Synthesis and Characterization of MSU-2 for CO₂ Adsorption

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Abstract. The advancement of adsorption technology is highly dependent on the characteristics of the adsorbents used. However, the commonly used one-dimensional (1D) MCM-41 and two-dimensional (2D) SBA-15 were found to induce gas diffusion difficulty. With this, MSU-2 was introduced as it shows high potentiality to be employed as CO₂ adsorbent due to its excellent properties such as the highly interconnected three-dimensional (3D) wormhole-like framework structure as well as large specific surface area, total pore volume and pore size. The focus of the current study focuses on the synthesis of MSU-2 and the investigation on the CO₂ adsorption capacity of the synthesized MSU-2 at various temperatures and pressures. The preparation of the MSU-2 was achieved by fluoride-assisted two-step process of solution precipitation method involving the addition of tetraethylorthosilicate (TEOS) to the acidified aqueous solution of nonionic surfactant, Triton X-100. The resultant MSU-2 was characterized by SEM, TEM, SAP analyser, XRD, FTIR spectrometer and TGA. The analyzed results showed that MSU-2 with desired features was successfully synthesized. The CO₂ gas adsorption studies of the MSU-2 was demonstrated at different temperatures (25 °C and 85 °C) and different pressures (1 bar and 5 bar) to study the effect of temperatures and pressures on the CO₂ adsorption capacity of the MSU-2. The highest CO₂ adsorption capacity of the MSU-2 was found to be 0.98 mmol-CO₂/g-adsorbent when the operating conditions were set at 25 °C and 5 bar. This adsorption experiment also deduced that low adsorption temperature and high operating pressure promote adsorption process which give higher CO₂ adsorption capacity.

1. Introduction
In recent years, the environmental issues which are directly or indirectly ascribed to the enormous emission of carbon dioxide (CO₂) into the Earth’s atmosphere have become more severe. CO₂ gas, which is one of the greenhouse gases (GHGs) has caused adverse effect on the environment, which include global warming [1], increasing temperature of the oceans [2], melting of the ice sheets [3], increasing of sea level [4] as well as other extreme weather phenomena [5]. The main source of CO₂ is from the...
power plants used for fossil fuels combustion in order to generate energy for industrial application [6]. According to the assessment conducted by the International Energy Agency (IEA), in year 2018, the increment of the average annual global surface temperature was 1 °C where 0.3 °C of this elevation was attributed to the CO2 emitted from the burning of fossil fuels, mainly coal [7]. Besides, the records of the average annual global CO2 concentration since year 1980 reported by the National Oceanic and Atmospheric Administration (NOAA) is displayed an increment trend where the highest record was 412.02 ppm for December 2019 [8]. This is a bad sign as the value is very close to the critical CO2 concentration in the atmosphere which is 426 ppm [6]. Therefore, various measures need to be used to reduce the release of CO2 to the atmosphere. CO2 capture and sequestration (CCS) is one of the commonly used approach in controlling CO2 emission by conducting either pre-combustion capture, oxy-fuel combustion or post-combustion capture [9-11]. Among these techniques, post-combustion CO2 capture is extensively utilized because of its low cost, flexibility and excellent CO2 capturing capability [12, 13].

A few types of gas separation technologies which are generally used in post-combustion CO2 capture include absorption, membrane separation, cryogenic distillation and adsorption. However, these technologies possess their respective drawbacks which hinder their expansion in large-scale application. Absorption technology using liquid amine is energy intensive because of the stripping process consumed a large amount of energy to regenerate the absorbent [14]. Besides, this technology is also facing other problems such as the corrosive nature of liquid amine, rapid solvent evaporation and amine degradation in the presence of oxygen [11, 14-18]. For membrane technology, it is not suitable to be used when the partial pressure of CO2 in the feed gas stream is low and it is difficult to be upscaled for industrial operation due to high energy requirement and high cost [11, 16, 18, 19]. Moving on, high energy requirement during operation and the issue of CO2 refrigeration at low temperature are the two main concerns of utilizing cryogenic distillation in industrial-scale [11, 20]. On the contrary, as compared to the CO2 capture technologies abovementioned, adsorption technology exhibits relatively better properties where this technology consumes lesser energy and thus incurs lower operating cost. In addition, the solid adsorbents are cheap due to easy regeneration process, no fouling issue, as well as low toxic level as the amines are impregnated onto the adsorbent surface instead of mixing in bulk liquid solvent [21]. Moreover, adsorption technology is the best choice for post-combustion CO2 capture due to its suitability for CO2 separation from low concentration feed gas stream [11]. Therefore, a lot of researchers have been investigating on this adsorption technology in order to enhance its effectiveness.

The working principle of the adsorption technology is basically governed by the surface energy of the adsorbent surface. During adsorption process, the targeted gas adsorbate molecules are automatically drawn to the surface of the solid adsorbent and attached on it to form a molecular film, leaving a gas stream which has high concentration of less strongly adsorbed components [11, 22]. Therefore, in order to ensure excellent CO2 adsorption performances, the typical characteristics for suitable adsorbent include highly interconnected porous structure, adjustable pore size, large total pore volume and specific surface area [23], as well as possess high affinity with the targeted gas [24]. Zeolites, activated carbons [1], metal organic frameworks (MOFs) [25], metal oxides [26], microporous organic polymers (MOPs) [23] and mesoporous silicas [11, 23] are the types of porous adsorbents which are usually utilized for CO2 adsorption application. Among all these solid CO2 adsorbents, mesoporous silicas have been employed for CO2 adsorption application since a decade ago. For example, Kishor et al. [27] have studied the effect of the structural parameters of KIT-6, MCM-41 and SBA-15 on the CO2 adsorption capacity while Wang et al. [28] have investigated the influence of the adsorbents pore structures with different dimension on the CO2 adsorption performance. The comprehensive outcomes from these research works indicated that 3D adsorbents such as KIT-6, MSU-J, MCF and HMS have better performance in CO2 adsorption as compared to the 1D MCM-41 and 2D SBA-15. This is due to the 1D and 2D arrangement induce gas diffusion difficulty, hence the problem of diffusion resistance in the pore framework is the main key challenge faced by the existing mesoporous silica adsorbents.

Therefore, in order to overcome the problem addressed above, the utilization of the adsorbent from the mesoporous silica family of Michigan State University (MSU) was proposed. A few types of MSU
materials such as MSU-1 and MSU-J have been synthesized and used in CO₂ adsorption application. For instance, MSU-1 has been synthesized by Wang et al. [29] whereas MSU-J has been produced by Jiao et al. [19, 25]. By comparing the results obtained from these studies, the MSU-2 synthesized by the previous literature [30-32] possesses similar morphology and pore structure as MSU-1 and MSU-J. With this, MSU-2 appears to be a promising CO₂ adsorbent due to its 3D wormhole-like framework structure which is highly interconnected, as well as large specific surface area and total pore volume. This outstanding mesopores connectivity not only eases the molecular diffusion of CO₂ gas within the pore channels of MSU-2 and thus minimizes the CO₂ gas diffusion resistance, but also facilitate post surface modification of MSU-2 to further enhance the CO₂ adsorption performance of MSU-2 [28]. Generally, there are various methods used to synthesize mesoporous silica, including MSU-2. The most suitable technique is the solution precipitation method as the resultant adsorbent contains a large amount of silanol hydroxyl groups which act as the sites for surface modification by using functional organics [33]. Although the synthesis method of the MSU adsorbents are the same, they are using different surfactants and operating conditions to obtain the desired morphology and pore structure.

In this study, MSU-2 was synthesized by using TEOS and Triton X-100 as the silica precursor and surfactant, respectively under 55 °C for 48 hours via a fluoride-assisted two-step process of solution precipitation method. There were two distinct steps involved in this process including the aggregation of the silica and surfactant and followed by the silica condensation catalysed by using fluoride ions [30, 31, 34]. To the best knowledge, no study has been reported on the CO₂ adsorption performance of MSU-2 so far. The synthesized MSU-2 in this study was characterized by using various analytical instruments in order to evaluate its properties which include morphology, pore structure, pore characteristics, crystallinity, functional groups and thermal stability. Besides, CO₂ gas adsorption experiment was conducted to investigate the effect of temperatures and pressures on the CO₂ adsorption capacity of the MSU-2 synthesized. The aim of the current study was to determine the optimum operating conditions of MSU-2 adsorption where the highest CO₂ adsorption capacity is achieved.

2. Experimental

1.1. Materials
Tetraethylorthosilicate (TEOS, ≥99%, C₄H₉O₄Si, MW = 208.33, density = 0.94 g/ml) was purchased from Merck and used as the silica precursor for synthesis of MSU-2. Triton X-100 [(C₆H₄O)nC₄H₂NO (n = 9-10), MW = ~647, density = 1.065 g/ml] was obtained from Sigma Aldrich and used as the template for MSU-2 fabrication. Hydrochloric acid fuming 37% (HCl, EMSURE®, ACS, ISO, Reag. Ph Eur) was also purchased from Merck whereas sodium fluoride (NaF, ≥98.5%, MW = 41.99) was obtained from R&M Chemicals. All the chemicals were of analytical grade. The CO₂ gas tank (99.99%), CH₄ gas tank (99.99%) and N₂ gas tank (99.99%) were purchased from Air Products.

1.2. Synthesis of MSU-2
MSU-2 was synthesized via solution precipitation method involving a fluoride-assisted two-step process where firstly the self-assembly of silica-surfactant followed by the silica condensation for the formation of colloidal sol [31]. TEOS was added into 100 mL of 0.02 M Triton X-100 aqueous solution under stirring where the molar ratio of TEOS to Triton X-100 is 8:1. Diluted HCl was added drop by drop until the TEOS/Triton X-100 mixture reached pH 2. The resulting solution was then undergone a 30-minute sonication, 5-hour stirring and 10-hour aging process (no stirring) in order to obtain a clear solution. To facilitate silica condensation process, NaF powder was mixed into the aged solution under high rpm stirring with a NaF/TEOS molar ratio of 0.04:1. Next, the beaker was placed into a pre-heated water bath at 55 °C for 48 hours without stirring. After filtration step, the white substance obtained was washed thoroughly with distilled water, dried in the oven and followed by calcination in air at 620 °C for 6 hours with a 3 °C/min heating rate to remove the surfactant.
1.3. Characterization

Scanning electron microscope (SEM, Hitachi TM3030 Tabletop) operated under an acceleration voltage of 5.00 kV was used to study the morphology of the MSU-2. The pore structure of the MSU-2 was investigated by using transmission electron microscope (TEM, Hitachi HT7830) operated with a LaB₆ filament as a source of electrons under an accelerating voltage of 80 kV. Prior to the TEM characterization, the powdered MSU-2 was sonicated in ethanol. The suspension was then dropped onto a copper grid and dried under ambient environment. Surface area and porosity analyser (SAP, Micromeritics TriStar II 3020) was used to evaluate the pore characteristics of the MSU-2. The MSU-2 was pre-treated under vacuum at 65 ºC for 24 hours before the SAP measurement in order to remove the moisture and pre-adsorbed gases on the MSU-2 surface. X-ray diffractometer (XRD, Bruker D2 PHASER Benchtop) was utilized to study the crystallinity of the MSU-2. The XRD diffraction patterns were obtained at room temperature by using Cu Kα X-ray radiation in the 2θ range from 0.01° to 10° with a scanning step of 0.01°. The functional groups and chemical bonding present in the MSU-2 were determined by using Fourier transform infrared spectrometer (FTIR, PerkinElmer Frontier). By utilizing the KBr wafer technique, the absorption band of the MSU-2 was obtained by conducting the scans from 4000 cm⁻¹ to 400 cm⁻¹. Thermogravimetric analyser (TGA, PerkinElmer Simultaneous Thermal Analyzer STA 6000) was used to investigate the thermal stability of the MSU-2 where it was heated under N₂ atmosphere from room temperature (25 °C) to 800 °C at a 5 °C/min heating rate.

1.4. CO₂ adsorption studies

The CO₂ adsorption studies of the MSU-2 was conducted in a column reactor with 10 mm inner diameter and a gas analyser (Gensonic Technology NDIR sensor, Model G2-03) was used to collect the raw data throughout the experiment. Firstly, MSU-2 was loaded into the gas adsorption column reactor and installed into the gas rig. A 3-hour pre-treatment process was conducted by passing N₂ gas with a flow rate of 100 ml/min through the MSU-2 in the column reactor at 100 ºC in order to remove the gases and moisture adsorbed on the surface of MSU-2. Next, the feed gas mixture of 50% CO₂ gas and 50% CH₄ gas was passed through the MSU-2 for CO₂ gas adsorption measurement. The operating pressures and temperatures being studied in this experiment were 1 bar, 5 bar, and 25 °C, 85 °C, respectively. The values of the outlet gas compositions were collected by Gensonic gas analyser at 1-second interval. The CO₂ adsorption capacities of the MSU-2 under different operating conditions were then calculated by using the breakthrough curve plotted from the raw data.

3. Results and discussion

3.1. Characterization of the MSU-2

The morphology and pore structure of the synthesized MSU-2 was characterized by using SEM and TEM, respectively. The SEM image in Figure 1 shows that the synthesized MSU-2 possessed a monodisperse spherical morphology which is in good agreement with the results reported in the previous research works [30, 32]. The formation of these spherical particles was ascribed to the mild acidic TEOS/Triton X-100 solution in hydrolysis step [30] as well as the steady silica condensation process before the addition of NaF which catalysed the condensation step [31, 35]. Besides, it is observed that the MSU-2 produced exhibited a disordered wormhole-like porous structure as shown in Figure 2 which is similar to the TEM image reported by Pérez-Quintanilla et al. [32]. No amorphous pattern was detected in the TEM image of the MSU-2.
Figure 3 illustrates the nitrogen adsorption/desorption isotherm of the MSU-2. In accordance with the IUPAC classification of adsorption isotherms [36-38], the N$_2$ adsorption isotherms of the MSU-2 is a Type IV adsorption isotherm, indicating that the MSU-2 adsorbent consisted of a well-defined mesostructure which is in close agreement with the research works presented by Jiao et al. [25], Jiao et al. [19], and Wang et al. [29]. The typical characteristic of this Type IV adsorption isotherm is the strong interaction between the adsorbent surface and the targeted gas adsorbate molecules [36-38]. Besides, the hysteresis loop which appeared around the relative pressure (p/p$_0$) of approximately 0.43 to 0.80 denoted that the pore condensation of N$_2$ molecules took place in the mesoporous structure of the MSU-2 [14, 19, 28, 32]. Moving on, the pore characteristics of the synthesized MSU-2 which tabulated in Table 1 show that it exhibited a high BET specific surface area (S$_{BET}$) of 1223 m$^2$/g, a large total pore volume (V$_p$) of 0.87 cm$^3$/g as well as a large pore size (d$_p$) of 4.0 nm. As compared to the results reported by Boissiere et al. [31], the results obtained show that the current study was able to fabricate MSU-2 with enhanced pore characteristics using the same silica precursor and surfactant. This improvement might be due to the operating conditions and the techniques used during the synthesis process where the mild acidic TEOS/Triton X-100 solution during hydrolisis step quickly broke the emulsion and formed a homoegeneous colourless aqueous solution. Owing to the mild acidic solution, the silica condensation process was stable and lead to the formation of spherical nanoscopic micellar hybrid objects with enhanced pore characteristics.

In addition, the characterization analyses obtained from XRD and FTIR indicated that the MSU-2 was successfully synthesized via the fluoride-assisted two-step process of solution precipitation method in the current work. The XRD diffraction pattern of the MSU-2 is shown in Figure 4. It exhibited a narrow XRD peak at 2θ value of about 1.0° which resembles the XRD pattern reported in the previous study [32, 39]. This well-resolved low angle XRD pattern was assigning to (100) crystal face diffraction [29, 32]. The significant features presented by this XRD pattern such as high intensity and narrow peak showed that the MSU-2 produced in the current study possesses high crystallinity which also can be interpreted as the formation of wormhole-like mesostructure [30].

| BET specific surface area, S$_{BET}$ (m$^2$/g) | Total pore volume, V$_p$ (cm$^3$/g) | Pore size, d$_p$ (nm) | Reference |
|--------------------------------------------|-------------------------------|-----------------|-----------|
| 1223                                      | 0.87                          | 4.0             | This study|
| 980                                       | 0.47                          | 2.5             | [31]      |
Furthermore, the significant features which is similar to the results reported in the previous literature [29, 32] were also observed from the FTIR spectrum of the MSU-2 synthesized in this study as shown in Figure 5. These significant features include a large broad band which centred at the peak of about 3467 cm\(^{-1}\), and the peaks at wavenumber of 1647 cm\(^{-1}\), 1077 cm\(^{-1}\), 800 cm\(^{-1}\) and 461 cm\(^{-1}\). According to
the correlations of group vibrations to regions of infrared absorption [40, 41] and spectra-structure correlations of infrared analysis for organosilicon compounds [42, 43], Table 2 shows the type of bonding represented by each of the peaks in the FTIR spectrum of the MSU-2. To be more detailed, the strong peak at 1077 cm$^{-1}$ was associated with a shoulder at around 1217 cm$^{-1}$. This phenomenon is due to the overlapping of two bands which indicate that the siloxane chains in the MSU-2 structure are bulky or long, hence causing the -$\text{Si-O-Si}$- absorption to become more complicated [42, 43].

![FTIR spectrum of the MSU-2.](image)

**Table 2.** Characteristic bonds shown in the FTIR spectrum of the MSU-2 [40-43].

| Wavenumber (cm$^{-1}$) | Characteristic bond                                                                 |
|------------------------|-------------------------------------------------------------------------------------|
| 3467                   | O-H stretching vibrations of the hydrogen bonded surface silanol groups and the remaining adsorbed water molecules [14, 19, 25, 44] |
| 1647                   | Deformation vibrations of physically adsorbed water molecules [14, 19, 29, 44]        |
| 1077                   | Asymmetrical stretching ($\nu_{as}$) of siloxane (-$\text{Si-O-Si}$-) band [14, 19, 25, 29, 44, 45] |
| 800                    | Si-O bond symmetrical stretching ($\nu_s$) of the silanol groups [19, 25, 29]          |
| 461                    | Bending vibration ($\delta$) of siloxane (-$\text{Si-O-Si}$-) band [29]                |

Lastly, the TGA profile and DTG curve shown in Figure 6 were analysed to evaluate the thermal properties of the MSU-2. Basically, TGA profile indicates the weight percentage loss of the adsorbent as a function of temperature, whereas DTG curve provides the degradation temperature of the adsorbent which is the temperature at the maximum weight loss rate. From the TGA profile of MSU-2, it can be observed that the MSU-2 exhibited one-step degradation which ranged from 30 °C to 40 °C and the further temperature increment until 800 °C did not incur any significant weight loss. This result indicates that the MSU-2 possessed high thermal stability as the total weight loss was a mere 9.02%. Prior to 100 °C, the small drop of 3.6% in weight percentage was attributed to the removal of physically pre-adsorbed water and gas molecules from the MSU-2. At 800 °C, the remaining silica solid residue of the MSU-2 was about 90.98%, which is relatively high. The TGA profile and DTG curve of the MSU-2 obtained
from the current study displayed similar pattern as compared with the results reported in the previous literature [14, 19, 21, 27, 29, 44, 46, 47].

Figure 6. TGA profile and DTG curve of the MSU-2.

### 2.1. CO$_2$ gas adsorption on the MSU-2

The CO$_2$ adsorption experiment of the MSU-2 was conducted under two different temperatures (25 °C and 85 °C) and two different pressures (1 bar and 5 bar). The results are shown in Figure 7 and it is observed that the CO$_2$ adsorption capacity of the MSU-2 is 0.67 mmol-CO$_2$/g-adsorbent at 25 °C and 1 bar. This result is relatively higher than the CO$_2$ adsorption capacity of various MSU adsorbents in the previous research works, where MSU-1 and MSU-J possessed 0.50 and 0.54 mmol-CO$_2$/g-adsorbent, respectively [19, 29].

From Figure 7, it can be observed that when the temperature increased from 25 °C to 85 °C, the CO$_2$ adsorption capacity of the MSU-2 obtained from both 1 bar and 5 bar pressures experienced a decrement. On the other hand, at a fixed temperature, it is found out that increasing pressure enhanced the CO$_2$ adsorption capacity of the MSU-2. These two phenomena were complied with Le Chatelier’s principle. According to the equilibrium equation shown in Figure 8, the process is reversible in which the forward reaction is adsorption whereas the backward reaction is known as desorption. In order to favour the forward adsorption process, low adsorption temperature and high system pressure are preferred [48, 49].

Generally, CO$_2$ adsorption process is an exothermic process where heat of adsorption is emitted when 1 g of CO$_2$ gas is adsorbed on the adsorbent surface. Thus, increasing adsorption temperature caused the CO$_2$ gas molecules to gain a higher kinetic energy and collide more frequently with the MSU-2 surfaces. With this, the adsorption process evolved a large amount of heat energy which is not favourable for the forward adsorption process to occur [49]. To revert to the equilibrium state, backward desorption process took place by removing the CO$_2$ gas adsorbate molecules from the MSU-2 surface. In short, at a constant pressure, the adsorbed CO$_2$ gas molecules on the MSU-2 surface was decreasing with increasing adsorption temperature [48]. Therefore, a lower CO$_2$ adsorption capacity of MSU-2 was obtained at higher adsorption temperature.
Figure 7. CO$_2$ adsorption capacity of the MSU-2 at different pressures and temperatures.

\[
\text{adsorption: } \text{Gas} + \text{Adsorbent} \rightleftharpoons \text{Gas}---\text{Adsorbent} \\
\text{desorption:}
\]

Figure 8. The equilibrium equation of adsorption/desorption process [48, 49].

Besides, the CO$_2$ adsorption using MSU-2 is governed by physisorption where the CO$_2$ gas molecules are physically adsorbed on the surface of MSU-2 via van der Waals’ forces of attraction [23, 50]. Basically, the pressure of the system dropped continuously during CO$_2$ adsorption process. This is due to the CO$_2$ gas molecules in the bulk phase decreased when they adhered to the adsorbing surface of the MSU-2 [51]. Hence, by increasing the operating pressure, the amount of CO$_2$ gas molecules per unit volume in the bulk phase also increased. With this, the system tends to shift forward by forming adsorbate layers on the MSU-2 surface in order to reduce the amount of CO$_2$ gas molecules in the bulk phase [48]. Therefore, higher operating pressure led to a higher CO$_2$ adsorption capacity of the MSU-2 at a constant temperature.

4. Conclusion
In the current study, as verified by the analysis obtained from SEM, TEM, SAP, XRD, FTIR and TGA, MSU-2 has been successfully synthesized via solution precipitation method involving hydrolysis and condensation process. Triton X-100 and TEOS were utilized as the surfactant and source of silica, respectively. SEM and TEM showed that the MSU-2 synthesized possessed a monodisperse spherical morphology and a disordered wormhole-like porous structure, respectively. Besides, SAP analysis indicated the MSU-2 produced in this research work has high $S_{\text{BET}}$ of 1223 m$^2$/g, $V_p$ of 0.87 cm$^3$/g and $d_p$ of 4.0 nm. Furthermore, the narrow XRD peak at 2θ value of 1.0° and all the characteristic bands appeared in the FTIR spectrum have confirmed that the sample synthesized is MSU-2. Moreover, the TGA analysis showed that the MSU-2 exhibited high thermal stability where the total weight loss was small which is only 9.02%. Lastly, from the CO$_2$ gas adsorption experiment, it was found out that the CO$_2$ adsorption capacity of the MSU-2 is affected by the adsorption temperature and operating pressure of the system. In conclusion, a higher CO$_2$ adsorption capacity of the MSU-2 was obtained by decreasing the adsorption temperature as well as increasing the operating pressure.
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