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A comparative study of CO₂ capture by amine grafted vs amine impregnated zeolite 4A

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Abstract. Zeolite 4A was functionalized via grafting and impregnation techniques using 0.3 wt% of isopropylamine (IPA) and (3-aminopropyl) trimethoxysilane (APTMS) respectively. Physicochemical properties of zeolite 4A containing binder were changed after amine loading, as both specific surface area and pore volume was decreased by 1.52 %, 18.18 % and 28.06 %, 90.90 % for Z4A-IPA (IPA impregnated zeolite 4A) and Z4A-APTMS (APTMS grafted zeolite 4A) respectively. The adsorbed CO₂ amount of Z4A, Z4A-IPA, and Z4A-APTMS were 1.58 mmol g⁻¹ (6.95 wt%), 2.31 mmol g⁻¹ (10.16 wt%) and 1.05 mmol g⁻¹ (4.62 wt%) respectively at 1 bar and 25 °C. Noticeably, among all the studied adsorbents, Z4A-IPA displayed the highest adsorption capacity of 1.39 mmol g⁻¹ (6.11 wt%) at 0.15 bar, which is akin to the CO₂ partial pressure of post-combustion flue gas. The multilayer tethering of the bulkier APTMS on the zeolite external surface resulted in the partial blockage of the pores and hence the facile access of CO₂ inside the pores of zeolite was hindered. However, Z4A-APTMS was found to be more thermal stable than Z4A-IPA. Furthermore, we observed that physisorption was playing a vital role in CO₂ adsorption for pristine zeolite 4A (Z4A), whereas, after amine incorporation, the presence of amine induced the heterogeneous interaction between CO₂ and sorbents.

1. Introduction

Carbon dioxide produced by the burning of fossil fuel is the main anthropogenic contributor to the climate change. As greenhouse gas emission is a serious environmental concern, various strategies have been devoted to capturing and storage of CO₂. Among several approaches for Carbon Capture and Storage (CCS), such as pre-combustion, post-combustion and oxy-fuel combustion, post-combustion technique shows fruitful results over the others [1]. Although CO₂ capture by absorption process based on the different types of amine is a well-established procedure, it suffers several drawbacks like equipment corrosion, energy consumption, toxicity, inefficient regeneration. Hence one good alternative called solid based adsorption system generates a great interest among researchers because it delivers several advantages such as high CO₂ adsorption capacity, low energy consumption for regeneration, faster kinetics, easy handling, insensitive to moisture, and so on [2]. However, global CO₂ emission is a matter of huge concern, as the amount of CO₂ release is quite higher globally compared to its capture. Therefore a suitable and sustainable adsorbent should be chosen to fulfill the roaring demand. An efficient solid adsorbent should not only show higher CO₂ adsorption capacity but also display higher selectivity, low heat of adsorption and thermochemical stability over prolonged capture cycles. Several porous materials like Zeolite, Metal-Organic Framework (MOF), Activated Carbon (AC), and Silica have been synthesized and extensively investigated for carbon capture and storage. Although adsorbents like MOF-177 exhibited relatively high CO₂ adsorption capacities (33.5



mmol g⁻¹) at room temperature and super-ambient pressure (35 bar), their capacity decreases rapidly to 0.8 mmol g⁻¹ at atmospheric pressure (1 bar) [3,4]. Moreover, their cost is relatively high compared to other adsorbents [3,4]. It is well proven that the post-combustion requires an adsorption at the atmospheric and sub-atmospheric condition, so owing to wide availability, low cost, large surface area, easy in tailoring pore structure, low energy requirement for regeneration, easy surface functionality, zeolite materials are considered to be one of the most promising adsorbents for carbon capture and storage. Siriwardene et al. reported that synthesized zeolite 13X and 4A displayed an adsorption capacity around 3.63 and 3.06 mmol g⁻¹ at room temperature and 1 bar CO₂ partial pressure conditions respectively [5]. As adsorption is an exothermic process, in spite of these synthesized zeolites showed relatively high adsorption capacity at room temperature, their capacity rapidly declines with a rise in temperature and in the presence of moisture. Hence, one way to improve the CO₂ adsorption capacity of zeolite is to create a surface basic sites by incorporating nitrogen functionalities into the zeolite framework [6,7]. In this context, two different strategies have been adopted to increase the nitrogen functionality by means of chemical methods: i) amine grafting and ii) amine impregnation. In amine grafting technique, aminosilane reacts with the surface hydroxyl group of zeolite by base catalyzed condensation reaction to form stable Si-O-Si chemical bonds. Such amine functionalized zeolites exhibit considerably greater thermal stability during adsorption and regeneration cycle. On the other hand, in amine impregnation technique amine diffuses into the porous cavity of a solid bed, due to chemical affinity and concentration gradient [6]. Madden et al. used the post-modification grafting method to graft (3-aminopropyl) trimethoxysilane (APTMS) on zeolite β (40 % loading), which shows superior CO₂ adsorption capacity up to 4.4 mmol g⁻¹ at 0.15 bar partial pressure and temperature of 35 °C [8]. Similarly Chatti et al. impregnated zeolite 13X matrix by monoethanolamine (MEA), isopropanol amine (IPPA) and observed that the adsorption capacity of zeolite modified by MEA (50 wt% loading) increased by approximately 20-30 % (from 0.84 mmol g⁻¹ to 1.10 mmol g⁻¹) in comparison to bare zeolite matrix at 75 °C and 1 bar pressure purged with 15 vol % CO₂ gas [2]. However, in each case, they used high amine loading and large pore size zeolite like 13X, Y etc. The amount of amine loading for both the cases has the significant role in CO₂ adsorption capacity. Despite the overwhelming evidence supporting the enhancement of CO₂ adsorption ability in amine functionalized adsorbents, a concrete comparative study on the effect of both grafting and impregnation of amine at same loading on the CO₂ adsorption capacity of low pore size zeolite like zeolite 4A is essential. In this paper, we report a chemical modification of zeolite 4A granule containing binder via grafting and impregnation using low loading of amine and investigated the effect of the amine on physiochemical properties and CO₂ adsorption properties of amine-modified zeolite 4A.

2. Experimental method

2.1. Materials and synthesis of amine-modified zeolite 4A

Zeolite 4A granule, anhydrous high purity ethanol, isopropylamine (IPA), (3-Aminopropyl) trimethoxysilane (APTMS) were purchased from Sigma-Aldrich and were used directly without any further purification. Prior to amine incorporation, the adsorbent (granule of size 3-5 mm) was continuously ground by means of a planetary ball mill (Fritsch Pulverizer-5) with the ball to powder ratio of 5:1 (w/w) for 12 h in atmospheric condition [9]. After milling, the samples were dried at 120 °C for 12 h under vacuum. Subsequently, the preheated samples were added to IPA and APTMS solution of a fixed concentration of 0.3 wt% in ethanol under slight agitation for 20 min in a round bottom flask. The resultant mixture was undergone vigorous stirring for 24 h, at room temperature. Finally, the mixture was filtered off, washed with ethanol and degassed at 120 °C for 3 h under vacuum. The pristine sample, sample modified with IPA and APTMS were designated as Z4A, Z4A-IPA, and Z4A-APTMS respectively. For the gas adsorption measurements, the pure CO₂ and N₂ (99.99 %) were supplied by INOX Air product India.

2.2. Characterization

Powder X-ray diffraction patterns (P-XRD) were measured on a Rigaku SmartLab advanced diffractometer using monochromatic Cu K α radiation ($\lambda = 1.54 \text{ \AA}$) at a step size of 0.03° over a 2θ range of 5° to 80° . The surface morphology of studied adsorbents and the distribution of element present inside the adsorbents were examined using field emission scanning electron microscope, equipped with energy dispersive X-ray spectroscopy (FESEM model ZEISS Supra-55) operates at a voltage of 15 KV. The thermal characterization was performed using Mettler-Toledo TGA/SDTA851 thermal Analyser. The physicochemical characterizations of the adsorbents were determined using a Quantachrome Autosorb iQ2 TPX automated gas sorption system. Fourier transform infrared spectrometer (FTIR) equipped with an attenuated total reflectance (FTIR/ATR Model FTIR-SP-1 Spectrum one, Perkin-Elmer, MA, U.S.A) used for measuring variation in chemical property during amine modification.

2.3. Adsorption measurements

The CO₂ adsorption measurements at 1 bar and 25 °C were performed using Quantachrome Autosorb iQ2 TPX automated gas sorption system equipped with highly accurate pressure transducers and thermostatic bath. The experimental error during the experiment was less than 5%. Prior to the experiment, samples were degassed at 120 °C for 8 h.

3. Results and discussion

3.1. Amine modification of zeolite 4A matrix

The surface modification of zeolite 4A was done by using both one-pot amine grafting and impregnation procedure as illustrated in Figure 1. The amine grafting and impregnation was carried out using APTMS and IPA respectively.

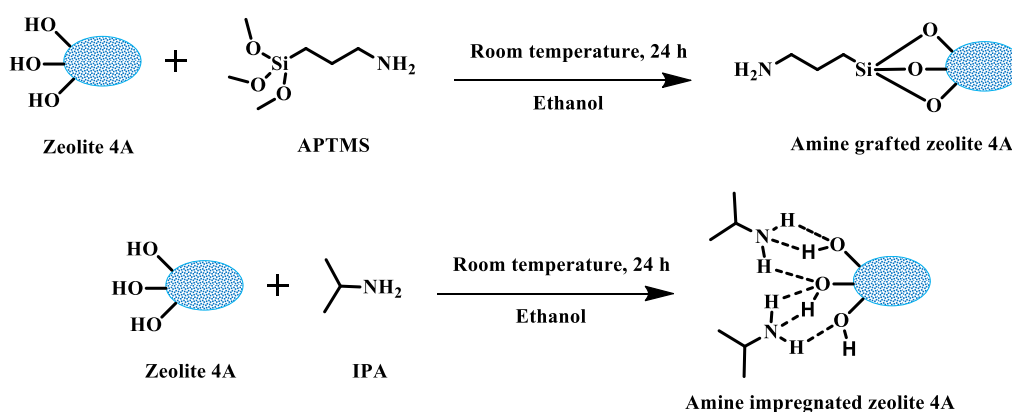


Figure 1. Schematic representation of the modification of zeolite 4A.

P-XRD patterns (Figure 2) of Z4A, Z4A-IPA, and Z4A-APTMS displayed no remarkable difference in peak patterns, suggesting that the crystalline framework of zeolite 4A was well preserved after amine functionalization. Similarly, FESEM images of studied adsorbents inferred the intactness of surface morphology (cubical shape with an average particle size *ca.* 2.5 μm) after amine modification (Figure 2a-c). Moreover, in order to estimate the elemental composition and their distribution, the elemental mapping (Figure 2d-f) was carried out on the studied adsorbents on before and after modification. As APTMS is a silane coupling agent which is basically a silicon-based chemical, so there is an intense increase of silicon and nitrogen content after amine grafting (Figure 2f) as compared to amine impregnation (Figure 2e).

FTIR analysis of the Z4A, Z4A-IPA, and Z4A-APTMS are shown in Figure 2h. The increment in intensity and broadness of band ranging from 3600 cm^{-1} to 2800 cm^{-1} as compared to Z4A was due to several overlapping bands in the single region [10]. All the adsorbents showed vibration bands at 3450 , 975 , 660 cm^{-1} for O-H asymmetric stretching vibrations of the hydrogen-bonded silanol group (Si-O(H)) and Si-Al-O symmetric stretching vibration respectively. The broad absorption band observed at *ca.* $3010\text{--}3500\text{ cm}^{-1}$ was assigned to asymmetric NH_2 stretch and NH_2 deformation. Moreover, the increment in the intensity of peaks within the range $750\text{--}680\text{ cm}^{-1}$ for Z4A-IPA and Z4A-APTMS was noticed due to various rotations and vibration of alkyl and aromatic C-H that confirmed the presence of amine impregnation and grafting in zeolite 4A [11].

Figure 2i displayed a typical type-IV N_2 adsorption-desorption isotherm having a well-defined plateau with modest hysteresis for all the studied adsorbents. Physiochemical properties of the adsorbents are listed in Table 1. S_{BET} (specific surface area) of the studied adsorbents were determined from sub-critical nitrogen isotherms by using Brunauer-Emmett-Teller (BET) equation (at $P/P_0=0.05\text{--}0.3$), V_{micro} (micropore volume) was calculated from Dubinin-Astakhov equation (at $P/P_0=0.2\text{--}0.5$); V_{meso} (mesopore volume), average pore size distribution was analyzed by Barrett-Joyner-Halenda (BJH) equation and V_{total} (total pore volume) was calculated by Vads (at $P/P_0=0.995$). The observation of inverse H_3 type hysteresis at P/P_0 range of $0.4\text{--}0.9$ indicated the presence of bigger and slit-shaped pores, characteristics of hierarchical porous materials [12]. These bigger pores may be generated due to the presence of binder in the zeolite [13]. The specific surface area of zeolite 4A granule before milling was (over $27.11\text{ m}^2\text{g}^{-1}$), whereas after milling found to be ($17.71\text{ m}^2\text{g}^{-1}$). Whereas all the studied adsorbents showed drastically decreased surface area after amine modification. Such low surface area of the pristine zeolite 4A was also supported by literatures due to decreasing aperture size of zeolite 4A by the thermal contraction at 77 K [14–16]. The pore size of the zeolite observes to be too small (0.3 nm) to allow N_2 to probe for measurement at 77 K . Hence the access surface area will be the external surface area. The specific surface area and total pore volume of Z4A, Z4A-IPA, and Z4A-APTMS decreased in the following order $\text{Z4A} > \text{Z4A-IPA} > \text{Z4A-APTMS}$. Although zeolite 4A is highly microporous (pore size $< 2\text{ nm}$), the observed meso-porosity (figure 2i) is due to the presence of binder in zeolite granule [13, 17]. The decreasing order of the modified zeolite 4A is in accordance with the order of molecular sizes and density of respective amine. It is interesting to observe that after amine modification, the mesopore volume fraction was decreased and micropore volume fraction was increased, presumably because of the uniform deposition of amine group mostly on meso and macropores of the original matrix, as well as some degree of micropore development under the action of amine and temperature [18]. The average pore size distribution of Z4A was under mesoporous range (Figure 2i in the inset), became broad after amine modification due to a surface coverage of amine on a fraction of smaller pores (less than 3.3 nm), which were initially present on Z4A [19,20]. The effect of partial pore blockage was more pronounced in grafting condition as compared to impregnation. As amino group plays the role of the hydrogen-bond acceptor, aminopropylsilane anchors on the outer surface of zeolite form hydrogen bond with the available surface silanol group. Sometime self-condensation also happens between aminopropylsilane molecules as a result; they may form a cluster on the zeolite surface and block the pores [20].

Figure 3a reveals the TGA curve of studied adsorbents. All the studied adsorbents displayed similar thermogram with two distinct weight loss stages ranging between temperatures $100\text{--}160\text{ }^\circ\text{C}$ and $200\text{--}650\text{ }^\circ\text{C}$ respectively. Whereas there was only one continuous weight loss up to $650\text{ }^\circ\text{C}$ observed on Z4A. The first major weight loss region in between the temperature range ($100\text{--}160\text{ }^\circ\text{C}$) can be attributed to the removal of moisture, volatilization, and degradation of ethanol from the samples.

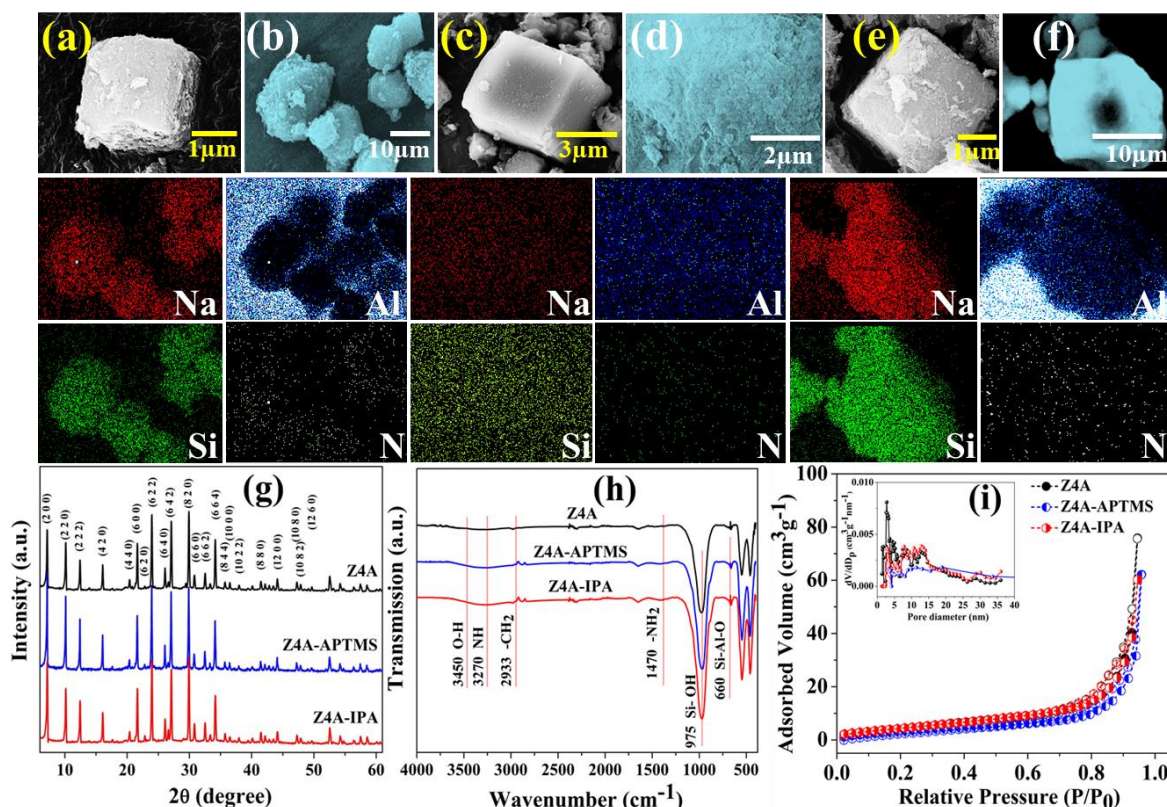


Figure 2. a,c,e) FESEM images of Z4A, Z4A-IPA, and Z4A-APTMS, b,d,f) corresponding elemental mapping, g) P-XRD spectra, h) FTIR spectra and i) N_2 adsorption-desorption isotherm and pore size distribution (inset) of studied adsorbents.

However, a very smooth inflection point was observed between (200–300 °C) which can be attributed to the elimination of amine [15]. The TGA curve of Z4A, Z4A-IPA, and Z4A-APTMS exhibited a weight loss of 18.4%, 20.4%, and 18.2% respectively. It is well known that when zeolite grafted with an amine in presence of polar solvent like ethanol, the Si–OH may be converted into Si–OC₂H₅ by transesterification which makes zeolite hydrophobic compared to prior modification [21]. Hence it favors higher thermal stability for Z4A-APTMS compared to Z4A and Z4A-IPA.

3.2. CO₂ adsorption studies on modified zeolite 4A matrix

The CO₂ adsorption isotherm of studied adsorbents at 1 bar and 25 °C are listed in Table 1. It is obvious that CO₂ adsorption capacity depends upon both specific surface area and the type of chemical functionality anchored on zeolite surface. The hybrid adsorption mechanism (both physisorption and chemisorption) was shown in studied adsorbents. In zeolite 4A, physisorption played a vital role in CO₂ adsorption, exhibited only 1.58 mmol g^{−1} adsorption capacity (6.95 wt%). However, after amine impregnation and grafting, chemisorption played a dominant role (Figure 3b) especially at low pressure [22]. The adsorption capacity increased to 2.31 mmol g^{−1} (10.69 wt%) after IPA loading, whereas it was decreased after APTMS loading to 1.05 mmol g^{−1} (4.62 wt%). Despite that the elemental mapping (Figure 2d-f) suggested a higher nitrogen content for Z4A-APTMS, compared to Z4A-IPA, Z4A-APTMS displayed lower CO₂ adsorption properties. It is well understood that due to amine immobilization, the basicity increases with the formation of extra adsorption sites by the presence of the free NH₂ group.

Table 1. Physicochemical properties and CO₂ adsorption performance of the adsorbents.

Adsorbents	$S_{\text{BET}}^{\text{a}}$ ($\text{m}^2 \text{ g}^{-1}$)	PV^{b}					Q^{h}			
		$V_{\text{total}}^{\text{c}}$	$V_{\text{micro}}^{\text{d}}$	$V_{\text{meso}}^{\text{e}}$	$F_{\text{micro}}^{\text{f}}$	$F_{\text{meso}}^{\text{g}}$	(mmol g^{-1})		wt(%)	
							P (bar)			
0.15	1	0.15	1							
Z4A	17.71	0.11	0.02	0.09	18.18	81.81	0.27	1.58	1.18	6.95
Z4A-IPA	17.44	0.09	0.02	0.07	22.22	77.77	1.39	2.31	6.11	10.16
Z4A-APTMS	12.74	0.01	0.01	0.00	100	00	0.10	1.05	0.44	4.62

^a specific surface area; ^b pore volume; ^c total volume; ^d micropore volume; ^e mesopore volume; ^f micropore volume fraction ($V_{\text{micro}}/V_{\text{total}}$); ^g mesopore volume fraction ($V_{\text{meso}}/V_{\text{total}}$) and ^h the amount of CO₂ adsorption at 25 °C at given pressure.

The amine present inside the zeolite cavity binds CO₂ through lone pair electron on nitrogen atom to form Zwitterion intermediates followed by deprotonation of another free amine to form carbamate and ammonium species which augmented the adsorption capacity [2]. However, in grafting case due to the presence of three methoxy group in the APTMS, the amount of multilayer tethering was quite high on the zeolite external surface and blockage the pore mouth. As a direct result, there was a drastic decrease in mesopore volume and increase in micropore volume fractions.

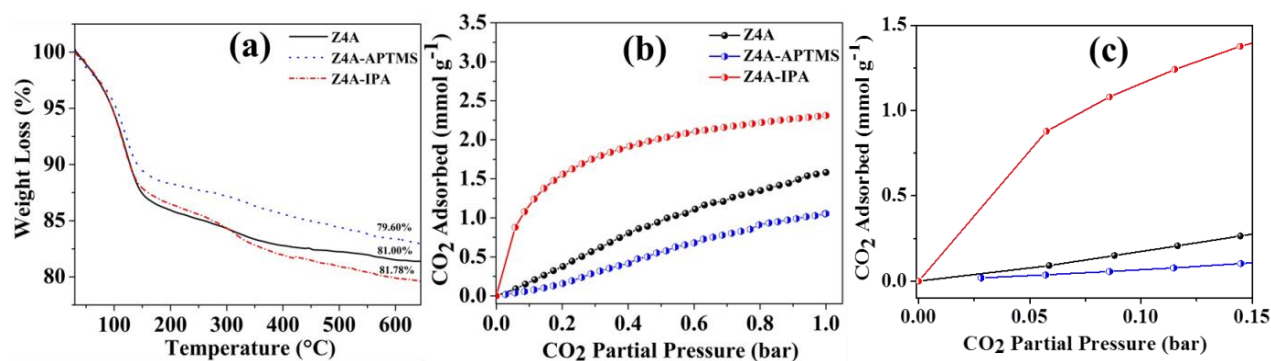


Figure 3. a) TGA curves, b and c) CO₂ adsorption isotherms at b) 1 bar and c) 0.15 bar and 25 °C of the studied adsorbents.

It is also important to discuss that enriched silane provided excess steric hindrance results decrease in CO₂ adsorption capacity [23]. At the same time due to the lower molecular size and density of IPA, it can easily diffuse inside the zeolite surface containing pore, hence due to diffusion control mechanism, it can be exposed to more CO₂. Recently Cogswell et al. explain the effect of amine density on pore blockage of 3-dimensional silica mesoporous framework composed of agglomerated spheres of microporous zeolite beta [24]. He proposed that if the amine density exceeds some critical point, then it starts to hinder CO₂ diffusion to the internal amine site. should be less Nevertheless, the CO₂ adsorption capacity of Z4A-IPA was quite high about 1.39 mmol g^{-1} (6.11 wt%) compared to Z4A, Z4A-APTMS and other adsorbents studied by various researchers, as listed in Table 2 at sub-atmospheric condition (0.15 bar). The steep increase in adsorption trend line (Figure 3c) at 0.15 bar suggests the stupendous utility of Z4A-IPA in post-combustion carbon capture.

Table 2. Comparative data of CO₂ adsorption performance of the amine modified adsorbents in literature.

Adsorbents	Amine	Loading (wt%)	Q	P	Ref.
			(mmol g ⁻¹)	(wt %)	
			0.15		
Zeolite 13X	MEA ^a	50	0.72	3.16	[20]
AC	MEA	0.2	0.18	0.79	[20]
AC	TEA ^b	98	-	-	[20]
MCM-41	APTES ^c	66.41	0.6	2.64	[25]
SBA-12	APTES	61.09	1.03	4.53	[25]
SBA-15	APTES	60.21	1.53	6.73	[25]
ITQ-6	APTMS ^d	42 ^f	0.67 ^g	2.94 ^g	[26]
SBA-15	APTMS	24 ^f	0.24 ^g	1.05 ^g	[26]
SBA-15	APTMS	46 ^f	0.54 ^g	2.37 ^g	[26]
Zeolite 4A	IPA^e	0.3	1.39	6.11	This study

^a monoethanolamine; ^b triethanolamine; ^c (3-aminopropyl)triethoxysilane; ^d (3 aminopropyl)-trimethoxysilane; ^e isopropylamine; ^f amine coverage in % and ^g amount of CO₂ adsorption at 0.11 bar and 20 °C.

4. Conclusions

Herein, we synthesized amine functionalized zeolite 4A (Z4A) via impregnation and grafting of IPA and APTMS respectively. The amine incorporation in Z4A was confirmed by FTIR and elemental mapping. Among amine impregnation and grafting, impregnation was found to be more suitable in post-combustion CO₂ adsorption, where due to the low molecular size and density of IPA, it could easily diffuse inside the zeolite surface containing pore, so diffusion control mechanism became predominate which helped in assessing enrich CO₂. Hence it exhibited superior adsorption capacity of around 1.39 mmol g⁻¹ (6.11 wt%) at 0.15 bar and 2.31 mmol g⁻¹ (10.16 wt%) at 1 bar respectively. However, multilayer tethering of high density APTMS on the zeolite led to the blockage of pore mouth. Therefore, the drastically reduced micropore volume and low surface area resulted in hindered assess of CO₂ and hence adsorption capacity decreased. However, compared to Z4A-IPA, Z4A-APTMS displayed higher thermal stability due to the hydrophobic nature of Z4A-APTMS commenced after silanation. We believe, the augmented adsorption capacity of Z4A-IPA may attract researchers to promote its potential application in efficient CO₂ capture.

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