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# Performance Evaluation of the Developed MOF-177 for $CO_2$ Capture

Aqeel Ahmad[1,2], Bryan Pango[1], Marhaina Ismail[1,2], Mohamad Azmi Bustam[1,2,*]

[1] Centre of Research in Ionic Liquids (CORIL), Universiti Teknologi PETRONAS, Perak 32610, Malaysia
[2] Chemical Engineering Department, Universiti Teknologi PETRONAS, Perak 32610, Malaysia

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**ABSTRACT**

The emission of $CO_2$ into the atmosphere has emerged as a predominant concern in recent times. Metal organic frameworks (MOFs) are excellent materials that are well suited to capture $CO_2$ due to their large surface area and pore volume. Therefore, this research involved synthesis of MOF-177 through the solvothermal method to produce both powder and pellets. The pelletizing procedure, which included the use of 5 wt % polylactic acid (PLA), was carried out using the extrusion method. To compare the performance of the synthesized MOF-177 powder and pellets, XRD, SEM, FTIR and $CO_2$ adsorption isotherm measurements were conducted. The synthesis confirmation of powdered and pelletized MOF-177 was performed by XRD. The SEM images demonstrated that the particle size of the synthesized MOFs was between 20-25 µm, indicating that PLA was an effective binder for MOF-177. The FTIR results showed that the MOF-177 bonds and functional group present in both samples with the addition of ester groups in pellet samples due to the presence of PLA. It was also noted that by addition of PLA, the structure of MOF-177 was unaffected. Lastly, the $CO_2$ uptake obtained from the $CO_2$ isotherm is 0.27 mmol/g for powders and 0.47 mmol/g for the pellets. Hence, the proposed method to synthesize MOF-177 powder and pellet can be effectively used to capture $CO_2$.

## 1. Introduction

Greenhouse gases, such as carbon dioxide ($CO_2$), are of international significance because they contribute to global warming by trapping heat produced at the Surface of the earth. The majority of $CO_2$ emissions come from the burning of fossil fuels for industrial purposes, such as the generation of electricity and the operation of vehicles running on fossil fuels. The Energy Information Administration (EIA) forecasts a 57% increase in energy usage by 2030. The Intergovernmental Panel on Climate Change (IPCC) has acknowledged global warming and weather change as urgent problems, and they have found that $CO_2$ is a major contributor [1]. During the industrial revolution, the atmospheric $CO_2$ concentration has grown from 270-400 ppm [2], and its influence on the environment has sparked a growing interest in developing innovative materials and technologies suitable for effective carbon capture and storage [3].

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[*] *Corresponding author.*
*E-mail address: azmibustam@utp.edu.my*

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Cryogenic distillation, membrane separation, absorption and absorption are some of the current methods for $CO_2$ capture [4-6]. Membrane filtration is a simple process since $CO_2$ molecules are smaller and more soluble, allowing them to flow through a membrane more quickly than $N_2$ and other pollutants. Nevertheless, creating a large-scale membrane without any flaws is difficult, leading to a significant loss of selectivity [7]. Cryogenic separation techniques permit the liquid-state concentration of $CO_2$. However, this technique requires a considerable amount of energy to maintain temperatures below -56 °C. Additionally, it is essential to eliminate other components of the flue gas prior to chilling. Water in the stream can cause the pipes to freeze, clog, and corrode in addition to freezing and clogging the machinery [8]. Amine solutions such as Triethanolamine (TEA), Diethanolamine (DEA) and Monoethanolamine (MEA), are often employed in chemical absorption processes. Nevertheless, these procedures still have issues with solvent loss, and a high energy need for solvent recovery [9]. In contrast, pressure swing adsorption (PSA) operations can make use of adsorption techniques without encountering comparable difficulties because of the greater binding of $CO_2$ molecules to solid adsorbents compared to $N_2$ molecules [10].

Porous coordination polymers [11], carbon-based materials [12], zeolites [13], and mesoporous silica [14] are only a few examples of the several porous adsorbents that have been investigated for $CO_2$ adsorption. Nevertheless, traditional porous materials have limits in their stability, $CO_2/N_2$ selectivity, and $CO_2$ uptake capability [15-17]. Metal-organic frameworks (MOFs) have attracted considerable interest since they are composed of metal ions and organic linkers. These materials have several advantageous properties, including a higher specific surface area, a large crystal size, and the capability to modify the framework and surface characteristics precisely [18, 19]. MOF-177 was chosen among the various MOFs due to its extraordinary characteristics, which include a high surface area ranging from 2000 to 4500 $m^2/g$, exceptional thermal stability up to 380 °C, the presence of exposed metal sites, and a large pore volume ranging from 0.87 to 1.11 $cm^3/g$ [20-22]. These characteristics make MOF-177 a highly desirable material for numerous applications. Ullah *et al.,* [23] developed and tested MOF-177 for $CO_2$ adsorption behaviour. They synthesized MOF-177 using the solvothermal process, and XRD was employed to confirm it. The results of the adsorption test demonstrated that MOF-177 can successfully separate $CO_2$. However, low adsorption performance, difficulties in handling and transport are the challenges that need to be addressed [24].

In this work, we proposed the synthesis of MOF-177 powder and pellets to capture $CO_2$ using solvothermal process. The MOF-177 palletization was performed using a binder, polylactic acid (PLA) [25] using extrusion method. The comparative performance of MOF-177 powder and pellet samples was performed using XRD, SEM and FTIR. The BET (Brunauer-Emmett-Teller) technique was used to examine the $CO_2$ uptake capabilities of MOF-177 powder and pellet forms.

## 2. Methodology

### *2.1 Materials*

All of the chemicals employed in this study, including 4,4′,4′′-benzene-1,3,5-triyltribenzoate (BTB), N,N-dimethyl formamide (DMF), and zinc nitrate hexahydrate ($Zn\text{-}(NO_3)_2.6H_2O$), were acquired from Sigma-Aldrich and were of high purity (>95%). All the chemicals were laboratory scale chemicals and used without any further treatment.

### *2.2 Synthesis of MOF-177*

A solution of zinc nitrate hexahydrate (2.51 g, 11.4 mmol) and 4,4′,4″-benzene-1,3,5-benzenetribenzoate (H3BTB, 0.626 g, 1.43 mmol) was prepared with 50 ml of DMF and stirred for 3h on stirrer plate. After stirring, the sample solutions were transferred into Teflon autoclave and let sit in the oven for 26 h at 100 °C. Then, the autoclave was taken out of oven and cooled down in the fume hood for 1 h to room temperature. Following synthesis, the product was centrifuged before being washed with 10 mL of methanol. The sample was then submerged in 40 mL of $CHCl_3$ (HPLC grade). Over the course of 5 days, the solvent was replenished three times: after one day, four days, and five days. After this, the samples were desiccated in a 120 °C oven for 12 h. After drying, the samples were closed and stored.

### *2.3 Pretreatment and Palletization of MOF-177*

For pre-treatment, the sample was put into two sample cells and was inserted into two ports. Then the sample cells were put into a pretreatment system. The pretreatment system was switched on along with the vacuum pump. Then the temperature was set to 180 °C and left for 24 h in the system to remove moisture. After 24 h, the sample cells were purged with nitrogen ($N_2$) to remove the impurities and was let cool for 1 h. Finally, the samples were transferred into vials and sealed with parafilm. For shaping, a mixture of 1g of samples and 1.5g chloroform with ratio of 1:1.5 was prepared. Then, the mixtures underwent sonication for 30 min at 50 °C. After that, 5 wt.% of polylactic acid (PLA) was added into the solution after weighing it. Then, the sonication process was repeated for 30 min at 50 °C. Next, the solution was poured into a syringe. A petri dish was prepared beforehand with methanol for the extrusion process. Then, the solution was extruded from the syringe into petri dish. The extruded samples were cut into pellet form. The sample was then transferred into a centrifuge tube filled with methanol. The pellets were refreshed with methanol and stored in the refrigerator for 3 days. Each day the methanol was refreshed 2 times every day.

### *2.4 Characterization*

#### *2.4.1 MOF-177 synthesis confirmation by PXRD*

Powder X-ray diffraction was used to evaluate the crystalline structure of the produced samples (PXRD). Using a Bruker AXS-D8 Advance Diffractometer in Billerica, Massachusetts, the patterns were captured with Cu Kα radiation ($\lambda$ = 1.5406 Å), a step size of 0.02° in 2 ranging from 5° to 45°, and a scan rate of 1°/min.

#### *2.4.2 Surface properties of MOF-177 by SEM*

The morphology of synthesized MOF-177 was performed using scanning electron microscopy (SEM, Zeiss SUPRA 55VP). The sample was coated with gold using Emitech K550X sputter coating. Afterward, the coated sample was placed in a clamping device sample holder and set the gap of 4 mm.

#### *2.4.3 FTIR investigation*

FTIR analysis was conducted using a Perkin Elmer Inc instrument from the USA to identify the functional groups and chemical bonds present in the samples. The transmittance of radiation by each molecule was recorded by exposing the sample to be tested in all infrared frequencies in the range of 400-4000 $cm^{-1}$. The samples were inserted in the sample cell when the transmittance mode was in operation, and the spectrum was created by combining 16 scans with a resolution of 4 $cm^{-1}$.

### *2.4.4 Adsorption isotherm measurements*

The adsorption and desorption performance of synthesized MOF-177 powder and MOF-177 pellet was examined. The technique adopted for this experiment was nitrogen adsorption-desorption using Micromeritics ASAP 2020. The samples were first placed in a tube in the pretreatment system to remove moisture at 180°C for 24 h before taking measurements.

## 3. Results

### *3.1 MOF-177 synthesis confirmation by PXRD*

For the synthesized MOF-177, PXRD (powder X-ray diffraction) was used to confirm the product's formation and diffraction patterns. The PXRD pattern findings exhibited remarkable similarity and agreement with previously published MOF-177 [23]. The peaks at 6.2°, 10.1°, 14.95°, 18° and 20.75° were well identified, as shown in Figure 1. Similar diffraction patterns were observed in MOF/PLA pellet, indicating that the incorporation of PLA had not affected the crystal forms of MOF. However, by including PLA into MOF-177 considerably decreased the peak size. This might be ascribed to strong interactions between MOF-177 and PLA which restricted the movability of MOF chain. These findings are in corroboration with the previous findings [26].

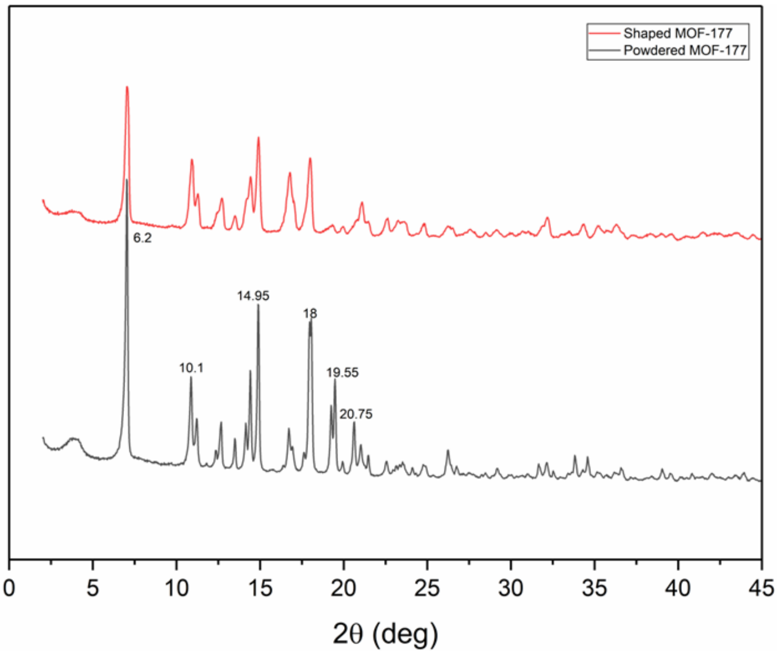

**Fig. 1.** XRD of synthesized powdered and pelletized MOF-177

### *3.2 Surface Properties of MOF-177 by SEM*

Figure 2 illustrates that of MOF-177 powder samples that were prepared during the experiments. The SEM profiles obtained at 100X and 3KX magnifications were used to assess the size range of the particles. When observed, as shown in Figure 2 (a) at the magnification of 100X, the smaller particles at the back seemed to be the particles of MOF-177 which were comparable as reported by Burgaz *et al.,* 2019 [27]. which were around 20-25 µm. Meanwhile, at magnification of 3000x it can be observed that the clumped particles have pores seen as depicted in Figure 2 (b). In Figure 2 (c), PLA binder used during the shaping of MOF-177 has bonded well with MOF-177 particles due to their existing organic frames [26]. In Figure 2 (d) at 3KX magnification, it was seen that the pores were also observed. The bonded MOF-177 showed that the extrusion method has successfully bind MOF-177 powders with polylactic acid as the binder. To support these findings, Ullah *et al.,* 2020 [20] revealed a similar finding for the synthesized MOF. They demonstrated that the produced MOF had the size range of (1 um- 200 nm).

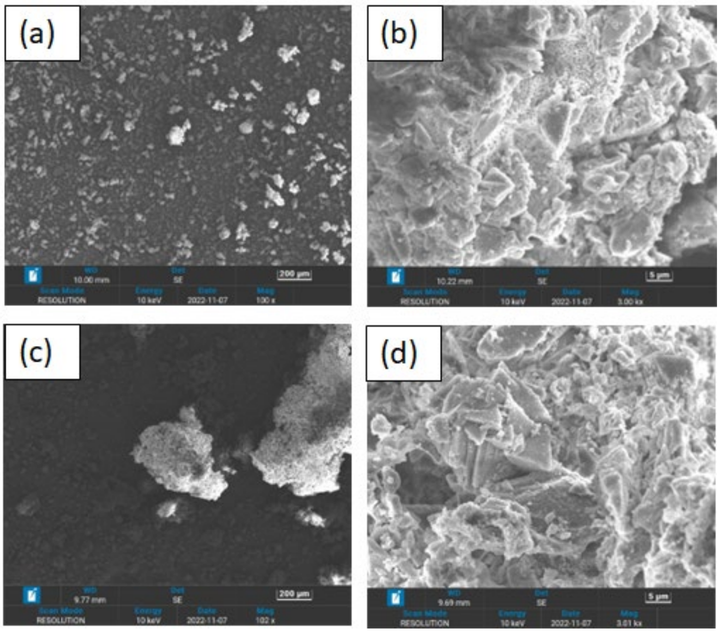

**Fig. 2.** SEM images of synthesized MOF-177 (a) powder at 100X (b) powder at 3KX (c) pellet at 100X (d) pellet 3KX

### *3.3 Confirmation of Binder to MOF by FTIR*

Figure 3 shows the FT-IR spectra of MOF-177 and MOF-177 that has been shaped. The vibrational stretching frequencies of the framework (O-C-O) were responsible for the strong bands at 1591 and 1400 $cm^{-1}$, which were evidence that the MOF-177 framework contained dicarboxylate linker. The vibrations of benzene rings appeared at 855, 781, and 707 $cm^{-1}$ [28]. The bands around 1661 and 1349 $cm^{-1}$ most likely corresponded to the in-plane and out-of-plane bending modes of single-bond COOH groups. The peak at 1758 $cm^{-1}$ matching C=O stretching for PLA [26]. These results are in accordance with the previously published findings [29].

Figure 4 shows adsorption and desorption isotherm of $CO_2$ at 25 °C and 1 bar for MOF-177 powder and MOF-177 pellet. The isotherm trend showed that it was a Langmuir model isotherm therefore the adsorption process was reversible. In Figure 4 (a), from lower pressure region, it was observed that the $CO_2$ uptake was high and reached and the adsorption rate decreased as relative pressure reached $p/p_o = 1$. MOF-177 powder adsorbed up to 0.27 mmol/g of adsorbent. Figure 4 (b) showed the adsorption and desorption isotherm of $CO_2$ at 25 °C and 1 bar for MOF-177 pellets. It also showed the same Langmuir model isotherm as MOF-177 powder samples. But, in pellet forms, the high adsorption capacity was 0.47 mmol/g which was higher than the powders. It might be due to more purity of MOF-177 as PLA bonded the MOF-177 particles together which provided more active sites to capture $CO_2$. In addition, powdered MOF is undesirable for industrial uses due to the fact it can migrate and clog the device under continuous fluid flow in the system [30, 31].

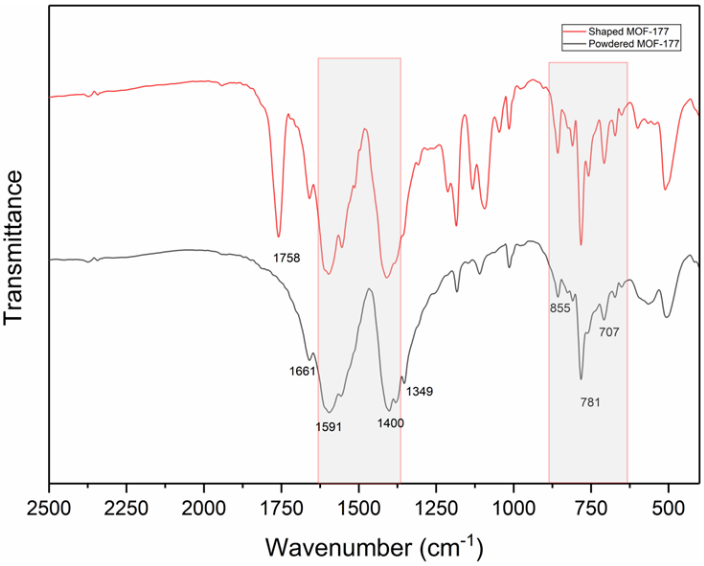

**Fig. 3.** FT-IR spectrum of MOF-177 (a) powder (b) shaped

### *3.4 $CO_2$ Adsorption Isotherm*

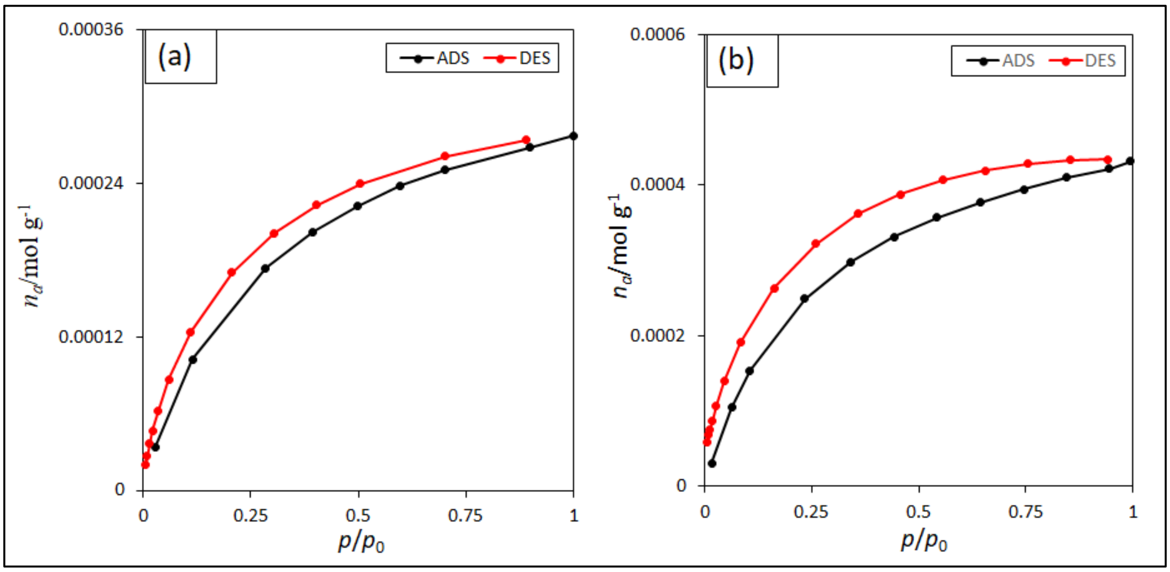

**Fig. 4.** $CO_2$ Adsorption Isotherm at 25 °C and pressure of 1 bar for MOF-177 (a) powder (b) pellet

## 4. Conclusions

MOF-177 powders and pellets were synthesized successfully with solvothermal method in the autoclave. The extrusion method of pelletizing was achieved by adding 5 wt % polylactic acid as the binder. Then, both samples in powder and pellet forms were characterized using XRD, SEM, FTIR, and application of $CO_2$ adsorption isotherm to see the comparative performance. The XRD results have successfully confirmed the formation of MOF-177 powder and pellets. The SEM images of the synthesized MOF-177 showed the porous structure after the PLA incorporation. The addition of PLA to MOF-177 was confirmed by FTIR, in which PLA peak was seen at 1758 $cm^{-1}$. The $CO_2$ adsorption isotherm revealed that the maximum adsorption capacity of MOF-177 powder was 0.27 mmol/g and MOF-177 pellet is 0.47 mmol/g. Thus, further investigations are needed to boost the adsorption capability and develop MOFs with increased stability by using inorganic filler for $CO_2$ adsorption.

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