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Synthesis and Characterization of Aluminum and Zinc Based Aminoterephthalate Metal-Organic Frameworks (Al-ATA and Zn-ATA) for Enhanced Gas Adsorption and Separation

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Abstract:
Metal-organic frameworks (MOFs) have emerged as highly promising materials for gas sorption and separation, owing to their extensive surface areas, customizable pore sizes, and diverse structural properties. This study delves into the mechanisms of gas sorption, strategies for gas separation, and synthesis methodologies of MOFs, with a particular focus on (M=Al, Zn) with aminoterephthalate. The synthesis of these MOFs involves the reaction of metallic salts with 2-amino-terephthalic acid under mild solvothermal conditions, using an ethanol and water solvent mixture. The synthesized MOFs samples were characterized using techniques such as FTIR, SEM, EDX, XRD, BET, and TGA to determine functional groups, surface morphology, elemental analysis, molecular structure, pore sizes, surface areas, and thermal stability respectively. The findings underscore the potential of MOFs such as Zn-amino terephthalate exhibits high CO_2 uptake capacities at low temperature while at high temperature reduces the affinity for CO_2 and adsorbed CH_4 higher compared to Al-amino terephthalate, at various temperatures range (30°C, 60°C, and 100°C) and 1.0 bar pressure of the findings are consistent across various studies.

Keywords: Metal Organic Frameworks, Terephthalate, Metal Clusters, Adsorption Gas, Separation, Gas Storage

1. Introduction

Metal-organic frameworks (MOF), an inorganic–organic hybrid materials with porous crystalline structures, large specific surface area, sufficient active sites, structural diversity and easy modification, formed by self-assembly of metal ions (or clusters) and organic ligands. MOFs have garnered significant attention since their inception in the 1990s due to their unique structural and chemical properties (Rowsell & Yaghi, 2004). MOFs have gained a lot of attention due to their noteworthy characteristics, which included a large specific surface area, a large number of unsaturated sites, and modifiable structure and function (Arendzen et al., 2021; Li et al., 2018). The vital area of gas separation involves the purification of ethane (C_2H_6) from ethylene (C_2H_4), as well as the separation of paraffins and olefins (Bao et al., 2016). Various techniques employed for gas storage and separation, adsorption in porous materials has gained prominence due to its energy efficiency compared to conventional methods like cryogenic distillation or amine scrubbing (Ruben et al., 2004; Singdz et al., 2024).

A range of solid adsorbents has been explored for gas adsorption, including activated carbons, alumina, silicates, zeolites, and MOFs. An effective adsorbent should ideally exhibit both high gas uptake capacity and selectivity (Lyndon et al., 2020; Mehri Lighvan et al., 2023). MOFs, characterized as a versatile class of crystalline nonporous materials composed of metal nodes and organic linkers, are regarded as promising candidates for gas separation due to their tunable properties (Josephine et al., 2010).

MOFs offer two notable advantages: they feature various cage types with adjustable sizes and unsaturated coordination sites that enhance host-guest interactions (Bao et al., 2016), and their small pore windows and closed faces facilitate effective molecular sieving for size-based separations (Liao et al., 2017).

Additionally, MOFs are recognized for their high surface area, porosity, low density, and adaptability in pore size, shape, and structure (Furukawa et al., 2013). Recent years have seen the emergence of numerous MOFs that demonstrate superior performance in separating critical industrial gas pairs, such as C_2H_2/C_2H_4, C_2H_4/C_2H_6, C_2H_2/CO_2, CO_2/CH_4, C_3H_4/C_3H_6, and C_3H_6/C_3H_8, often outperforming traditional zeolites (Li et al., 2018; Liao et al., 2017). The growing demand for efficient gas separation and purification technologies has underscored the limitations of current materials and methods, which often exhibit inadequate adsorption capacity, selectivity, and stability (Chen et al., 2018). MOFs have emerged as promising candidates for gas adsorption and separation applications, owing to their high surface areas, tunable pore sizes, and versatile chemical functionalities (Furukawa et al., 2013; Xu et al., 2020). However, the development of MOFs with optimal properties for specific gas separation applications remains a significant challenge. The synthesis of MOFs with controlled crystal size, morphology, and chemical functionality is crucial for understanding their gas adsorption and separation behavior (Furukawa et al., 2014; Liao et al., 2017).

Compressed Natural Gas (CNG) storage involves compressing natural gas to high pressures in tanks. While effective, this method requires robust, heavy tanks and can be costly due to high-pressure equipment and infrastructure. In contrast, MOFs can store gas at lower pressures, enhancing safety and reducing costs while providing higher storage capacity (Guo et al., 2018). Liquefied Natural Gas (LNG) storage involves cooling natural gas to -162°C to convert it into liquid form for transport and storage. This process is energy-intensive and requires specialized cryogenic tanks. MOFs can adsorb methane at ambient temperatures, making them more energy-efficient and easier to handle (Zura et al., 2016). Activated Carbon and zeolites were widely used for gas adsorption and storage due to its high surface area, but it lacks the selectivity and tunability as MOFs. MOFs can be engineered to enhance interactions with specific gases, allowing for more efficient separation and storage of gases like CO_2 and H_2 (Singh et al., 2024; Zhu & Xu, 2014).

Based on our knowledge, there is no record of MOF synthesis that employed ethanol/water solvent system specifically for gas storage applications. This suggests that the approach adopted by combining ethanol and water as solvents in the synthesis of MOFs is novel and has not been explored in the context of gas storage before. The findings contribute a new approach to the MOF synthesis. Potentially offering unique advantages over existing methods for MOFs gas storage purposes.

The main objective of this study is to conduct a systematic analysis of the physical and chemical properties, separation efficiency, and adsorption capacities of metal-organic frameworks (MOFs) synthesized using two distinct metal centers, namely aluminium (Al), and Zinc (Zn). This research aims to elucidate the effects of temperature variations on the MOFs' performance while maintaining a constant pressure, with a specific focus on their application in utilizing automotive exhaust gases as the source material.

2. Research Methods

Materials

Aluminum chloride hexahydrate, 2-amino terephthalic acid (H_2ATA), zinc nitrate hexahydrate, cobalt (II) chloride hexahydrate, and copper sulfate pentahydrate were sourced from Burgoyne Burbidges & Co. Solvents used include ethanol (EtOH, Merck, 99.9%), butanol (Merck), and distilled water. All chemicals and gases were used as received without any further purification.

Synthesis

Aluminium-amino terephthalate (Al-ATA) was synthesized solvothermally by the addition of 0.63 g of 2-amino terephthalic acid in 75 mL of the solvent (ethanol and water). The mixture was heated to 130 °C in a 500 mL round-bottomed flask in a Teflon-lined autoclave and heated for equipped with a magnetic stirrer. Then, a clear solution containing 1.81 g of previously dissolved aluminum chloride hexahydrate in 75 mL of the solvent (slow dissolution) was added dropwise with the help of an addition funnel over a period of 1 h. The mixture was then stirred further at 130 °C for 14 h. The yellow precipitate was recovered through centrifugation at 10.000 rpm and washed several times with distilled water until a clear supernatant was obtained. The MOF was recovered as a yellow powder upon drying in a vacuum oven at 60°C for about 1 h. To avoid any degradation from moisture, the MOF was stored in an argon-filled glovebox. The method was repeated for the syntheses of Zinc amino terephthalate (Zn-ATA).

Characterization

The synthesized MOF samples were characterized using various techniques, such as X-ray diffraction (XRD) (PHILIPS PW1730, Netherland) of the synthesized samples were acquired by Cu-Kα radiation in 2θ range from 2° to 50° and scanning rate of 2°C min^{-1}, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy FTIR EQUINOX 55 (Bruker Optics, Germany) using KBr pellets as the substrate at RT in the wavenumber range of 400–4000 cm^{-1}., Brunauer-Emmett-Teller (BET) technique was carried out on a BELSORP MINI II analyzer (BEL Japan, Inc.). The N_2 adsorption isotherms were determined, the specific surface area, pore size, and total pore volume values were calculated and all samples were degassed at 393 k for 12 h for the removal of any impurity. Thermogravimetric analysis (TGA) was carried out under heating rate of 10°C min^{-1} under inert gas (N_2) atmosphere at 50 ml min^{-1} flow rate on a Mettler Toledo instrument. The adsorption activity of the MOFs for the oxidation of hydrocarbons, carbon dioxide, and nitrogen, emitted by gasoline-powered engines, was assessed using a fixed-bed reactor and gas analyzer.

Activity test of synthesized Al-ATA and Zn-ATA MOFs

The synthesized MOFs undergo an activity test to assess its performance under varying temperature and pressure conditions. In this experiment, the MOF was subjected to different temperatures—specifically, 30°C, 60°C, and 100°C while keeping a constant pressure of 1.0 bar (Figure 1). This is conducted in a plug-free reactor designed to facilitate smooth gas flow without interruptions. The MOF sample was introduced into the reactor via a dedicated pipeline. The gas source for the experiment was generated from a gas generator, ensuring a consistent and controlled supply of the target gases. As the gas flows through the reactor, it passes over the MOF material, where selective adsorption occurs. This process allows the MOF to interact with the gases, capturing specific molecules based on its unique properties.

Figure 1. Testing of synthesized MOF using Gas Analyzer

3. Results and Discussion

X-ray Diffraction (XRD) Analysis

The XRD patterns, crystallites size, and d- spacing of the synthesized MOFs are illustrated in Figure 2. and Table 1. respectively. The framework of Al-ATA MOF exhibits high crystallinity, confirmed by diffraction peaks at 2θ = 20.5° (0.70), aligning with JCPDS 00-062-1030 (Zhao et al., 2018). Additionally, Zn-ATA MOF shows well-defined diffraction peaks at 2θ = 21.5° (001), supporting its high crystallinity, consistent with literature data. There is no impurity phase observed, demonstrating the successful synthesis of pure-phase MOFs. The XRD findings correlate with FTIR analysis, confirming the integrity of the individual MOFs within the hybrid structure.

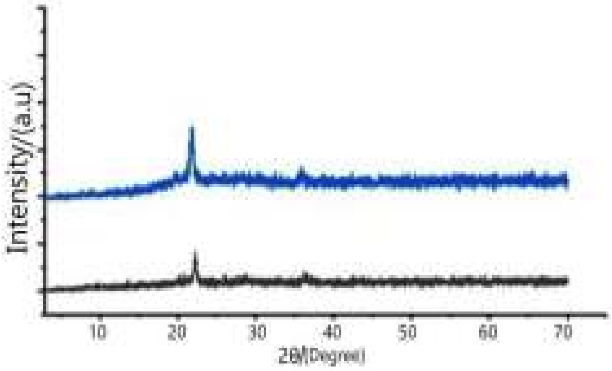


Figure 2. XRD patterns of Al-ATA and Zn-ATA MOFs

Table 1. Crystallite size, d-spacing of the synthesized MOFs calculated using the Scherrer equation and Bragg's law

Sample	2θ	Intensity %	d-spacing (Å)	β (FWHM)	Crystallite size (nm)
Al-ATA	20.15	100	2.15	0.70	116.8
Zn-ATA	21.50	100	2.00	1.00	79.5

BET Surface Area Analysis and Particle Size

Table 2. summarised the specific surface areas and total pore volumes of the samples. The surface areas measured were 709.005 m^2/g for Al-ATA and 2077.340 m^2/g for Zn-ATA, with the latter pore size surpassing previous literature values. The Zn-ATA MOF exceeded the reported surface area of 5900 m^2/g for MIL-101-(Cr)-NH_2 (Younas et al., 2022). Zn-ATA demonstrated the highest specific surface area of 2077.34 m^2/g, with a BJH pore volume of 0.189 cm^3/g, while Al-ATA had the lowest at 705.005 m^2/g and a pore volume of 0.129 cm^3/g. The pore sizes indicated potential fair physical adsorption capacities with an enhanced adsorption performance (Mehri Lighvan et al., 2023). The isotherms of the both MOFs indicated mesopores range structures ($2 < d < 50$ nm) of typical type-I profile according to the IUPAC classification (Elbashir et al., 2024).

Table 2. Brunauer–Emmett–Teller (BET) surface area, pore size and pore volume of the synthesized MOFs.

MOFs	Langmuir S_{BET} (m^2/g)	Pore size (nm)	Pore volume (cm^3/g)
Al-ATA	20.15	100	2.15
Zn-ATA	21.50	100	2.00

FTIR Analysis

The FTIR spectra of synthesized MOFs (Figure 3) revealed distinct absorption bands. Al-NH_2 exhibited strong bands at 1706 cm-1 indicates the presence of absorbed water (C=O stretching), 2213 cm-1 (C≡N stretching), 3017 cm-1 (C-H stretching), and 3328/3688 cm-1 (O-H stretching), indicating the presence of organic linkers, nitrogen-containing species, and surface hydroxylation. Conversely, Zn-NH2 displayed bands at 2107 cm-1 (C≡N/C≡C stretching), 2871 cm-1 (C-H stretching), and 3221/3380/3533/3718/3821 cm-1 (O-H stretching), suggesting nitrogen-containing linkers, unsaturated hydrocarbons, and higher surface hydroxylation. The presence of these characteristic peaks confirmed the integrity of individual MOFs, with a slight decrease in the intensity of amine-related peaks in Zn-NH_2 (MOF)potentially due to the metal center's electron-withdrawing effect (Bauer et al., 2008; Chong et al., 2021; Gascon et al., 2009).

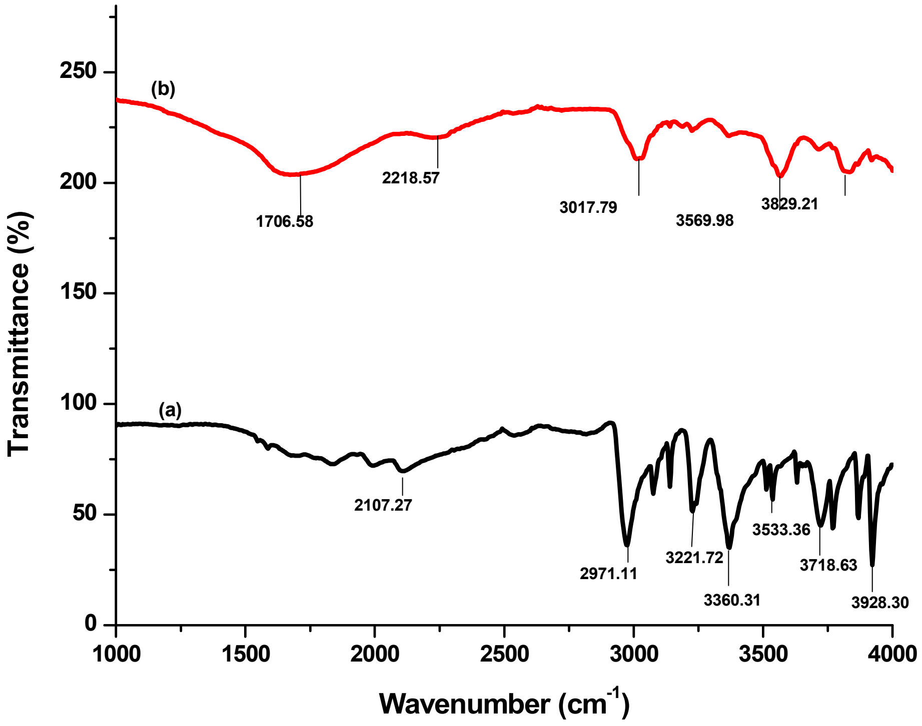


Figure 3. FTIR spectra for (a) Al-ATA and (b) Zn-ATA MOFs

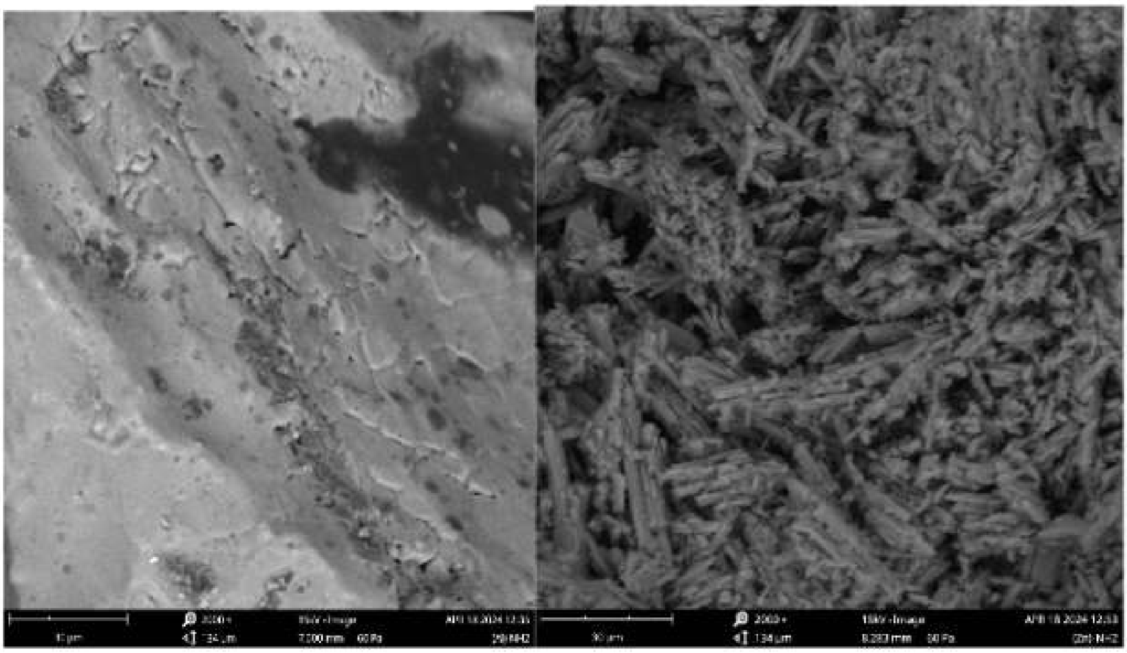
SEM-EDX Analysis

The SEM analysis has produced high-resolution images of the Metal-organic framework, clearly illustrating its porous structure (Figure 4). These images reveal the intricate arrangement of pores within the MOF, which is a defining feature of these materials. The porous nature is crucial as it significantly influences the MOF's ability to adsorb gases and liquids, making it suitable for various applications, such as gas storage, separation processes, and catalysis. The SEM images also allow for the assessment of surface morphology, including the size and distribution of the pores, which can affect the material's overall performance in practical applications.

In addition to the morphological insights provided by SEM, EDX analysis has quantitatively detailed the elemental composition of the MOF. This technique identifies and measures the concentration of each element present in the framework, offering a comprehensive understanding of its chemical makeup. The EDX results can confirm the presence of specific metal ions and organic linkers that constitute the MOF, which is essential for evaluating the framework's structural integrity and functionality. Furthermore, the elemental distribution maps generated by EDX can highlight how uniformly the elements are dispersed throughout the MOF, which may influence its reactivity and efficiency in various applications.

Elemental Composition and Surface Textural morphology

In order to confirm the formation of MIL-101-(Al)-NH$_2$, and MIL-101-(Zn)-NH$_2$, EDX analyses were performed. The corresponding concentrations are shown in Table 3. the elements Al, Zn, Cl, N, C, S and Ca in the synthesized MOFs can be seen in the EDX Table. It has depicted that the samples are not hundred percent pure due to the presence of some foreign elements, calcium, and phosphorus in all the samples. The appearance of the impurities can be attributed to the percentage impurity in the analytical reagents or due to human error. However, the XRD patterns have not indicated any appearance of the impurities, suggesting their insignificance. Moreover, the SEM images of the MOFs' surfaces designated rough, porous morphological apparent materials and have irregular shapes and sizes. These suggested good potentials for the adsorption capacity of the materials.



Al-ATA Zn-ATA

Figure 4. SEM Images of synthesized MOFs

Table 3. The weight percentage of various elemental analysis of Al-ATA and Zn-ATA from EDX analysis

Atomic number	Symbol	Element Name	Atomic Con.
17	Cl	Chlorine	65.13
13	Al	Aluminium	26.16
30	Zn	Zinc	28.78
17	Cl	Chlorine	10.90
6	C	Carbon	22.68
15	P	Phosphrorus	14.34

Thermogravimetric Analysis (TGA)

The thermal stability of the synthesized hybrid MOF was compared with Al-ATA and Zn-ATA under N_2 flow (Figure 5). Al-ATA exhibited minor weight loss up to 250°C due to trapped gases and solvents. A significant weight loss between 400°C and 600°C likely resulted from the decomposition of organic linkers and the collapse of the Al-ATA structure. Zn-ATA showed more considerable weight loss in the 250°C to 300°C range, attributed to trapped molecules, with further weight loss indicating the elimination of OH and NH_2 groups. The hybrid structure displayed weight loss before collapse, attributed to guest molecules, with a notable 30% weight loss from 100°C to 500°C due to H_2ATA decomposition. The TGA curves indicated a downward trend from 500°C to 800°C, signalling the collapse of Zn-ATA and ongoing degradation of Al-ATA suggesting improved thermal stability of the hybrid MOFs compared to Al-ATA (Gecgel et al., 2019).

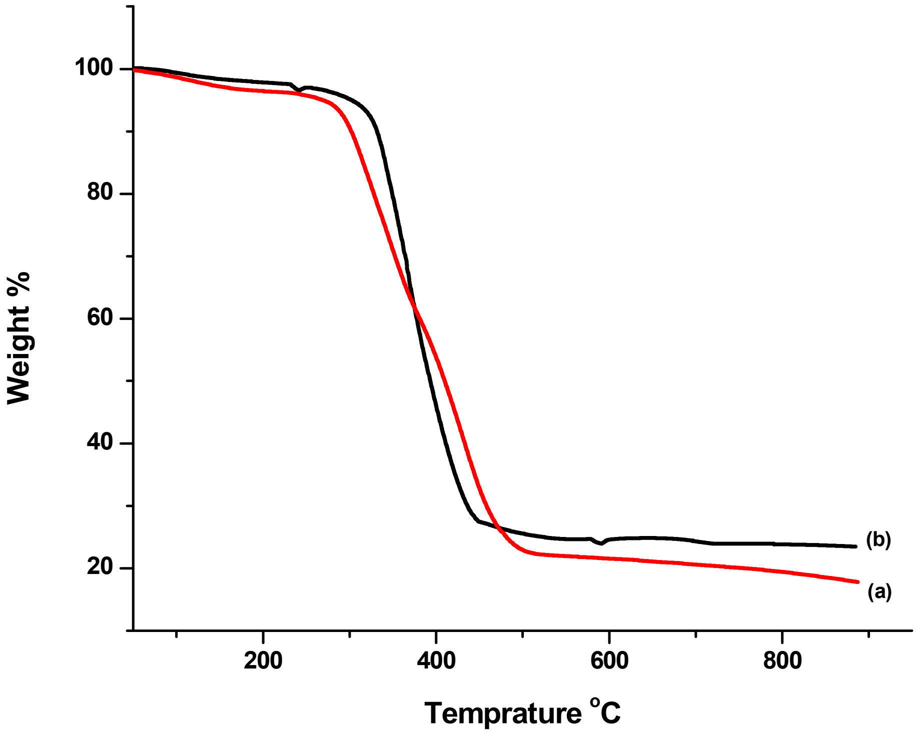


Figure 5. TGA for (a) Zn-ATA (b) Al-ATA

Adsorption of Gas

The gas adsorption for CO_2, N_2, and CH_4 at temperatures of 303 K, 333 K, and 373 K for the synthesized MOFs are summarized in Table 4. The data indicates gas adsorption capacity decreases at higher temperatures under constant pressure, confirming the physical nature of gas adsorption (Furukawa et al., 2010). Notably, N_2 exhibits the lowest adsorption capacity due to its lower condensability (Tc = 126.1 K) and polarizability (17.4 × 10^-25 cm^3), leading to weaker interactions with the MOF surfaces (Xiang et al., 2020). The Zn-NH_2 MOF structure showed a significant reduction in N_2 adsorption, resulting in a CO_2/N_2 selectivity at 303 K that is greater than that of NH_2-MIL-101(Al). This selectivity enhancement is attributed to the pore size of NH_2-MIL-101(Zn), which effectively sieves N_2 /CO_2. Moreover, Table 4 also highlights that CO_2's higher quadrupole moment and critical temperature (Tc = 304.05 K) contribute to its superior adsorption compared to CH_4 and N_2. The presence of amine groups in NH_2-MIL-101(Al) enhances CO_2 uptake through favourable Lewis acid-base interactions (Chong et al., 2021). The Zn-MOF achieved a CO_2 uptake of 3.0 mmol g^{-1} at 303 K, an increase of approximately 83% compared to NH_2-MIL-101(Zn), and about 120% higher than NH_2-MIL-101(Al). CH_4 uptake in the Al-MOF was measured at 3.0 mmol g^{-1}, reflecting a decrease from NH_2-MIL-101(Zn) (5.0 mmol g^{-1}) at elevated temperatures. The limited entry of CH_4 into NH_2-MIL-101(Al) is explained by its pore size (2.467 Å), which is smaller than CH_4's kinetic diameter (3.8 Å), suggesting a structural gate-opening phenomenon.

The pore structure significantly influences gas adsorption; larger pores may accommodate more gas but often result in weaker interactions per molecule, particularly for gases like CH_4 (Furukawa et al., 2013). pH variations can also affect the MOF's surface charge and gas affinity, although specific studies on MIL-101's pH effects are scarce (Baker & Low, 2015).

The CO_2/CH_4 selectivity for Zn-MOFs at 303 K was found to be 3.0, surpassing NH_2-MIL-101(Al) at 2.5 due to size exclusion effects that limit access for larger molecules to the inner core MOF, enhancing CO_2 selective behaviour (Banerjee et al., 2009). The Zn-MOF shell plays a crucial role in molecular sieving by excluding molecules larger than its aperture pore size (3.4 Å), thereby improving adsorption selectivity.

Table 4. CO_2/ CH_4 and CO_2/N_2 uptake of Al-ATA and Zn-ATA.

MOFs	Temperature (°C)	Pressure (bar)	CO_2 uptake capacity (mmolg^{-1})	CH_4 uptake capacity (mmolg^{-1})	N_2 uptake capacity (mmolg^{-1})
Al-ATA	30	1.0	2.5	0.3	0.5
	60	1.0	1.0	2.0	0.3
	100	1.0	0.5	3.0	0.2
Zn-ATA	30	1.0	3.0	0.5	1.2
	60	1.0	2.0	3.5	0.6
	100	1.0	1.0	0.5	0.4

4. Conclusion

In this study, we successfully synthesized and characterized two metal-organic frameworks (MOFs), Al-ATA and Al-ATA, utilizing a crystallographic approach. The analysis confirmed the high crystallinity and structural integrity of the synthesized MOFs. Notably, Zn-ATA demonstrated a significantly higher specific surface area and pore volume compared to its aluminum counterpart, indicating enhanced gas adsorption potential.

The activity tests, which involved exposing the MOF to different temperatures (30°C, 60°C, and 100°C) while maintaining a constant pressure of 1.0 bar, provided a comprehensive understanding of the material's gas adsorption capabilities. The experimental setup, utilizing a plug-free reactor, facilitated smooth gas flow, allowing for effective interaction between the MOF and the target gases. The results demonstrated that the adsorption performance of the MIL-101 series of MOFs, particularly those with different metal centers, varied significantly with temperature. Notably, Zn-ATA exhibited the highest CO_2 uptake capacities compared to their counterparts Al-ATA, highlighting the influence of the metal center on gas adsorption affinity. The data indicated a general trend of increased methane (CH_4) uptake with rising temperatures, while carbon dioxide (CO_2) uptake tended to decrease, consistent with previous literature.

Overall, this research contributes to the growing body of knowledge on MOFs and their applications in environmental remediation. The findings suggest that these materials could play a crucial role in mitigating harmful emissions and addressing pressing environmental challenges, paving the way for further exploration and development in this field. Future studies should focus on optimizing the synthesis process and exploring the functionalization of these MOFs to enhance their performance in specific applications.

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