

EFFECT OF PRESSURE ON CO₂ CAPTURE USING PIPERAZINE-IMPREGNATED 13X ZEOLITE IN FLUIDIZED BED.

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Abstract: By focusing on adsorbent modification, we can enhance CO₂ adsorption efficiency and contribute to mitigating its environmental impact. Using modified 13X zeolite with amines can improve its CO₂ capture efficiency under non-conventional conditions. SEM, EDS, and BET analyses can evaluate the loading of 2% piperazine onto 13X zeolite. The impregnation process was successful, as evidenced by the detection of nitrogen in the scans. Surface modification resulted in a reduction of pore space, which affected the specific pore volumes and surface areas. We performed hydrodynamic tests in a continuous fluidized bed by varying the pressure. We found that while porosity remained unchanged, increasing the pressure decreased the minimum fluidization velocity. The lowest speeds (umf) for pristine 13X at gauge pressures of 0, 1, 2, and 3 bars were 5.3, 6.6, 7.1, and 7.9 cm/s, respectively. For 13X zeolite modified with piperazine, they were 5.9, 7.2, 7.8, and 8.6 cm/s. After hydrodynamic tests, the employed superficial fluidization velocity was $u_g = 1.5$ umf at bed height = 5 cm and temperature = 25 °C. We assessed the adsorption of CO₂ on 13X zeolite through breakthrough curve experiments, determining the adsorption capacity before and after impregnation with piperazine under varying pressure conditions. The results indicated that at a pressure of 3 bars, the adsorption capacity of the modified zeolite was 3.02 mmol/g, compared to 2.85 mmol/g for the unmodified zeolite. This suggests that applying pressure along with piperazine loading enhances CO₂ adsorption on the zeolite.

Introduction

Growing concerns about adapting fossil fuels as a source for consuming energy can be considered a big problem due to releasing gas pollutants like methane (CH₄), nitrogen oxides (NO_x), and especially carbon dioxide (CO₂) gas into the atmosphere, which causes global damage to the environment as well as human beings [24].

According to the study mentioned by the intergovernmental panel on climate change (IPCC), the amount of greenhouse gas emissions has to be minimized by 50 to 80% by 2050 [19].

Absorbents and adsorbents are used in post-combustion capture technology to remove CO₂ from flue gases [16]. Despite its high energy costs, thermal degradation, and evaporative losses, absorption uses an amine-based solvent for chemical reactions [5]. CO₂ is separated from emission gases through a chemical reaction. Nevertheless, it encounters difficulties like thermal degradation and water in the amine solution.

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Through gas-solid interactions, adsorbents selectively capture CO₂, with pore size, partial pressure, and temperature being important variables [9]. Pressure swing adsorption (PSA) and temperature swing adsorption (TSA) are two forms of adsorption [10]. According to Lee and Park (2015), increasing adsorption capacity and CO₂ capture selectivity in adsorbents is essential to addressing different sources of combustion emissions.

Commercially available porous adsorbents such as activated carbon [1], [7] and zeolite [4], [2] have been widely studied as candidate materials for CO₂ adsorption. However, due to the relatively weak physical adsorption interaction between the adsorbent and CO₂, they have low CO₂/N₂ selectivity [14], [17]. Therefore, the performance of these adsorbents including 13X zeolite in capturing carbon dioxide could be changed positively if it is modified with amine groups, particularly in non-conventional conditions.

At room temperature, zeolites have a high capacity for CO₂ adsorption; however, as the temperature rises, this capacity typically falls. At higher temperatures, CO₂ adsorption can be enhanced by modifying zeolites with amines, i.e., at higher temperatures, polyethyleneimine (PEI) has been demonstrated to improve CO₂ adsorption. Chen et al. investigated the modification of zeolite 13X with PEI and discovered that, at 100°C, the modified zeolite had a higher capacity for CO₂ adsorption than the unmodified zeolite [3].

The exothermic nature of 13X zeolite towards the CO₂ adsorption process may limit its effectiveness at high temperatures. According to Karka et al., after the modification of 13X zeolite with PEI amine and utilizing high temperature, it actually facilitates CO₂ adsorption because the flexibility of the amine groups at elevated temperatures likely plays a crucial role in increasing the available active sites, improving the material's effectiveness for CO₂ capture [13].

Other conditions can enhance CO₂ capturing by pristine 13X zeolite, such as pressure. Higher pressure increases CO₂ concentration near zeolite, leading to more frequent collisions and interactions. This increases the driving force for gas molecules to occupy adsorption sites. Under higher pressures, zeolite can reach its saturation capacity more effectively, allowing for greater CO₂ uptake [23]. Adsorption isotherms show that CO₂ uptake increases with pressure until a maximum value is reached.

Zhang et al., however, investigated the CO₂ adsorption on high-surface area activated carbon (AC-h) and 13X zeolite. At various pressures and temperatures, they measured the CO₂ adsorption and kinetic curves on the two adsorbents. The findings demonstrated that zeolite adsorbed over 13 times as much CO₂ as AC-h at very low pressures. However, because of its larger surface area and total pore volume, AC-h demonstrated a greater adsorption capacity of CO₂ at higher pressures than 13X zeolite [25].

A technoeconomic study of amine-grafted zeolites and their kinetics for CO₂ capture was presented by Tejavath et al. and found that amine loading enhanced CO₂ capture up to a specific concentration over 13X-DETA-40, demonstrating an adsorption capacity of 1.054 mmol of CO₂ per gram of zeolite at atmospheric pure CO₂ gas at 75 °C in a very short amount of time [22].

13X zeolite was investigated by Fashi et. al. when the adsorbent was impregnated with piperazine 2% (the best results obtained with this ratio) in a batch reactor at 25 °C and 8 bar. The results showed positive variation in maximum adsorption capacity in batch experiment from 193.844 to 242.084 mg/g [6].

In this research, 13X zeolite has been used and also loaded with 2% piperazine, and it is tested in the adsorption process using the fluidized bed technique while utilizing

pressure as an effective parameter to investigate the adsorption capacity, also the bed height was examined in this study.

Experimental work

Material

13X zeolite purchased from China had the following characteristics: particle diameter 0.4-0.8 mm, total pore volume 0.250 cm³/g, surface area 560.5 m²/g, specific gravity 1.44, moisture content ≤ 0.5 wt.%, and bulk density 650 Kg/m³. Piperazine solid crystals (sigma Aldrich, 99%) was purchased from the market. Methanol (Lab Alley 99.8%) was also purchased from the market. CO₂ and N₂ gases of 98% purity each were provided from Al-Bilal Gas Company in Baghdad. Argon gas of purity 99% was provided from chemical department laboratory.

Impregnation

The pellet particles of 13X zeolite were exposed to 120 °C in a drying oven to eliminate any humidity. Also, the preparation of piperazine 2% was followed by the procedure that was proposed by Fashi et al. (the best results obtained by their investigation using this weight percent). The mixed percentage involving piperazine and methanol was conducted in a stirrer for 15 min. The amount of adsorbent used in our work was 45 g (5 cm), and 90 g (10 cm) bed. Then the mixture was subjected to further mixing under 35 °C for 2 h; after that, the sample was filtered, washed with methanol, and dried in the oven at 60 °C for 12 h to eliminate methanol.

Continuous experimental rig

A schematic of the laboratory fluidized bed setup for CO₂ adsorption tests using a CO₂/N₂ gas mixture is shown in Figure 1. The glass QVF column has an internal diameter of 40 mm and a height of 500 mm, featuring two 0.35 mm aperture sieves at both the top and bottom to ensure proper gas distribution while retaining packing materials. Temperature sensors and pressure gauges are installed to monitor the process conditions, and flow meters measure the gases entering and leaving the system. For the experiments, CO₂ and N₂ gas cylinders were obtained from Al-Bilal Gas Company in Baghdad, each with a purity of 98%. The concentration of CO₂ at the outlet was analyzed using a GEOTECH CO₂ analyzer (Geotechnical Instruments, BIOGAS 5000, United Kingdom).

In this experiment, the particles used are group Geldart B. The minimum fluidization velocity u_{mf} for the solid particles has been calculated experimentally with the variation of pressure while utilizing the Wen and Yu equation to confirm voidage at minimum fluidization was independent of pressure [21].

$$\Delta p = (\rho_s - \rho_g)(1 - \varepsilon_{mf})Hg \quad (1)$$

Where Δp (pa) is the pressure drop, ρ_s (kg/m³) is the solid particle density, ρ_g (kg/m³) is the gas density, ε_{mf} is voidage at incipient fluidization, H (m) bed height at incipient fluidization, g (m/s²) is gravity constant.

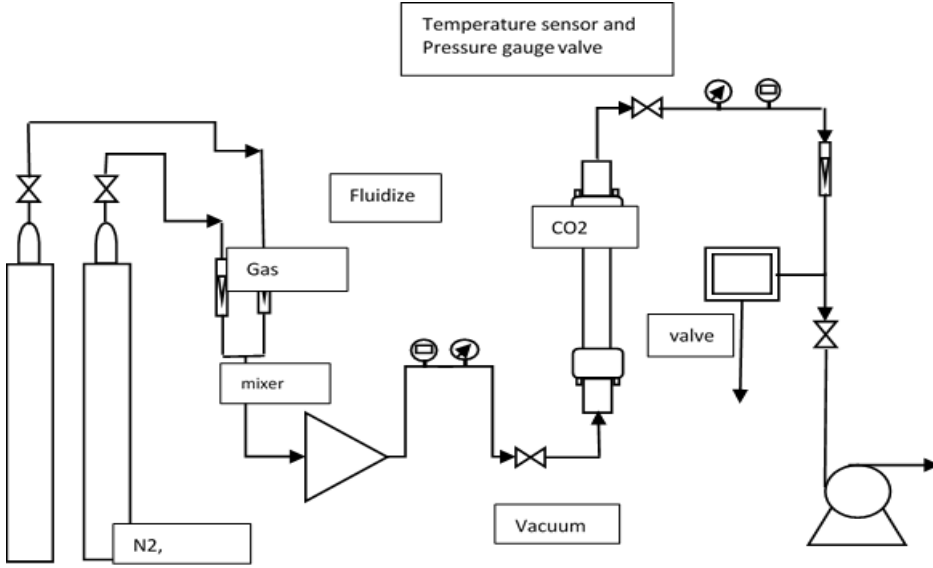


Figure 1: Schematic diagram of experimental setup for CO₂ adsorption in fluidized bed

For a given temperature and pressure of CO₂, the specific equilibrium amount of adsorbed CO₂ can be determined by integrating the breakthrough curve based on the following mass balance equation [20]:

$$q_{ads} = \frac{Q}{W} \int_{t=0}^{t=t} (C_0 - C_t) dt \quad (2)$$

where q_{ads} (mmol/g) is the adsorption capacity, Q (L/min) is gas feed flow rate, W (g) is amount of sorbent, t (min) is time of adsorption, C_0 and C_t (mmol/L) are gas inlet and outlet concentrations respectively. Time used in figures were in seconds. The conditions for each adsorption experiment can be found in Table 1. The solid adsorbent had been heated to 100 °C to eliminate any traces of moisture and CO₂ adsorbed molecules, and then it was exposed to argon gas prior to each run. In a typical experiment, the sorbent was loaded in the column to reach the desired bed height level.

Table 1: Experimental data

Experimental condition	Data
Bed height, H	5 cm
Column length, L	500 mm
Column diameter, D	40 mm
Inlet CO ₂ concentration, C ₀	14 %
Inlet gas velocity, u _g	u _g = 1.5u _{mf}
Vacuum pressure	0.1 bar
Adsorption temperature, T	25 °C
Gauge pressure, P	0, 1, 2, and 3 bar

A series of adsorption experiments were performed at a temperature of 25 °C and different pressures while keeping other variables constant (bed height, temperature, and initial concentration of CO₂). This process was carried out in a continuous

operation method, and the amount of carbon dioxide adsorbed was equivalent to the area under the breakthrough curve and through the use of Eq. 2.

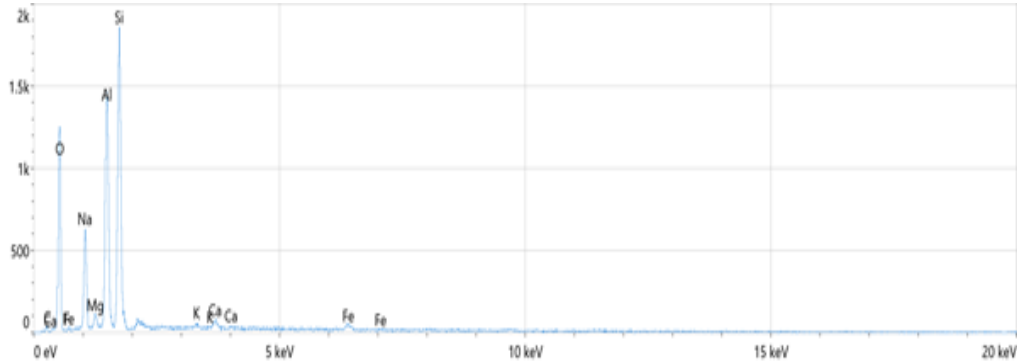
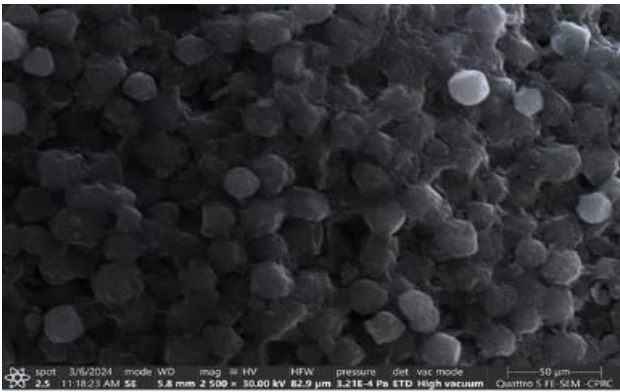
Results and discussion

Characterization

As demonstrated by Figure 2, the SEM images of the zeolite 13X and the modified one with 2% piperazine showed no discernible difference from those of the original zeolites, clearly demonstrating that the piperazine modification had not harmed the zeolites' crystal structures. On the surfaces of the modified zeolites, the N element was dispersed uniformly. According to EDS analysis, the modified zeolite 13X had a N content of 2.4%. The successful loading of the 2% piperazine modification onto the zeolites was further demonstrated by surface scanning and EDS analysis.

(a)

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	3.6	0.2	2.1	0.1
O	57.7	0.3	46.1	0.3
F	0.0	---	0.0	---
Na	9.6	0.1	11.1	0.1
Mg	0.8	0.1	1.0	0.1
Al	11.1	0.1	15.0	0.1
Si	16.5	0.1	23.1	0.1
K	0.1	0.0	0.3	0.0
Ca	0.3	0.0	0.5	0.0
Fe	0.3	0.0	0.9	0.0



(b)

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	4.5	0.2	2.8	0.1
N	3.3	0.3	2.4	0.3
O	60.4	0.3	50.7	0.3
Na	8.4	0.1	10.1	0.1
Al	9.4	0.1	13.3	0.1
Si	14.0	0.1	20.6	0.1

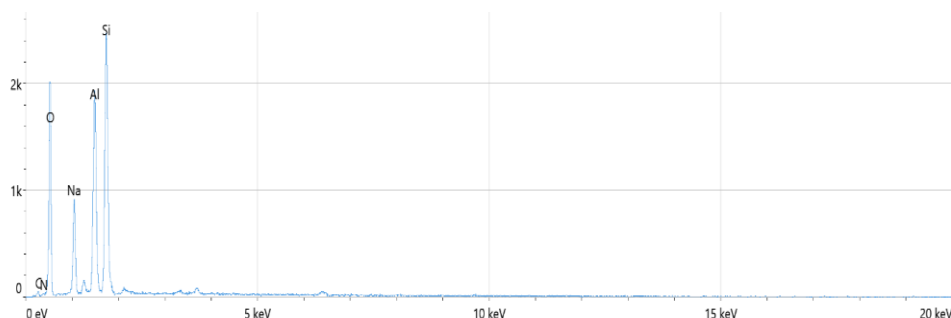
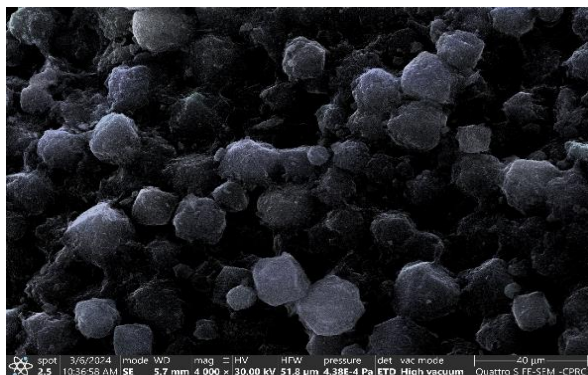


Figure 2: SEM and EDS pictures of a) pristine 13X, b) modified 13X zeolite

The BET method was utilized to measure pore volume and specific surface area. Table 2 presents the structural characteristics of the prepared adsorbents. The modified zeolite 13X exhibits a decrease in performance value, attributed to the infiltration of piperazine molecules into its pores. As indicated in Table 2, all parameter values declined following modification, suggesting that piperazine has filled and occupied the pores of zeolite 13X. This surface modification reduces pore space, leading to changes in specific pore volumes and surface areas [18], [11]. Two densities were been calculated experimentally one for pristine and the other for the modified and there was little difference as shown in Table 2.

Table 2: Pore volume and surface area of pristine and modified 13X zeolite

Material	Density kg/m ³	Pore volume, cm ³ /s	Surface area, m ² /g
pristine 13X zeolite	1420	0.2445	559.429
modified 13X zeolite	1450	0.201	503.43

Minimum fluidization velocity

We utilize the Kunii and Levenspiel chart to explore the impact of pressure on the minimum fluidization velocity. Figure 3a shows the relation between the superficial

velocity variation and the bed pressure drop (Δp) at different pressures in bed height of 5 cm. The obtained minimum fluid velocity was decreased as the pressure, respectively, in modified as shown in Figure 3b. It makes sense given that increasing the gas pressure led to greater gas density, which causes more drag and reduces the velocity needed to fluidize [26]. The superficial velocity was kept constant at $u_g = 1.5u_{mf}$.

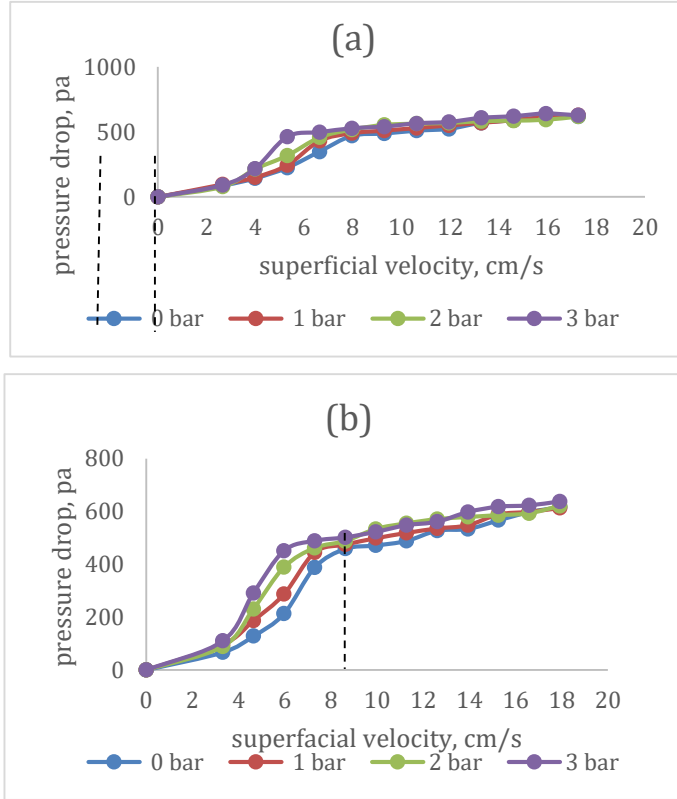


Figure 3: Relationship between superficial velocity and pressure drop at various pressures and constant bed height= 5 cm, a) pristine, b) modified

By utilizing Eq. 1, the bed voidage at incipient fluidization (ϵ_{mf}) was determined which it was 0.355 and can be regarded as pressure-independent. Because the pressure drop was influenced by the weight of the bed particles, the pressure did not significantly alter it [15]. The pressure, minimum fluidization velocity u_{mf} , superficial velocity u_g , and flow rate Q , are shown in Table 3. Zhang et al. found that the pressure behavior of Geldart B particles of 13X zeolite in comparison with activated carbon was in line with these patterns [25].

Table 3: Minimum fluidization velocity and voidage data at different pressures

Pressure, bar	Pristine 13x zeolite			Modified 13x zeolite		
	u_{mf} , cm/s	$u_g=1.5u_{mf}$	Q , L/min	u_{mf} , cm/s	$u_g=1.5u_{mf}$	Q , L/min
0	5.3	7.95	5.9	5.9	8.85	6.6
1	6.6	9.9	7.4	7.2	10.8	8.1
2	7.1	10.65	8.0	7.8	11.7	8.8
3	7.9	11.85	8.9	8.6	12.9	9.7

Breakthrough curve

As illustrated in Figures 4a and 4b, the effect of pressure was studied on CO₂ adsorption capacity using the breakthrough concept to visualize the variation in the pristine and modified 13X zeolite for each bed height. The superficial velocity was maintained at $u_g = 1.5u_{mf}$, with u_{mf} being determined in accordance with Figure 3, and bed height was 5 cm with gauge pressure variations in the range of 0, 1, 2, and 3 bars. Both mentioned figures emerged with the same response with respect to the higher pressure, which would result in an earlier breakthrough point, while the later ones were due to the lower pressure. Higher pressures cause faster adsorption rates, which causes breakthroughs to occur earlier. There are several consequences that arise when the pressure rises. Since a fixed inlet mole fraction is being used, the equilibrium sorbent loading first increases due to an increase in the CO₂ partial pressure. Secondly, the rate of CO₂ molar flow rises. There will be a third benefit: quicker intrinsic adsorption kinetics. In the context of fluidization and hydrodynamics, fourth, one could say that fluidization at high pressures is more [12].

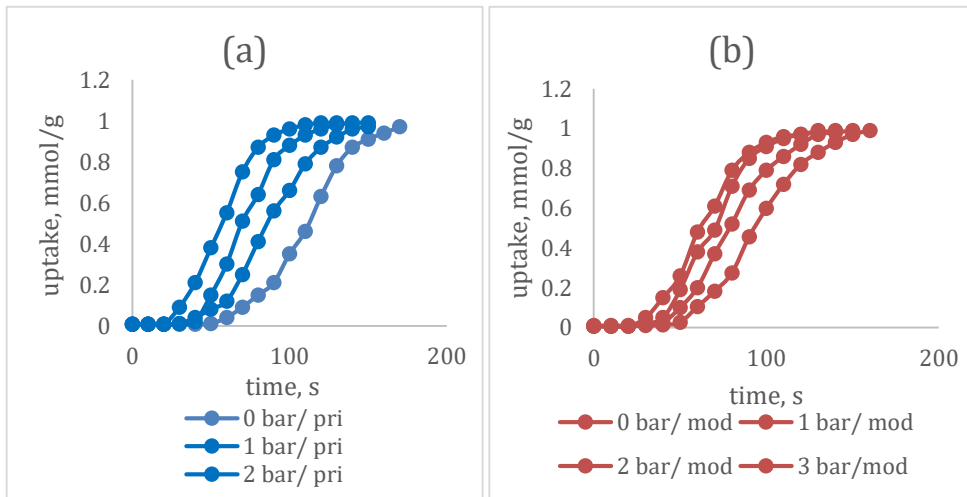


Figure 4: Breakthrough curve comparison for CO₂ capture on 13X zeolite under various pressures a) pristine, b) modified

Regarding the differences shown in Figures 5a, 5b, 5c, and 5d between the modified zeolite with 2% piperazine and the pristine zeolite along with the variations in pressures. Utilizing Eq. 2 to calculate adsorption capacity for each breakthrough curve, they showed us that there was a convergence between the two curves in the same figure. The adsorption capacities for pristine and modified for Figure 5a were 1.73 and 1.49 both at 0 bar, and for Figure 5b, they were 2.48 and 2.26 at 1 bar, respectively. This convergence reached Figure 5c, where the applied pressure value was 2 bar and the capacity records were 2.767 and 2.645, which we have noticed an almost match. Subsequently, the piperazine modification curve grows larger than the pristine material curve in Figure 5d, and the capacities were 2.85 and 3.02 for pristine and modified, respectively, where the applied pressure increases to 3 bars. Table 4 illustrates the dissimilarities in the capacity values.

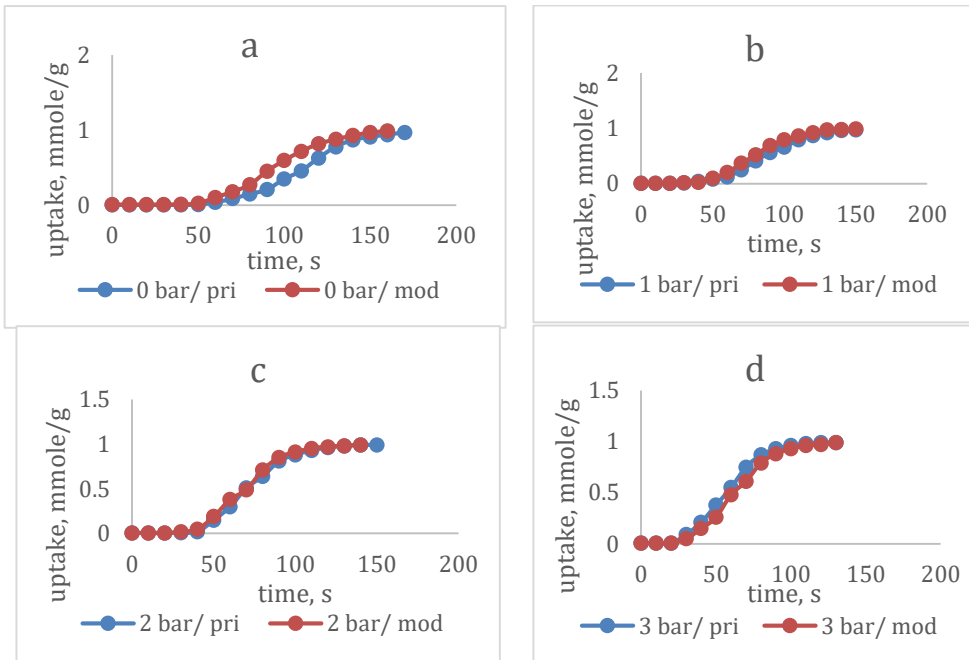


Figure 5: comparison between pristine and modified 13X zeolite under gauge pressures a) 0 bar, b) 1 bar, c) 2 bar, and d) 3 bar at bed height = 5 cm

Piperazine modification of 13X zeolite can increase its adsorption capacity, especially at high pressure for the following reasons [8]:

Surface Area: The modification of 13X zeolite by piperazine can reduce surface area but allowing for more active adsorption sites.

Enhanced contacts: By adding functional groups, piperazine can improve selectivity and capacity by enhancing contacts with particular adsorbates.

Pressure Effects: When there is high pressure, gas molecules are penetrated into the zeolite structure. This can cause the modified zeolite to more readily accept the higher adsorbate density, which would increase the amount of adsorption overall.

Chemical Affinity: Piperazine's amine groups can strengthen contacts with acidic or polar adsorbates, hence boosting capacity.

Structural Stability: By making this alteration, the zeolite may be able to withstand harsh environments and continue to function as intended.

Table 4: Adsorption capacities for pristine and modified under various pressures

Gauge pressure, bar	Pristine 13X, mmol/g	Modified 13X, mmol/g
0	1.73	1.49
1	2.48	2.26
2	2.767	2.645
3	2.85	3.01

Conclusions

After conducting EDS and SEM analyses of the material before and after the impregnation process, it was found that the nitrogen element appears in the analysis. There is also a decrease in pore volume and surface area after

impregnation, which means that the loading process was successful.

The minimum fluidization velocity of particles under different pressures was investigated through experiments. The superficial velocity was maintained constant at ($u_g=1.5u_{mf}$). The obtained minimum fluidization velocities decreased as the pressure increased. At gauge pressures of 0, 1, 2, and 3 bars, the minimum velocities for pristine 13X zeolite were 5.3, 6.6, 7.1, and 7.9 cm/s, while for modified 13X zeolite, they were 5.9, 7.2, 7.8, and 8.6 cm/s respectively. Notably, the voidage did not change.

Regarding the adsorption capacity, the effect of pressure was significant in the breakthrough curve using a fluidized bed. The effect gradually became noticeable on modified against pristine when the pressure was 3 bars and the capacity of impregnated was 3.02 mmol/g, while that of pristine was 2.85 mmol/g.

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