTA) zeolite, resulting in the development of mesoporous regions subsequently functionalized with alkylamines. This zeolite was specifically designed to enhance carbon capture applications. The hybrid structure, incorporating both mesoporous and microporous regions, was evaluated for its CO₂ uptake characteristics using equilibrium gas uptake measurements. Additionally, its adsorption and desorption behavior were examined under dynamic flow conditions. The novelty of this work lies in the successful induction of a hierarchy within a LTA zeolite, the integration of amines, and the verification of its CO₂ uptake capabilities under dynamic conditions, demonstrating superior performance compared to conventional amine-functionalized adsorbents.

2. Experimental section

2.1 Synthesis of hierarchical Ca-A zeolite

Sodium metasilicate pentahydrate, sodium hydroxide, sodium aluminate, and [3-(trimethoxysilyl)propyl]octadecyldimethylammonium chloride (TPOAC, 42 wt% in methanol) were sourced from Sigma-Aldrich and used without further purification. TPOAC, a readily available compound, acted as the template for inducing mesopore formation in our study. The synthesis of hierarchical LTA zeolites adhered to established protocols outlined in existing literature (Cho, Cho *et al.* 2009). A precursor gel with a molar ratio of $5\text{Al}_2\text{O}_3/20\text{Na}_2\text{O}/7.5\text{SiO}_2/1200\text{H}_2\text{O}/1.25\text{TPOAC}$ was prepared at room temperature and underwent hydrothermal treatment at 100°C for 4 hours. The quantity of TPOAC was determined based on its performance in CO₂ capture, as reported in prior research (Nguyen, Kim *et al.* 2016).

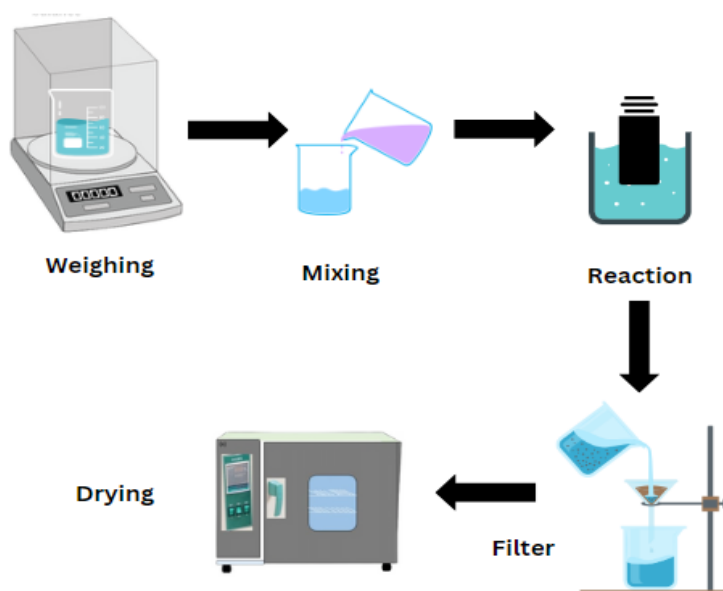


Figure 2: Schematic illustrating the synthesis process of hierarchical LTA zeolites, highlighting the incorporation of mesopore domains.

After the reaction concluded, the resulting products underwent filtration for collection, followed by extensive washing with deionized water to remove any potential impurities. Once purified, the zeolites were dried overnight in a convection oven at 80°C and then subjected to calcination in a furnace at 550°C for 3 hours. This calcination step aimed to effectively eliminate the templating agent TPOAC from the mesoporous framework. The synthesis process is depicted in **Figure 2**.

2.2 Amine grafting of the hierarchical zeolites

The amine functionalization of hierarchical Ca-A zeolites with (3-aminopropyl)-trimethoxysilane (APTMS, 97 wt%, Sigma–Aldrich) or N-[3-(trimethoxysilyl) propyl] ethylenediamine (TMPED, 97 wt%, Sigma–Aldrich) followed a previously reported method (Hicks, Drese *et al.* 2008, Yang, Lin *et al.* 2024). 1 g of hierarchical LTA zeolites was dispersed in 100 mL of toluene. Upon adding 5 mL of either APTMS or TMPED to the mixture, the reaction proceeded at room temperature with vigorous stirring under an argon atmosphere for 1 day. The resulting product was then collected via filtration, washed with toluene, and dried in a vacuum oven at 60°C for 1 day.

2.3 Synthesis of reference mesoporous material (MCM-41)

MCM-41 synthesis employed a two-step approach to generate the pore-expanded variant, as outlined in existing literature (Xu, Cui *et al.* 2024). Initially, a precursor solution for conventional MCM-41 was formulated by combining 2 g of hexadecyltrimethylammonium bromide (HTAB, Sigma–Aldrich) and 4.95 mL of TEOS with a mixture of 170 g of deionized water and 97 mL of ammonium hydroxide aqueous solution (28–30% NH₃). The reaction was conducted at room temperature with vigorous stirring for 2 hours. Subsequently, the resulting product underwent filtration, washing with deionized water, and drying at 60°C for 1 day in a vacuum oven. In the subsequent step aimed at pore expansion, 0.5 g of dimethyldecylamine (DMDA, Sigma-Aldrich) and 30g of deionized water were introduced into a Teflon-lined autoclave containing 0.8 g of MCM-41 synthesized in the initial step. The autoclave was then placed in an oven and maintained at 120°C for 3 days. Following cooling to room temperature, the pore-expanded MCM-41 was collected via filtration, washed with deionized water, and subjected to calcination at 550°C for 5 hours in a furnace.

2.4 Characterizations

Nitrogen physisorption at 77 K was conducted utilizing a volumetric gas sorption analyzer (AS1, Quantachrome). Prior to measurement, both untreated and amine-functionalized substrates underwent degassing at temperatures of 200°C and 130°C, respectively, under dynamic vacuum conditions for 24 hours. The BET surface area and pore volumes were determined utilizing the software integrated into the system. The contribution of the microporous domain to the total pore volume was quantified employing the t-plot method. Pore size distributions were assessed using the Barrett–Joyner–Halenda (BJH) method based on the adsorption branch. Fourier-transform infrared (FT-IR) spectra were acquired using the Spectrum One instrument (PerkinElmer) at room temperature, employing a resolution of 4 cm⁻¹ within the spectral range of 4000 to 500 cm⁻¹. Powder X-ray diffraction (XRD) analysis was carried out using a D8 Advance instrument (Bruker) equipped with CuK α radiation (40 kV, 40 mA). Data collection was conducted under ambient conditions with a step size of 0.018°.

2.4 Gas adsorption isotherms

The equilibrium CO₂ uptake properties of the adsorbents were assessed using a gas sorption analyzer (iSorb HP1-Quantachrome). Data points were collected over a pressure range of 0 to 1 bar at both 25°C and 60°C, with the latter temperature corresponding to the flue gas temperature (60°C) emitted by coal-fired power plants. Temperature control of the samples was carefully maintained throughout the procedure using a circulating fluid. Prior to the adsorption measurements, all samples were activated at 130°C under dynamic vacuum conditions for 12 hours.

3. Results and Discussion

3.1 Synthesis of hierarchical Ca-A zeolite

The X-ray diffraction patterns of the synthesized hierarchical LTA zeolite, prepared via the hydrothermal method, are illustrated in **Figure 3**. These patterns closely resemble those typically observed for standard LTA zeolites (Bae, Liu *et al.* 2011).

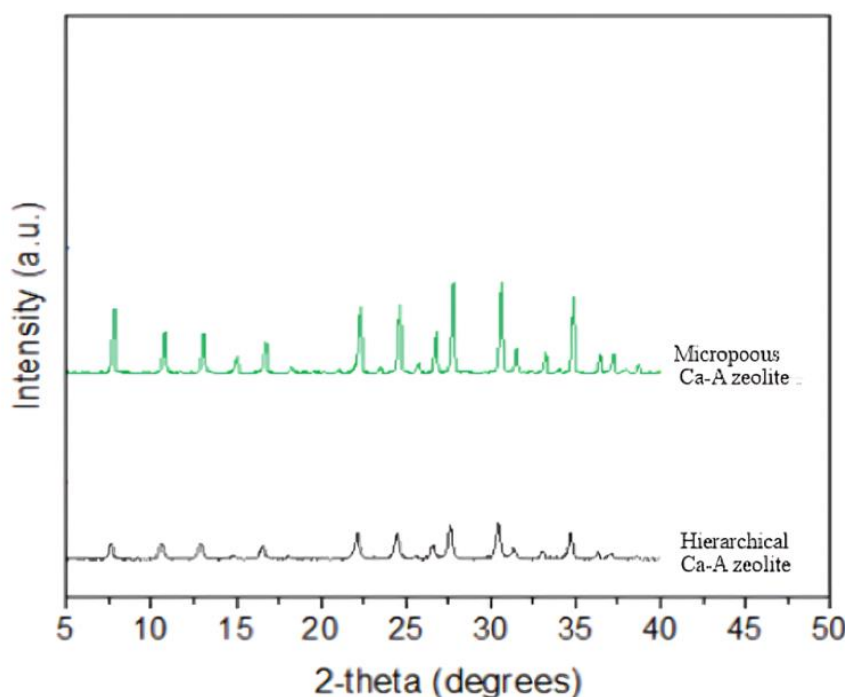


Figure 3: Powder X-Ray diffraction patterns of zeolite Ca-A and hierarchical zeolite H-Ca-A

Following the introduction of mesoporosity (as detailed in the experimental section), there was a gradual decrease in the intensities of the zeolite peaks. This phenomenon is likely attributed to a reduction in the volume fraction of the zeolite LTA domains within the overall sample. Additionally, the broadening of peak widths suggests a gradual decrease in the size of the crystallites comprising the hierarchical LTA zeolite.

To investigate the pore properties of the hierarchical LTA zeolites, N₂ adsorption isotherms were examined at 77 K. The adsorption behavior of Ca-A, synthesized without TPOAC (structuring agent), exhibited typical characteristics of a microporous material, lacking any indication of mesoporosity. In contrast, the presence of hysteresis loops in the adsorption-desorption curves of the zeolites synthesized with TPOAC indicated the presence of mesoporosity (see Figure 4a). As outlined in the table 1, an increase is observed in the total pore volume of the zeolite following the addition of TPOAC in the precursor gel. This increase can be attributed to the formation of mesopores.

Table 1: Properties of LTA hierarchical zeolite calculated via N₂ adsorption at 77 K

Sample	BET surface area (m ² g ⁻¹)	Micropore volume (cm ³ g ⁻¹)	Total pore volume (cm ³ g ⁻¹)	Average mesopore diameter (nm)
Ca-A	613	0.28	0.34	----
H-Ca-A	529	0.11	1.09	11.8

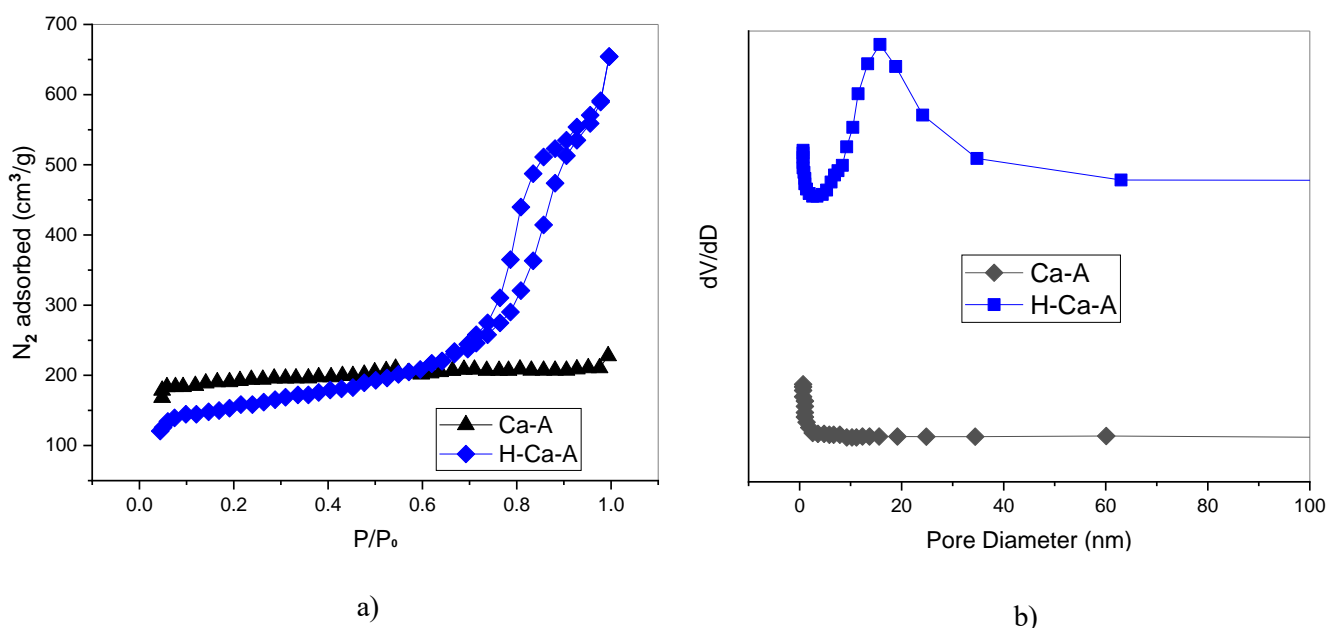


Figure 4: a) Nitrogen adsorption at 77 K and b) Pore size distribution of the LTA zeolites

3.2 Change in morphology of the crystals upon the addition of structuring agent (TPOAC)

The change in morphology of the synthesized zeolites was analyzed using field emission scanning electron microscopy (FESEM). In the precursor gel without TPOAC, characteristic cubic LTA crystals were observed, as shown in Figure 5(a). In contrast, when TPOAC was included in the reagent mixture, smaller crystals aggregated into larger cubic particles, potentially forming secondary pores within the mesopore range, as depicted in Figure 5(b). This indicates a morphological transformation with the

inclusion of TPOAC, leading to a decrease in crystal domain size, which aligns with the broadened peaks observed in the XRD patterns (see figure 3).

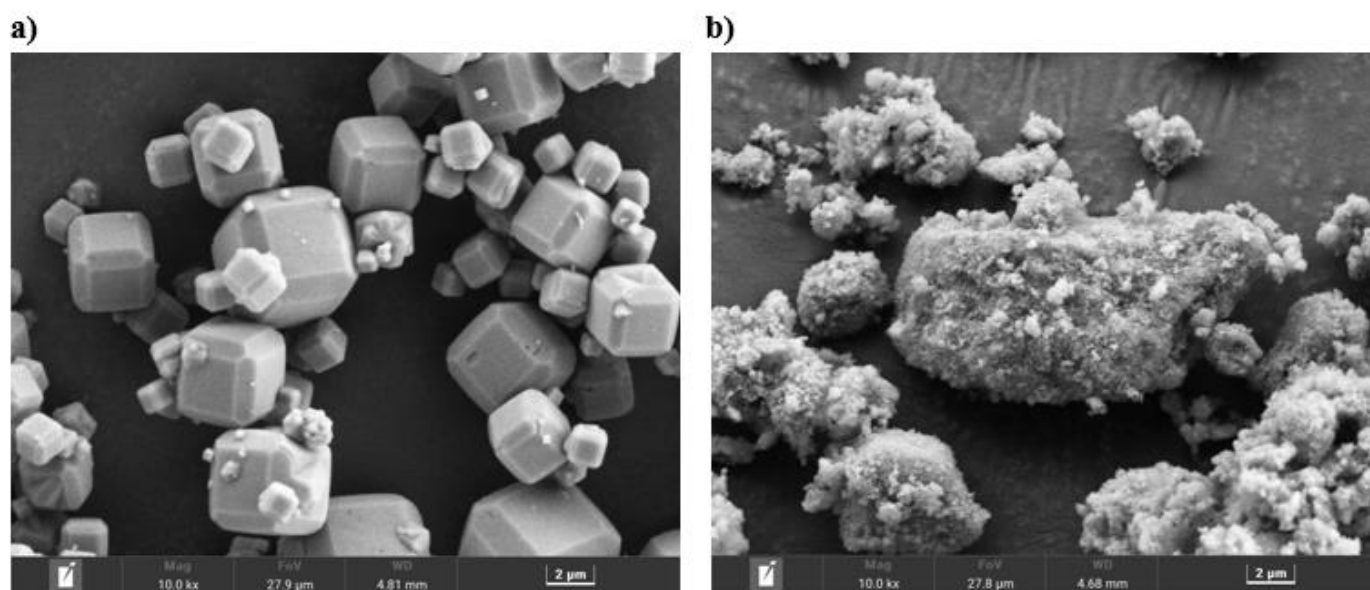


Figure 5: FESEM images of **a)** Ca-A zeolite (without structuring agent i.e, TPOAC), **b)** H-Ca-A zeolite (incorporating TPOAC structuring agent) at a magnification of 10,000.

3.3 Grafting of amines onto the Ca-A Hierarchical zeolite

Using a post-synthetic grafting approach, two different aminosilanes, one featuring a monoamine group, namely (3-aminopropyl)-trimethoxysilane (APTMS), and the other with a diamine group, N-[3-(trimethoxysilyl) propyl] ethylenediamine (TMPED), were grafted onto the hierarchical LTA zeolite samples (Ca-A zeolite). These modified samples were denoted as H-Ca-A-NH₂ and HCa-A-diamine, respectively. To confirm the integration of organic functional groups, the Fourier-transform infrared (FT-IR) spectra of H-Ca-A zeolite before and after the grafting process were compared. The analysis revealed the presence of absorption bands associated with amino groups in both H-Ca-A-NH₂ and H-Ca-A-diamine spectra (refer to Figure 6). Notably, the FTIR spectra of H-Ca-A exhibited a pattern consistent with that observed for Ca-A zeolite (Choi, Cho *et al.* 2006), suggesting the retention of zeolitic characteristics subsequent to the introduction of mesoporosity. Specifically, the intensity of bands observed at 462 cm⁻¹ and 1003 cm⁻¹ corresponded to Si–O–Al bending and Si–O–Al internal tetrahedral asymmetrical stretching vibrations, respectively. Moreover, notable peaks at 2934 cm⁻¹ and 1652 cm⁻¹, signifying N–H stretching and C–H bending vibrations of primary amines, respectively, were detected. Additionally, an additional peak at 1490 cm⁻¹, corresponding to N–H bending vibrations characteristic of secondary amines, was evident in the H-Ca-A-5A-diamine spectrum.

3.4 CO₂ capture properties of the amine grafted H-Ca-A zeolites

In Figure 7, the CO₂ adsorption isotherms of H-Ca-A post rafting with mono and di aminosilanes at 60°C is presented. Comparative analysis reveals that untreated H-Ca-A exhibits lower CO₂ uptake than conventional Ca-A zeolite (Figure 7b), a trend aligned with the observed decrease in micropore volume, as outlined in Table 1.

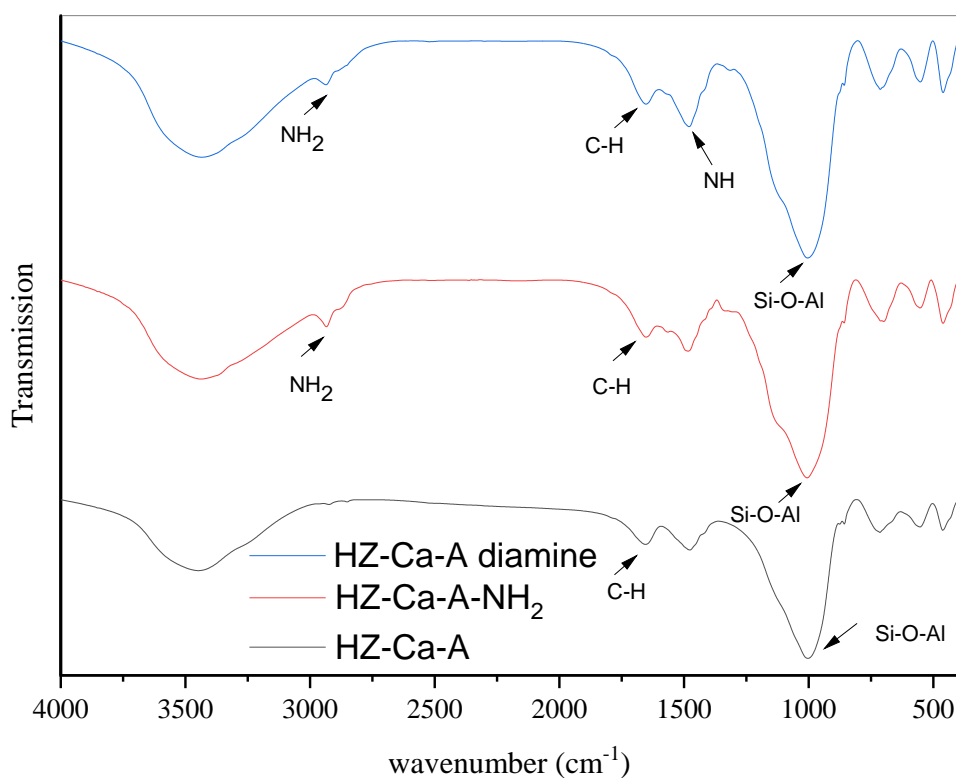


Figure 6: FT-IR spectra of H-Ca-A before and after grafting of amines

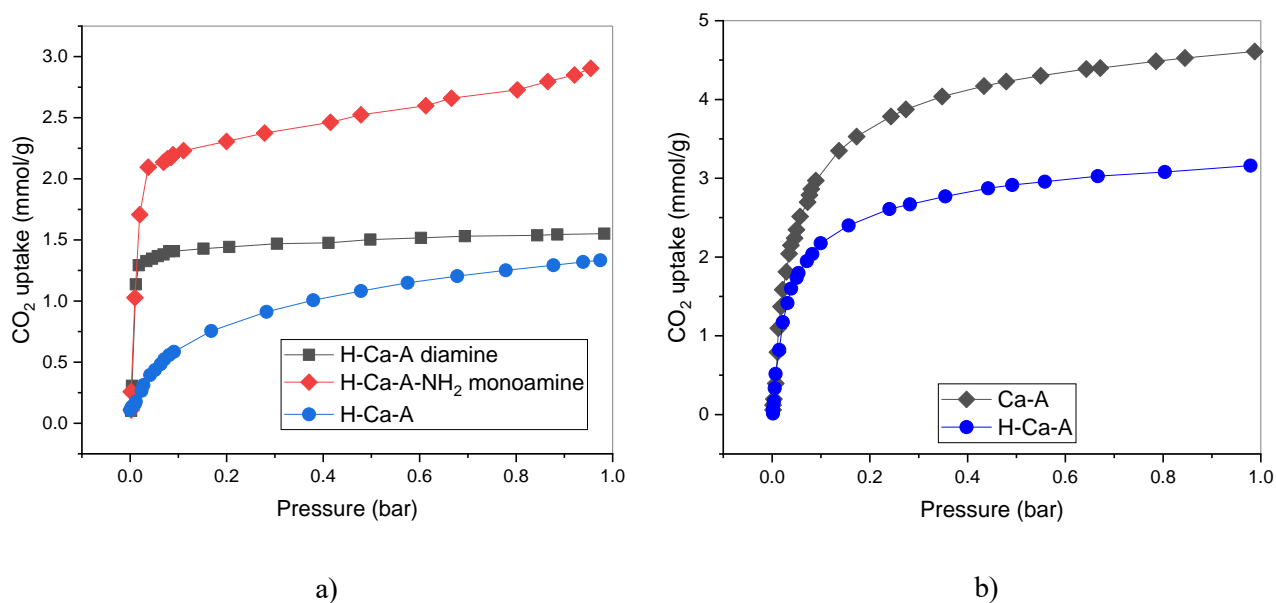


Figure 7: CO₂ uptake properties of a) aminosilane grafted H-Ca-A at 60 °C and 0-1 bar pressure, b) Conventional Ca-A zeolite and H-Ca-A at 25 °C and 0-1 bar pressure

Upon grafting with APTMS (monoamine), a significant enhancement in CO₂ uptake on H-Ca-A is evident, particularly evident in the low-pressure range. This improvement stems from the combined effect of aminosilanes grafted onto the mesopore surfaces and adsorption sites within the LTA zeolite, synergistically contributing to heightened CO₂ uptake. Conversely, H-Ca-A-diamine exhibited a reduced CO₂ uptake capacity compared to H-Ca-A-NH₂, although still showcasing an improvement

over untreated H-Ca-A. Notably, APTMS demonstrates superior efficacy in CO₂ capture compared to diamine (TMPED), underscoring the preference for APTMS functionalization across various substrates, including conventional mesoporous silicas, for comprehensive evaluation of substrate impacts on CO₂ capture properties. The most promising outcomes were observed with APTMS-functionalized H-Ca-A. To gauge the practical implications of its carbon capture capabilities, a comparison was made with a commercially available mesoporous adsorbent capable of accommodating aminosilanes. **Figure 8** illustrates the CO₂ adsorption isotherms of H-Ca-A-NH₂ alongside those of a conventional mesoporous material (MCM-41) functionalized with APTMS, recorded at 60°C. The incorporation of mesoporous domains notably bolstered the CO₂ adsorption capacities in the amine-functionalized hierarchical LTA zeolites, surpassing those of conventional aminosilicas across all pressure levels examined. Particularly noteworthy was the superior CO₂ capture performance of H-Ca-A-NH₂ among the hierarchical LTA zeolites, attributed to its larger mesopore volume and correspondingly higher amine content.

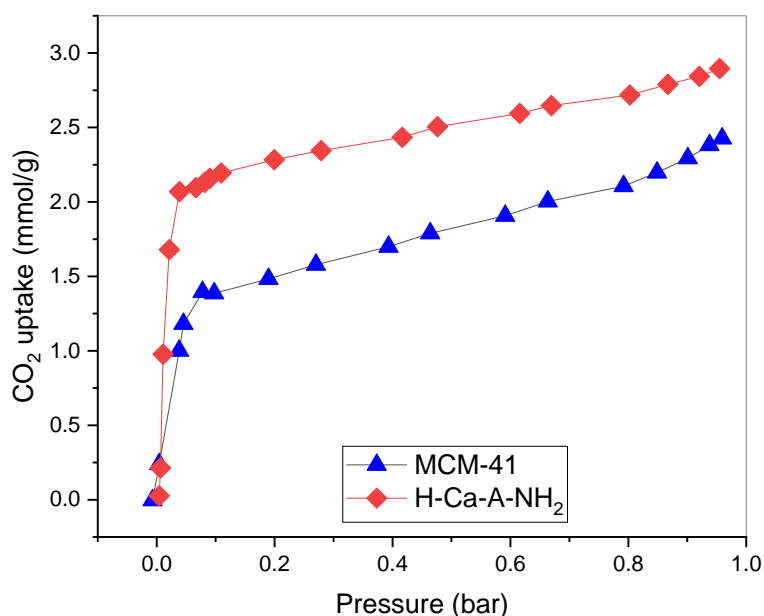


Figure 7: CO₂ Adsorption isotherm of APTMS functionalized H-Ca-A-NH₂ and APTMS functionalized conventional mesoporous material (MCM-41) at 60 °C.

The quantities of CO₂ adsorbed in conventional aminosilicas (MCM-41) fall short of the levels achieved by H-Ca-A-NH₂, as synthesized in this investigation. The potential application of the developed adsorbents primarily targets high-temperature CO₂ capture scenarios, such as those encountered in coal-fired power plants, which is why the CO₂ uptake measurements were conducted at 60 °C.

Conclusion

In this study, we successfully synthesized a hierarchical LTA zeolite with mesoporous architectures by incorporating a surfactant into the reagent mixture. Functionalizing these mesopores with alkylamine groups significantly enhanced CO₂ binding, even at low partial pressures. Optimal CO₂ uptake was achieved with APTMS (monoamine) grafted onto the hierarchical LTA zeolite. By leveraging both the

alkylamine groups on the mesopore surfaces and the active sites within the LTA zeolite, the material demonstrated significantly higher CO₂ uptake compared to conventional aminosilicas like MCM-41. The results indicate that the introduction of amine groups into the mesoporous structures of hierarchical zeolites is a promising strategy for developing high-performance CO₂ capture materials. Future research will aim to further improve CO₂ uptake capacity by exploring alternative microporous domains, such as ETS-10, AM-6, and SUG-27, to replace the LTA framework.

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Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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