

## Carbon Cryogel Preparation via Urea-furfural Gel Synthesis as an Adsorbent for CO<sub>2</sub> Capture Application

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Adsorption of CO<sub>2</sub> using porous solid materials is one of the suitable separation techniques due to its low cost and simple operation. Porous carbon from urea-furfural has been prepared as a potential adsorbent for CO<sub>2</sub> capture. In this work, urea-furfural carbon cryogel (CCUF) was prepared using a simple polymerisation reaction under ambient conditions with different water and acid loadings followed by a drying and calcination process. The effect of water and acid loadings on carbon synthesis were evaluated for carbon gel yield and CO<sub>2</sub> adsorption performance in a fixed-bed adsorption study. The selected CCUF was characterised with N<sub>2</sub> sorption isotherms, CO<sub>2</sub> temperature-programmed desorption (CO<sub>2</sub>-TPD), Fourier transform infrared spectroscopy (FTIR), and focused ion beam-scanning electron microscopy coupled with energy dispersive X-ray (FIBSEM-EDX) to understand the carbon surface structure and properties. The CCUF synthesised with 4 mL of water and 0.15 mL of acid loading was selected due to its high CO<sub>2</sub> adsorption capacity (2.04 mmol/g) compared to other carbon gels synthesised in this work. The large specific surface area (160 m<sup>2</sup>/g) of CCUF with high microporosity and nitrogen content provide capabilities for CO<sub>2</sub> adsorption under ambient conditions. The result shows that the N-enriched CCUF adsorbent has a potential for CO<sub>2</sub> adsorption.

### 1. Introduction

The global emission of greenhouse gases (GHG) such as CO<sub>2</sub> mainly results from the combustion of various industrial activities and transportation has drawn great attention as it has become one of the main factors of the global warming issue. Fossil fuel was estimated to contribute to 70 % of the global energy demand (Covert et al., 2016). Its utilisation has become an integral part of human activities where the combustion of fuel contributes to the emission of CO<sub>2</sub> (Rahmanian et al., 2018). For environmental protection and sustainable development, the researchers have started to find solutions to the emission of CO<sub>2</sub>, including combining CO<sub>2</sub> capture with the utilisation of GHG as a renewable energy source to minimise the hazards related to climate change. This is an attractive approach for reducing greenhouse gas capacity with the simultaneous transformation of CO<sub>2</sub> into various value-added chemicals (Ma et al., 2020).

Focusing on CO<sub>2</sub> capture, the adsorption via porous solids is among the available technologies and has been regarded as a promising technology due to its low cost and easy operation. The solid adsorbent can be used to replace the prevalent chemical absorption technology. Several porous solids have been investigated for CO<sub>2</sub> adsorption that can be categorised as non-carbonaceous (such as metal-organic framework (MOF) and porous polymers, functionalised silica, alkali-metal-based materials, zeolites, and metal oxide carbonates) and carbonaceous material (such as activated carbon, ordered porous carbon, carbon fibre, and graphene) (Lee and Park, 2015). The issues on corrosion in chemical absorption and the low CO<sub>2</sub> uptake have prompted the application of porous adsorbents for physical adsorption to obtain a high CO<sub>2</sub> adsorption capacity. MOF provides a high adsorption capacity compared to other adsorbents but involves a high synthesis cost. The carbonaceous adsorbents have shown their ability for high CO<sub>2</sub> adsorption due to their good surface texture and chemistry and low moisture content. The surface chemistry of the adsorbent can strongly influence CO<sub>2</sub> adsorption, while the

presence of water inhibits adsorption (Shafeeyan et al., 2010). The activated carbon from agricultural waste has become a competitive choice due to its low cost and high CO<sub>2</sub> adsorption capacity.

Carbon gel is another alternative carbonaceous adsorbent for CO<sub>2</sub> adsorption. Precursors such as urea and melamine can react with different aldehydes such as formaldehyde and furfural to form carbon gel. The health and environmental problems caused by formaldehyde have driven the substitution with furfural as a low cost, abundant in nature, and green aldehyde. Urea or melamine is used to provide the nitrogen functionalities that act as the Lewis base molecules that facilitate the adsorption process due to heteroatoms of the nitrogen functional group incorporated in the carbon matrix (Li et al., 2019). This is an advantage of porous carbon gel in providing a carbon matrix with a nitrogen-containing (N-enriched) precursor for high CO<sub>2</sub> adsorption. Previously, the modification of activated carbon functionalised with proper basic nitrogen was introduced to enhance the CO<sub>2</sub> adsorption performance (Shafeeyan et al., 2010). Liu et al. (2015) have used urea furfural resin in preparing microporous activated carbon for CO<sub>2</sub> adsorption with a 4.6 mmol/g CO<sub>2</sub> uptake.

As for this work, the synthesis of carbon gel aims to obtain an N-enriched porous adsorbent for CO<sub>2</sub> adsorption. Urea and furfural were used in gel synthesis with a simple reaction process at low temperatures to prepare carbon gel adsorbent for CO<sub>2</sub> adsorption. This study demonstrates the assessment of urea-furfural carbon cryogel (CCUF) synthesis under different conditions, including different water and acid loadings (adsorbent screening) on CO<sub>2</sub> adsorption. This work includes the characterisation of selected CCUF using different techniques, including N<sub>2</sub> sorption isotherms, CO<sub>2</sub>-TPD, FTIR, and SEM-EDX analyses.

## 2. Experimental

### 2.1 Material and chemicals

Urea (QRec, Asia), furfural (C<sub>5</sub>H<sub>4</sub>O<sub>2</sub>, Merck, Germany), and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 95-97 %, QRec, Asia) were purchased for use in carbon gel synthesis. Distilled water was prepared in the laboratory and used as the reaction medium during the gel synthesis. Carbon dioxide (CO<sub>2</sub>, 99.995 %), Helium (He, 99.995 %), Nitrogen (N<sub>2</sub>, 99.995 %), Compressed air (99.995 %), and Hydrogen (H<sub>2</sub>, 99.995 %) gas cylinders were supplied by Mega Mount Industrial Gases, Malaysia for gas adsorption and analysis.

### 2.2 Synthesis and characterisation of urea-furfural carbon cryogel

The synthesis of carbon cryogel from urea and furfural was conducted by mixing both chemicals at a molar ratio of 1:2. The reaction between urea and furfural was conducted in an acidic medium. The different volumes of distilled water (3 to 6 mL) were added to the mixture as a diluent, followed by adding different loadings of concentrated H<sub>2</sub>SO<sub>4</sub> (0.05 to 0.2 mL) as a catalyst to promote resinification. The amount of water and acid added into the mixture were studied to observe the effects on carbon gel production and its performance for CO<sub>2</sub> adsorption. The mixture was stirred for 30 min to produce a dark brown oligomer. The sample was pre-frozen for 2 h and freeze-dried at a condenser temperature of -60 °C for 4 h to form cryogel (CUF). The CUF sample was further calcined at 500 °C (10 °C/min) for 2 h to produce CCUF. The CCUF samples produced were labelled as CC-X-Y where X=1, 2, 3, and 4 represent 3, 4, 5, and 6 mL of water, and Y= 1, 2, 3, and 4 represents 0.05, 0.1, 0.15, and 0.2 mL of acid loading. The selected CCUF was characterised to determine the chemical and physical properties via N<sub>2</sub> sorption isotherms analysis (Thermo Scientific Surfer instrument), Fourier transform infrared spectroscopy (FTIR, Perkin-Elmer Spectrum), CO<sub>2</sub> temperature-programmed desorption (CO<sub>2</sub>-TPD, Micromeritics AutoChem II 2920 V4.03) and focused ion beam-scanning electron microscopy-energy dispersive X-ray (FIBSEM-EDX, ZEISS Crossbeam 340 with GEMINI column).

### 2.3 CO<sub>2</sub> adsorption performance by CCUF

The CO<sub>2</sub> adsorption process was carried out using various CCUF samples as adsorbents. The result of CO<sub>2</sub> adsorption capacity was used to evaluate the selection of CCUF synthesised with the different volumes of water and acid. The performance of the CCUF sorbents in the CO<sub>2</sub> adsorption was conducted through a fixed-bed adsorption process. A fixed-bed quartz tube column (L= 38.5 cm and D= 1.2 cm) was fitted into a set-up of an adsorption system that was equipped with a gas line, mass flow controller (MFC, Alicat Scientific MC Series), and online gas chromatography (Agilent 6890 Plus). The mass flow controller was used to set the gas flowrate with an accuracy of 1 % full scale and a repeatability of 0.1 % full scale. The system was purged by nitrogen gas and a flow of CO<sub>2</sub> gas was used to check the feed concentration (blank test) through gas chromatography (GC) without the adsorbent presence. The nitrogen gas was purged again before 3 g of adsorbent was added into the tube (placed in the centre of the quartz tube), and then the CO<sub>2</sub> gas was introduced (30 mL/min) at the standard temperature and pressure (STP) for the adsorption test. The outlet concentration of the CO<sub>2</sub> gas was evaluated using GC that was fitted with a thermal conductivity detector and a flame ionisation detector (TCD-FID). An online GC continuously monitored the outlet gas concentration until the saturation point. The saturation

adsorption capacity (in mmol/g) of CO<sub>2</sub> over CCUF was calculated based on Eq(1) (Kasikamphaiboon and Khunjan, 2018):

$$W_{CO_2} = \frac{Q_{in} \rho_{CO_2}}{m M_{CO_2}} \int_0^{t_{eq}} \left(1 - \frac{C_{CO_2, out}}{C_{CO_2, in}}\right) dt \quad (1)$$

where  $W_{CO_2}$  is the saturation adsorption capacity of CO<sub>2</sub> over CCUF,  $Q_{in}$  is the total volumetric feed flow rate at the standard temperature and pressure (STP);  $\rho_{CO_2}$  is the density of CO<sub>2</sub> at STP,  $C_{CO_2, in}$  and  $C_{CO_2, out}$  are defined as the initial feed and effluent concentration of CO<sub>2</sub>,  $t_{eq}$  is the equilibrium time at which the outlet concentration over the inlet concentration is equal,  $m$  is the mass of the CCUF placed into the fixed bed column, and  $M_{CO_2}$  is the molar mass for CO<sub>2</sub>.

### 3. Results and discussion

The study on the effect of water on the CUF and CCUF yield and CO<sub>2</sub> adsorption performance is shown in Figure 1a. The quantity of water used in the mixture of urea and furfural for carbon synthesis is selected based on the CCUF yield and CO<sub>2</sub> adsorption capacity performance. The high adsorption capacity of CO<sub>2</sub> is considered as the main factor for the selection of the water volume used in gel synthesis conditions as the CUF and CCUF yields show small differences. The increase of water in the mixture showed a small decrement in the CUF yield, but the CO<sub>2</sub> adsorption capacity showed a significant increase up to 2.04 mmol/g when the water used in the reaction was increased to 4 mL. A reduction in CO<sub>2</sub> adsorption capacity was observed as the water volume was further increased above 4 mL. The adsorption capacity was slightly increased but does not show a significant change when 6 mL of water is applied in gel synthesis. This is possibly due to the small difference in CCUF surface area or pore structure effect. The amount of water used during the urea-furfural gel synthesis is one of the important parameters as the added water is used to dilute the mixture concentration and help in the development of gel structure, especially textural properties of the carbon (Rey-Raap et al., 2017). The significant change in the adsorption capacity of CO<sub>2</sub> can be related to the changes in carbon surface structure such as surface area and pores affected by the different volumes of water used in the synthesis process. Tamon et al. (1997) found that the amount of water could affect the pores in the resorcinol-formaldehyde gel where the total pore volume is higher at lower resorcinol to water ratio. This explains the optimum amount of water required for the gel synthesis to obtain a good structure of gel and carbon. An excess of water can cause a negative effect on the gel synthesis as the polymeric gel is diluted and might lead to the destruction of gels and pore structure. Babić et al. (2004) found that a higher volume of water decreased the total surface area of the gel. This can be related to the reduction of the CUF yield when the volume of water is further increased as the reaction medium becomes diluted for the polymerisation reaction. The possible reduction of CCUF surface area caused by the increasing volume of water could directly affect and reduced the adsorption performance. Based on the result of CO<sub>2</sub> adsorption capacity, the CCUF synthesised at different water volumes, 4 mL of water was selected for further study on the effect of the different acid loading. This result is consistent with the previous work that reported a similar water volume of 4 mL used for the gel synthesis (Martínez-García et al., 2004).

The acid was used to initiate and catalyse the sol-gel polycondensation reaction for the final formation of the polymeric structure. Figure 1b shows the effect of acid loading on the CUF and CCUF yield and the CO<sub>2</sub> adsorption performance by synthesised CCUF. The results show that the CUF and CCUF yield increased with the increase of acid loading. An increase in CO<sub>2</sub> adsorption capacity by synthesised CCUF was observed when an acid loading up to 0.15 mL was applied. The increasing of acid loading could accelerate the reaction and reduce the gelation time in producing urea-furfural gel. More monomers can be clustered together to undergo the gelling process and lead to denser product material. The optimum amount of acid is required as an excess of acid leads to the self-reaction of furfural and reduces the reactant concentration (Martínez-García et al., 2004). The reduction of adsorption capacity was recorded when the acid loading was increased to 0.2 mL. The yield of CUF and CCUF did not show much difference at high acid concentrations. However, the adsorption capacity results show a reduction, possibly due to the CCUF surface structure and texture caused by increasing acid loading. The fast reaction due to high activity provided by high acid catalyst leads to the formation of non-porous carbon (Rey-Raap et al., 2017). Excess acid loading could induce more urea-furfural monomers to be clustered together and form non-porous carbon. This may cause a reduction in the surface area, leading to a plummet in the CO<sub>2</sub> adsorption capacity. Based on CO<sub>2</sub> adsorption capacity, the 0.15 mL acid loading was selected for CCUF synthesis, which can be used for further testing in the CO<sub>2</sub> adsorption study. The adsorption capacity of 2.04 mmol/g by CCUF could be further optimised. The previous work has reported on the CO<sub>2</sub> adsorption using carbon material that achieved an adsorption capacity in the range of 0.89-6.2 mmol/g (Wang et al., 2020). The adsorption capacity of CO<sub>2</sub> using CCUF samples synthesised at different water volumes and acid loadings are shown in Figure 2. Sample CC-2-3 represented the selected CCUF sample based on high

CO<sub>2</sub> adsorption capacity. All the samples mainly reach the equilibrium time between 30 to 40 min of the adsorption process with the CO<sub>2</sub> inlet flowrate of 30 mL/min. A longer time implies better retention of CO<sub>2</sub> adsorbate in the adsorbent to be completely saturated and corresponds to a better adsorption capacity. This explains that the adsorption of CO<sub>2</sub> using CCUF samples (at different water and acid loading) was saturated after 40 min.

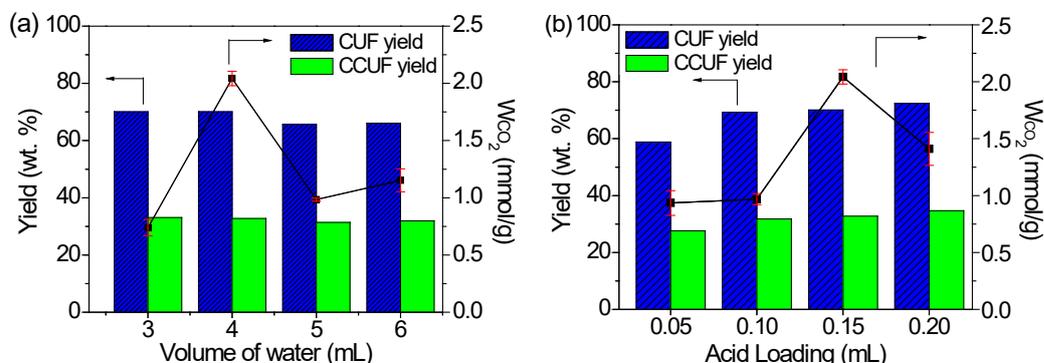


Figure 1: Effect of (a) water (at 1:2 feed ratio, 0.15 mL of H<sub>2</sub>SO<sub>4</sub>) and (b) acid loading (at 1:2 feed ratio, 4 mL of water) on CUF and CCUF yield, and CO<sub>2</sub> adsorption capacity.

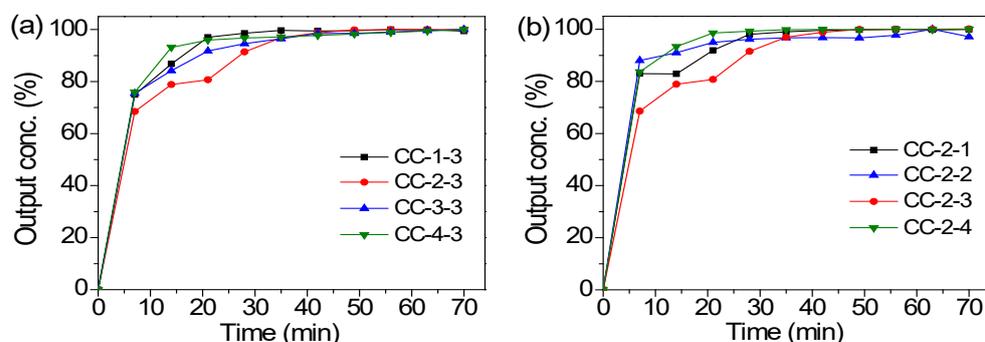


Figure 2: CO<sub>2</sub> concentration profile for adsorption by various CCUF at different (a) water and (b) acid loading.

Some characterisations were conducted on the selected CCUF samples (CC-2-3) to observe the surface structure and properties related to the application for CO<sub>2</sub> adsorption. The surface area analysis of selected CCUF shows a high microporosity (about 94 %) of carbon surface area, where the CCUF can be categorised as a microporous carbon material (Table 1). The total surface area was determined to be 160 m<sup>2</sup> g<sup>-1</sup> and the total pore volume of 0.12 cm<sup>3</sup> g<sup>-1</sup> was reported. The high microporosity of CCUF with a total microporous area of 160 m<sup>2</sup> g<sup>-1</sup> gives an advantage as a potential adsorbent for effective CO<sub>2</sub> adsorption. Previous work has stated that high microporosity carbon materials are required for highly efficient CO<sub>2</sub> adsorption (Wang et al., 2020). A large surface area, especially the microporous area, is required to increase the adsorption process. The smaller surface area decreases the CO<sub>2</sub>-philic sites exposed to adsorb CO<sub>2</sub> and lowers the chance for the interaction between the adsorbent and the CO<sub>2</sub> molecules, which directly reduces the CO<sub>2</sub> adsorption capacity (Wahby et al., 2010).

Table 1: The texture properties and basicity of the selected CCUF (CC-2-3) sample

Textural properties	Value	Surface basicity region	Basicity (mmol/g)
BET Surface Area, S <sub>BET</sub> (m <sup>2</sup> g <sup>-1</sup> )	160	Low (<100 °C)	0.04
Micropore surface area (m <sup>2</sup> g <sup>-1</sup> )	151	Moderate (200-500 °C)	0.55
Mesopore surface area (m <sup>2</sup> g <sup>-1</sup> )	9	High (>500 °C)	3.97
Total pore volume, V <sub>tot</sub> (cm <sup>3</sup> g <sup>-1</sup> )	0.12	Total basicity	4.56

The surface basicity of the CCUF sample was determined using CO<sub>2</sub>-TPD analysis and the result is shown in Table 1. The CO<sub>2</sub> molecules with acidic properties could interact with surface basic sites properties (CO<sub>2</sub>-philic sites) in the adsorption process. Based on the CO<sub>2</sub>-TPD thermograph, the plot can be divided into three main regions related to the low, moderate, and high basicity strength of the CCUF sample. This explains that the

amount of CO<sub>2</sub> desorbed reflects the amount of CO<sub>2</sub> chemically adsorbed on the sample (Tajbakhsh et al., 2020). The high basicity of CCUF is reported with 3.97 mmol/g from the total basicity of 4.56 mmol/g. Previous work has reported the basicity of the material and stated that the strength of basic sites is correlated to the active sites for CO<sub>2</sub> conversion (Han et al., 2020). Initially, the CO<sub>2</sub> conversion would involve the adsorption of CO<sub>2</sub> on the basic sites. High total basicity could explain higher adsorption of CO<sub>2</sub> on the adsorbent, which can be related to the adsorption capacity of CO<sub>2</sub>.

The broad peak between 3,200 and 2,500 cm<sup>-1</sup> corresponding to N–H stretching of urea was observed in the CUF structure. The other peaks at 1,400 – 1,700 cm<sup>-1</sup> represent the C=O bonding of amide and C=C bonding in the CUF and CCUF structure. The carbonyl group (C–O) is presented at a frequency around 1,000 - 1,100 cm<sup>-1</sup>. This peak detection has been reported by previous work on carbon gel formed from urea and furfural (Yussuf et al., 2021). The =C–N bonding of the amide group and –N–H bend are observed at the respective regions of 2,200 – 2,500 cm<sup>-1</sup> and 700 – 800 cm<sup>-1</sup>. All the IR peaks are clearly observed for the CUF sample, while some of the peaks are reduced or not clearly observed from the CCUF sample due to rearrangement of the CUF structure caused by calcination. The bonding and functional group present in the CCUF structure shows the potential interaction of the N atom with CO<sub>2</sub> for the adsorption process.

The morphologies of the samples of selected CUF and CCUF were observed from the scanning electron microscopy (SEM) images, as shown in Figure 3. The SEM images revealed that the polymer matrix of the CUF sample has a bulk morphology with a smooth surface with low porous structure formation on the surface. The CCUF prepared after the calcination of CUF showed a rougher surface morphology and the changes might be due to the surface deformation affected by heat treatment. The CCUF sample contains unevenly distributed and irregular shapes of microporous structures, which contribute to CO<sub>2</sub> adsorption. The EDX graph shows that the carbon composition of the CCUF increases compared to the CUF sample due to the decomposition of O and N elements during calcination. The degradation of the O and N elements has reduced their contents and caused the percentage composition of the remaining C element in the structure to increase. The N element present in the CCUF structure is useful for interaction with CO<sub>2</sub> in the adsorption process. The N element detected through EDX supports the N-containing functional group in the FTIR results.

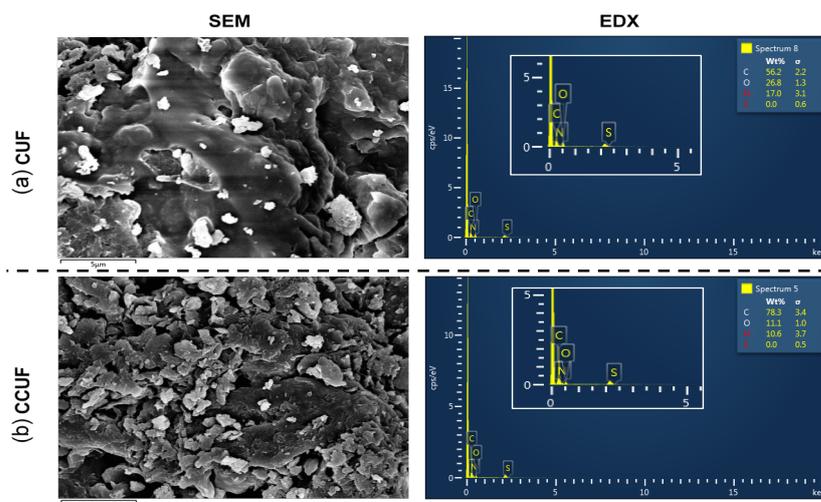


Figure 3: SEM images with EDX graph of selected (a) CUF and (b) CCUF (CC-2-3).

The relationship between the CCUF properties from the characterisation and its performance on CO<sub>2</sub> adsorption is important to explain the interaction in the adsorption process. Previous work has stated that van der Waals force or electrostatic interaction and their combination are related to the CO<sub>2</sub> adsorption in porous carbons (Kumar et al., 2015). Ma et al. (2020) have stated that the van der Waals force is associated with the porosity of carbon, and the doped heteroatoms (such as N and S) in the carbon structure can be related to the electrostatic or Coulombic forces. It has been highlighted that the preparation of high surface areas of microporous materials with tunable pore structures, and the association with a high amount of the doped heteroatoms, could be targeted to enhance the CO<sub>2</sub> adsorption.

#### 4. Conclusions

The CCUF was successfully synthesised via the sol-gel polycondensation with a large surface area and high basicity. The surface texture and properties of CCUF have enhanced its performance for CO<sub>2</sub> adsorption. High

microporosity and N-enrichment of the surface chemicals of CCUF become factors of the high adsorption capacity of CO<sub>2</sub>. The selection of the parameters in synthesised CCUF shows that the optimum loadings of 4 mL of water and 0.15 mL of acid are required for the mixing and gelling process. A longer time to reach for CO<sub>2</sub> saturation indicates the CCUF has high accessibility for CO<sub>2</sub> adsorption and can retain the CO<sub>2</sub> molecules inside the adsorbent porous structure. The cost-effectiveness of CCUF adsorbent due to low-cost material and simple process for carbon synthesis give advantages and potential for CO<sub>2</sub> capture applications. Future work could be conducted to optimise the adsorption parameters and CO<sub>2</sub> adsorption capacity, and this includes further modification of CCUF via suitable activation to improve the surface properties and enhance the CO<sub>2</sub> adsorption capacity.

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