Development of volumetric adsorption system for multicomponent gas capture: Utilizing bio-metal organic framework for CO_2 adsorption

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Abstract. A novel three-chamber volumetric multicomponent gas system is developed to monitor CO_2 adsorption for carbon capture systems. Using ultrasonic-synthesized Bio-MOF Co-Glu adsorbent, a water bath and coil heaters has been maintained isothermal conditions for CO_2 adsorption in an 85:15 CO_2/N2 at 27°C and pressures of 12 bars. Both measuring cells (Main Chamber-1 and Main Chamber-2) showed a maximum CO_2 adsorption capacity of 0.12 g/g. X-ray diffraction, Fourier-transform infrared spectroscopy, and Brunauer-Emmett-Teller confirmed, biological metal organic framework doped Cobalt Glu gas capture capabilities with its crystalline structure, functional groups, and wide surface area. Advanced volumetric methods for multicomponent gas adsorption estimate partial pressures and individual gas uptakes in mixtures based on a volumetric framework, with non-ideal gas behaviour, The results show that the biological metal organic framework doped Cobalt Glu adsorbent coupled with volumetric equipment may advance multicomponent gas adsorption research.

1 Introduction

High CO_2 levels in the atmosphere exacerbate global warming and climate change. According to the IEA, global CO_2 emissions reached 36.8 billion metric tons in 2022, mostly from the energy and industrial sectors [1]. To mitigate emissions, carbon capture, utilization, and storage (CCUS) technologies have gained popularity [2]. Adsorption-based CCUS techniques, notably those involving metal-organic frameworks (MOFs), show promise due to their exceptional selectivity, efficiency, and scalability in CO_2 collection [3]. However, MOFs high manufacturing costs and dependency on organic ligands derived from petrochemicals make them unsustainable and uneconomical for widespread implementation [4, 5]. This has spurred interest in bio-based MOFs (Bio-MOFs) that utilize bioligands as a more sustainable alternative. Bioligands derived from renewable, eco-friendly sources can potentially reduce reliance on petrochemical resources while enhancing MOF performance in CO_2 capture. Bio-MOFs thus offer a viable pathway to addressing both the sustainability and economic challenges associated with adsorption technologies. However, a significant research gap remains in understanding the performance of Bio-MOFs in realistic gas

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environments. Most studies on MOF-based CO_2 adsorption focus on pure CO_2 systems, which do not accurately represent industrial flue gases that contain multiple gas components such as nitrogen (N_2). CO_2 adsorption from multicomponent gas mixtures presents additional experimental challenges beyond material constraints. Traditional measurement techniques, including gravimetric and gas chromatography (GC) methods, are often expensive, sensitive to environmental fluctuations, and operationally complex [6, 7]. Gravimetric techniques also suffer from a limited dynamic range, making adsorption measurements across varying pressures and temperatures difficult. These limitations indicate the need for more practical, cost-effective, and reliable methods for studying multicomponent gas adsorption.

This study addresses these gaps by integrating bio-based metal–organic frameworks (Bio-MOFs) into a multicomponent gas adsorption system using a volumetric approach. An 85:15 CO_2/N_2 gas mixture was selected to simulate industrial conditions. The adsorption performance of an ultrasonically synthesized cobalt–glutamate Bio-MOF (Co-Glu) was evaluated under varying pressures up to 10 bar and temperatures from 27–60°C. Structural and textural properties of the adsorbent were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and Brunauer–Emmett–Teller (BET) surface area analysis. This integrated experimental framework provides a practical and cost-effective platform for studying multicomponent CO_2 adsorption using sustainable adsorbent materials.

2 Methodology

2.1 Materials

Sodium Hydroxide (99%), anhydrous ethanol (99.7%), L–glutamic acid ($HO_2CCH_2CH_2CH(NH_2)CO2H$, 99%), and Cobalt (II) chloride hexahydrate (98%) were acquired from Merck. Without purification, all chemicals were utilized. The deionized water was obtained from homemade laboratory. For the gas uptakes measurement, high purity of carbon dioxide (CO_2) and nitrogen (N_2) gases with purity >99.99% were used for adsorption studies.

2.2 Bio-MOF synthesis

In this research, sample preparation was conducted by adopting the principles of green synthesis using ultrasonic techniques, which can shorten reaction times and minimize byproducts [1, 2]. The synthesis procedure referred to our previous study, which utilized solvothermal techniques [3]. The solvent selection and pH adjustment procedures were modified to employ greener solvents. The Bio-MOF was synthesized through systematic solution preparation, pH control, ultrasonic treatment, and purification. First, 1.4 g of L-glutamic acid was dissolved in 60 mL of distilled water and stirred at room temperature for 20 min. Subsequently, 2.37 g of cobalt chloride hexahydrate was added, and the mixture was stirred for an additional 5 min. The solution pH was then adjusted to 7.5 using a sodium hydroxide (NaOH) solution and monitored until a neutral condition was achieved. After pH adjustment, the mixture was further stirred to ensure homogeneity. The resulting solution was subjected to ultrasonic treatment for 1 h to promote Bio-MOF formation. The product was then stirred in ethanol at 80 °C for 15 min and purified through repeated washing. Finally, the purified Bio-MOF was dried overnight at 80 °C.

The development of Cobalt–glutamate (Bio MOF Co-Glu) was selected due to its sustainability, coordination stability, and relevance to CO_2 adsorption applications. L-glutamic acid is a bio-derived ligand containing both amine and carboxyl functional groups, which facilitate strong coordination with transition metals and provide polar sites favorable

for CO_2 adsorption. Cobalt ions exhibit stable coordination with amino-carboxylate ligands, enabling the formation of a robust framework while maintaining accessible adsorption sites. Compared to conventional MOFs based on petrochemical ligands, This Bio-MOF offers a more sustainable alternative suitable for multicomponent gas adsorption studies.

2.3 Bio-MOF characterization

The synthesized Bio-MOF Co-Glu was further evaluated using the solvothermal method to guarantee its gas capture efficacy and compare its characteristics to previously synthesized samples. In this investigation, only chosen characterisation approaches were used to assess adsorbent feasibility. FTIR spectroscopy was used to study the chemical properties of ultrasonic-assisted Bio-MOF Co-Glu using the KBr technique on a Thermo Scientific Nicolet 6700, encompassing a wavelength range of 500-4000 cm^{-1}. To analyze the material's crystalline structure, a Shimadzu XRD-600 diffractometer was used at 40 kV and 40 mA. The Brunauer-Emmett-Teller (BET) technique measured Bio-MOF Co-Glu surface area and pore volume. Full BET isotherm analysis was performed using N_2 as the adsorbate on a Quantachrome Nova 1200E at 77K. To eliminate moisture and volatile contaminants, the adsorbent was degassed at 120°C for 8 hours before analysis.

2.4 Multicomponent adsorption design and measurement

The study investigated the adsorption of CO_2/N_2 multicomponent gas separation using a volumetric approach as presented in Figure 1. The device consists of four chambers: an initial chamber serving as a gas-mixing agitator, a second chamber functioning as a reference cell, and two subsequent chambers containing the Bio-MOF adsorbent for sequential gas adsorption. Conventional multicomponent gas adsorption measurements typically rely on gravimetric techniques combined with gas chromatography to resolve individual gas uptakes. Although highly accurate, these methods are often expensive, sensitive to environmental disturbances, and operationally complex. In contrast, the proposed multi-chamber volumetric system enables sequential adsorption and estimation of individual gas uptakes using pressure–temperature measurements, offering a simpler and more cost-effective approach for laboratory-scale multicomponent adsorption studies.

The chambers were connected by valves and monitored using Class A Type K thermocouples and 0-40 bar pressure transmitters. The National Instruments Data Acquisition Tool was used to acquire data. The chambers were made from 304 stainless steel and kept in an isothermal water bath. CO_2/N_2 adsorption was tested at 27°C to 60°C to represent operating conditions relevant to industrial gas separation and carbon capture applications. These conditions are typical of post-combustion environments, where adsorption processes are often operated under near-ambient to moderately elevated temperatures and pressures to balance separation and energy consumption [4, 5]. Then, helium was chosen because its tiny molecular size allowed it to enter the adsorbent's tight pores [6, 7]. The system was vacuumed after helium purging to remove impurities and calculate effective adsorption volume using adsorption cell void volume. The system was then evacuated to eliminate remaining helium gas before gas adsorption experiments. A water bath-maintained chamber temperature was maintained for stability. CO_2 and N_2 gasses were added to the agitator chamber, and the gas mixture was mixed uniformly to imitate reality. The mixture was put into the reference cell to reach equilibrium, and pressure and temperature changes were monitored after homogeneity. The operation was repeated at three isothermal temperatures and 12 bar, enabling exact measurement and comprehensive adsorption investigation in various conditions. The ideal gas law was used for gas mixture

adsorption, and each gas component could be calculated using the ideal gas equation. The equilibrium mixture pressure was estimated by mixing gas partial pressures using Dalton's Law described in equation (1) [8, 9]. Volumetric adsorption calculations were performed using an ideal-gas-based framework as a reference. Deviations from ideal gas behavior were explicitly accounted for through compressibility factors (Z) calculated from experimentally measured pressure and temperature using the NIST REFPROP database, which is based on real-gas equations of state [10]. This approach enabled quantitative evaluation of non-ideal gas effects under the investigated pressure and temperature,

$$P_{total} = \sum_{i=1}^{n} Pi \quad (1)$$

When the total number of moles in the chamber (n_{total}) is known, the number of moles of a specific gas (e.g., CO_2 or N_2) in the chamber (n_{gas}) can be calculated using equation (2)

$$n_{gas,chamber} = \frac{n_{total,chamber} \times Cgas \times R \times T \times P_{gas,chamber} \times V_{chamber}}{R \times T \times P_{total} \times V_{total}} \quad (2)$$

Where C_{gas} represents the gas concentration (mol/m³), R is the ideal gas constant (8.314 J/mol.K), T and P denote the temperature (K) and pressure (bar), respectively, and V represents the volume (m³). Once the number of moles of gas is determined, the mass of carbon dioxide or nitrogen absorbed in the charging cell can be calculated using equation (3).

$$m_{total\ gas,chamber} = n_{gas,chamber} \times Mr_{gas} \quad (3)$$

The adsorption capacity of each gas ($q_{gas\ chamber}$) was then calculated using equation (4):

$$q_{gas\ chamber} = \frac{m_{total\ gas,chamber}}{m_{adsorbent}} \quad (4)$$

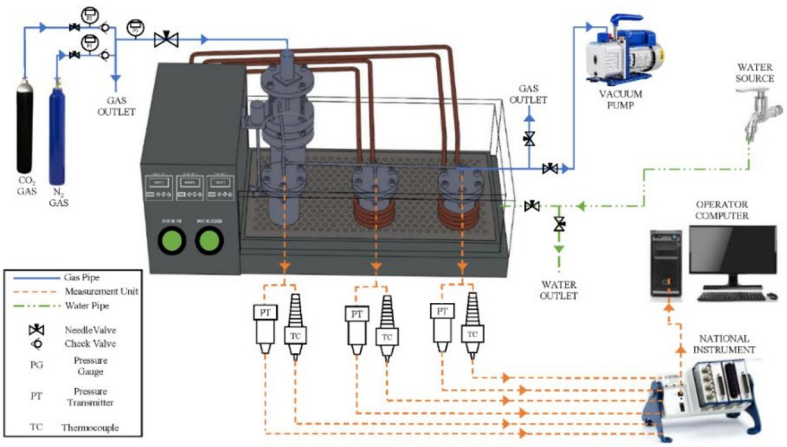

Fig. 1 . Schematic diagram of volumetric multicomponent gas adsorption.

3 Result and Discussion

3.1 Chemical functionalities of bio-MOF co-glu

The functional groups in Bio-MOF are verified by FTIR as shown in Figure 2. We evaluated synthesis efficacy by comparing FTIR data to our previous solvothermal investigations [10]. Solvothermal and ultrasonic-assisted synthesis peaks matched. Ultrasonic-assisted Bio-MOF Co-Glu synthesis peaks and atomic interactions are listed in Table 1. FTIR study of key functional groups verified bio-MOF Co-Glu production. At 3333.14 cm^{-1}, medium-intensity N-H stretching vibrations were found for Aliphatic Primary Amine groups. This indicates L-glutamic acid amino group coordination. Carboxylic Acid groups exhibited a large O-H stretching vibration at 3254.16 cm^{-1}, suggesting loosely coupled hydroxyl functionalities in MOF structure. Protonated amine groups from cobalt ion interactions may be the cause of strong N-H stretching vibrations from Amine Salt (NH^+) at 2920.49 cm^{-1}. The presence of NH_3 amine groups, essential for metal ion coordination and structural stability, was verified by medium-intensity N-H bending vibrations at 1606.10 cm^{-1}. N-O stretching vibrations at 1544.59 cm^{-1} are seen in nitro compounds or nitroalkanes, perhaps from byproducts or functionalized precursors during production. Carboxylic Acid groups caused medium-intensity O-H bending vibrations at 1421.11 cm^{-1}, showing L-glutamic acid's carboxyl functionality was preserved. The MOF framework showed medium-intensity O-H bending vibrations at 1360.17 cm^{-1}, perhaps from Alcohol groups, which might be residual solvents or functionalized linkers. Significant C-O stretching vibrations at 1063.19 cm^{-1} indicate the existence of Primary Alcohol groups in the Bio-MOF structure, suggesting functionalized organic components.

Table 1. FTIR frequency of bio-mof co-glu.

Reference Wavenumber (cm^{-1})	Bio-MOF Co-Glu (cm^{-1})	Intensity	Vibration Type
3400 - 3300	3333.14	Medium	N-H *Stretching* (*Aliphatic Primary Amine*)
3300 - 2500	3254.16	Strong	O-H *Stretching* (*Carboxylic Acid*)
3000-2800	2920.49	Strong	N-H *Stretching* (*Amine Salt*)
1650 - 1580	1606.10	Medium	N-H *Bending* (*Amine*)
1550 - 1500	1544.59	Strong	N-O *Stretching* (*Nitro Compound*)
1440 - 1395	1421.11	Medium	O-H *Bending* (*Carboxylic Acid*)
1420 - 1330	1360.17	Medium	O-H *Bending* (*Alcohol*)
1085 - 1050	1063.19	Strong	C-O *Stretching* (*Primary Alcohol*)
880 - 1000	877.04	Medium	C-H bending

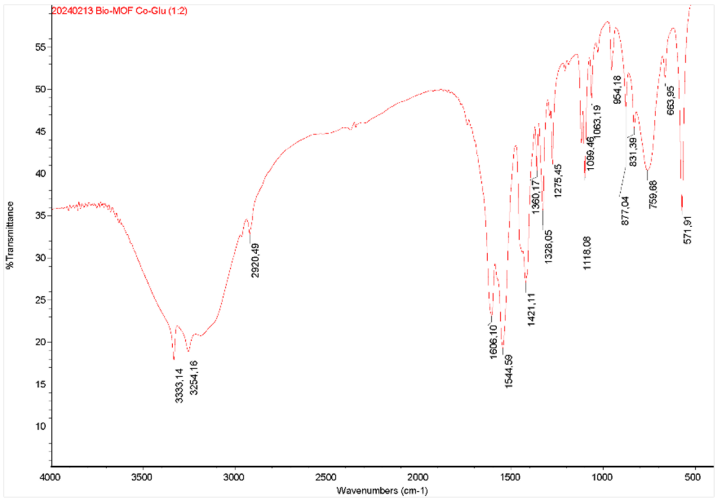

Fig. 2 FTIR bio-mof co-glu.

3.2 XRD analysis

The structure and diffraction patterns of Bio-MOF Co-Glu were verified through XRD characterization. Low-angle peaks are observed in the XRD data at 2θ = 16.88° and 21.96°, as illustrated in Figure 3. The integration of the organic linker into the Bio-MOF structure was confirmed by the presence of L-glutamic acid ligand in the produced sample [3]. A prior research found that the peak at 2θ=38.76° indicates cobalt metal interaction with the bio-MOF framework [11]. Peak changes, as 2θ = 14.96° to 15.61° and 16.85° to 17.77°, suggest pores open, even in Glutamic-containing phases [12]. The peak at 2θ=29.52° indicates a high-quality crystalline structure of cobalt (II) chloride hexahydrate, supporting recent findings [13]. The degree of crystallinity of the synthesized Bio-MOF Co-Glu was further quantified from the XRD pattern using peak deconvolution analysis performed in Origin software. The crystallinity percentage was determined by calculating the ratio of the integrated area of crystalline diffraction peaks to the total diffracted area, yielding a crystallinity of 83.53%. This relatively high crystallinity confirms the successful formation of an ordered Bio-MOF structure, despite the use of ultrasonic-assisted synthesis, which is often associated with broader diffraction peaks due to rapid nucleation. The obtained crystallinity is sufficient to support stable framework formation and effective gas adsorption performance.

Increasing peaks in the XRD graph indicate poor crystallinity in this adsorbent. More peaks in Bio-MOF crystals indicate impurities and alter physical and chemical properties. This contradicts solvothermal XRD. Ultrasonic synthesis raised XRD peaks. Solvothermal synthesis yields sharper XRD peaks with fewer impurities due to controlled reaction conditions and slower kinetics. High pressure and temperature in a sealed autoclave improve crystallinity and reduce defects by causing reactant contact and gradual crystal formation [14]. Ultrasonic-assisted synthesis employs sound waves to induce rapid, high-energy reactions that cause quicker kinetics and cavitation, resulting in less ordered crystal structures and more impurities. To remove pollutants, purification may be used. Despite differences in synthesis methods, significant peaks in the XRD pattern show that Bio-MOF had a well-defined structure and excellent gas adsorption porosity.

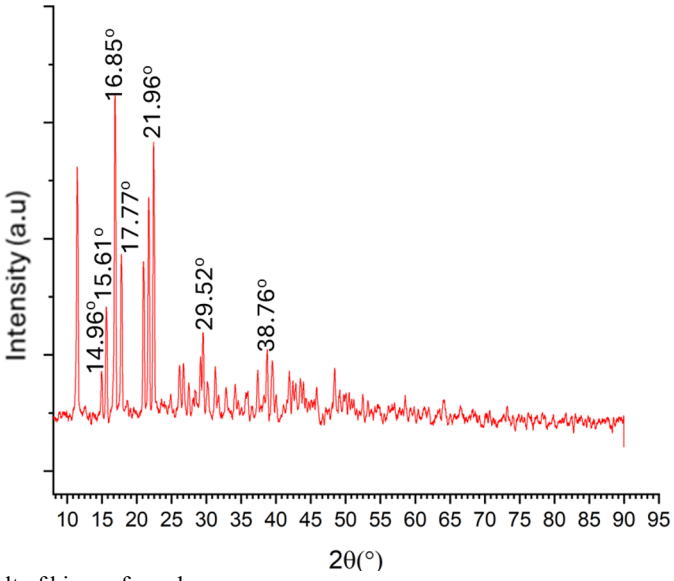

Fig. 3 XRD result of bio-mof co-glu.

3.3 BET analysis

The BET technique was used to evaluate the surface area and pore volume of bio-MOF Co-Glu, which was degassed at 120°C before measurement. The N_2 adsorption/desorption isotherm for BET analysis is presented in Figure 4. The study revealed a specific surface area of 4.32 m^2/g, pore volume of 0.047 cm^3/g, and an average pore size of 219 Å. Compared to other Bio-MOFs, bio-MOF Co-Glu has a reduced surface area and pore volume [15, 16]. Porosity is lower than our solvothermal Bio-MOF. The ultrasonic technique may restrict pores. Cavitation bubbles also inhibit crystalline lattice formation. However, gas adsorption effectiveness is affected by several parameters beyond surface area and pore volume. Gas adsorption effectiveness is also affected by pore size distribution, functional groups, and material stability.

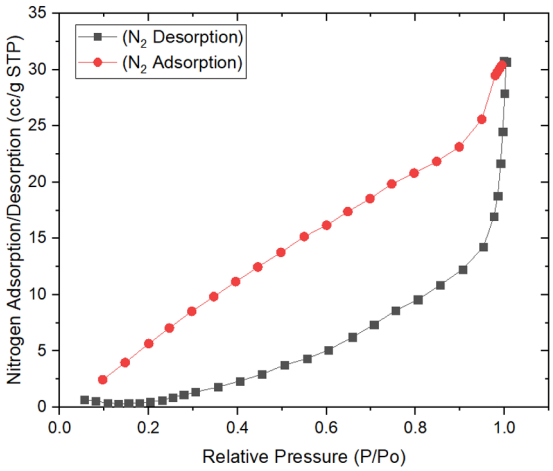

Fig. 4 N_2 adsorption/desorption isotherm on bio-mof co-glu.

3.4 Gas adsorption study

The adsorption data for bio-MOF Co-Glu samples was obtained through volumetric adsorption testing of CO_2/N_2 gas at various temperatures as illustrated in Figure 5. The test showed a partial mass of 0.12 grams of CO_2 at 12 bar pressure, with the strongest adsorption at high pressure. The lowest temperature (27°C) had the highest adsorption, indicating physical adsorption. To optimize gas adsorption, the second measurement cell (MC-2) received residual gas from the reference cell. The adsorption of CO_2 in MC-2 was comparable, with a maximum of 0.12 g/g at 27°C and 10 bar pressures. The method employs two measurement cells to fully utilize the residual gas from the reference cell, effectively separating CO_2 from gas mixtures and achieving high purity. This design is essential for high-purity gas separation or storage applications, optimizing the use of adsorbent material and gas mixtures. The comparison with other types of solid adsorbent on CO_2 adsorption are described in **Table 2**. Further, if it is compared with our previous research in single gas CO_2 with the same type of Bio-MOF, the performance of CO_2 adsorbed has decreased from 0.5 g/g to 0.12 g/g [17].

Table 2 CO_2 Adsorption Capacity with Other MOFs

Adsorbents	CO_2 Adsorption Capacity (g/g)	Reference
ZIFs with spinel magnetic	0.13	[18]
Bio-based MIP-202	0.0367	[19]
MOF-GO	0.13	[20]
MIL-101 Cr	0.3	[21]
CALF-20-SOL	0.14	[22]
Bio-MOF Co-Glu in Gas Mixture	0.12	This Work

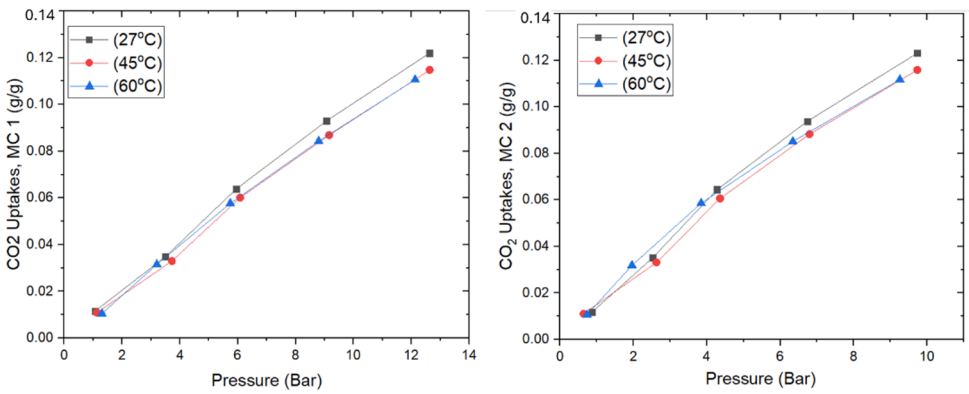

Fig. 5 Gas adsorption isotherm (a)MC-1 (b) MC-2.

4 Conclusion

With three chambers, a gas mixture agitator, a reference cell, and measurement cells—the volumetric adsorption testing system works well. A coil-equipped water bath maintains isothermal conditions in these chambers during adsorption. The system effectively measured

CO_2 adsorption capacity at various temperatures and pressures, recording a maximum of 0.12 g/g for both MC-1 and MC-2 at 27°C and 12 and 10 bar, respectively. Dual-adsorption chamber gas adsorption evaluations are more effective. The Bio-MOF-derived adsorbent was produced ultrasonically, continuing solvothermal synthesis research. The combination of this novel adsorbent with the bespoke system offers a solid foundation for multicomponent gas adsorption studies and sustainable CO_2 capture systems.

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