Synthesis and characterization of MSU-2 synthesized by using tetraethylorthosilicate and Triton X-100

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Abstract. Adsorption technology is one of the well-established gas separation techniques as it can minimize cost and energy requirement for CO2 separation. Mesoporous silicas such as MSU-2 appears to be a good adsorbent as it comprises of three-dimensional (3D) wormhole framework structures that are highly interconnected which minimize the diffusion resistance of CO2 through its pore structure. Current study focus on the preparation of MSU-2 and investigation on the CO2 adsorption on the synthesized MSU-2. In this study, MSU-2 was prepared by using tetraethylorthosilica (TEOS) as a source of silica in the presence of non-ionic polyethyleneoxide (PEO)-based surfactants under an acidic condition where the pH is 2 at 55 ºC for 48 hours via the fluoride-assisted two-step synthesis process. The two main steps involved are hydrolysis of TEOS and condensation of silica. The morphology, crystallinity, functional groups and pore characteristics of MSU-2 were investigated by using characterization method of Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-Ray (EDX) Spectrometry, X-ray Diffractometer (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET). The synthesized MSU-2 was well crystallized and possessed a uniform monodisperse microspherical morphology with BET surface area, pore volume and pore size of 964 m²/g, 0.98 cm³/g and 4.1 nm, respectively. All the characterization results showed that MSU-2 was successfully synthesized via solution precipitation method. In conclusion, the high BET surface area of the synthesized MSU-2 shows that MSU-2 is a very potential candidate as a good adsorbent for gases.

1. Introduction
In recent years, natural gas industrial is gaining increasing popularity. Natural gas are commercially used as a fuel and the raw material for the synthesis of carbon black, helium, solid carbon dioxide, and numerous chemicals [1, 2]. This is because natural gas possesses a few advantages as compared to the other sources of fuel, such as manufactured gas, oil, coal, coke, wood, and electricity. Firstly, natural gas has relatively lower carbon content and contains higher heat units than manufactured gas [3, 4]. Besides, as compared to coal and oil, natural gas is more environmental-friendly as it burns cleaner [4, 5]. Generally, natural gas consists of largest portion of methane (CH4), which is in a range of 70% to 90%, while approximately 8% is CO2 [6, 7]. The energy produced by using natural gas is mainly contributed by CH4, however, the presence of CO2 not only decreases the energy content and the
heating value of the natural gas, it also leads to the occurrence of corrosion in the equipment and
transportation pipelines [8, 9]. Hence, it is crucial to reduce the content of CO\textsubscript{2} in the natural gas by
performing CO\textsubscript{2}/CH\textsubscript{4} separation via gas separation technologies. According to the standard for the
delivery of natural gas set by U.S. pipeline, only less than 2 mol\% of CO\textsubscript{2} can present in the gas piping
during transportation [8, 10].

Generally, there are various gas separation technologies being utilized for CO\textsubscript{2}/CH\textsubscript{4} separation
which include absorption, adsorption, membrane separation and cryogenic distillation [11]. However,
it is found out from the previous studies that there are a few drawbacks which limit the large-scale
application of absorption, membrane and cryogenic distillation technology. The stripping process of
the absorbent which is performed after the absorption process to regenerate the absorbent is energy
intensive [12]. Besides, other problems also include the fast evaporation of solvent, high viscosity of
the solution, degradation of amines in the presence of oxygen and amine loss [13, 14]. Membrane
technology offers low effectiveness when the natural gas feed has low CO\textsubscript{2} partial pressure or when
the flow rate of the natural gas feed is high [15]. On the other hand, operational problems are the main
concern of utilizing cryogenic separation in industrial-scale. Commonly, cryogenic separation is also
energy intensive, and hence incur high cost investment [15]. In contrast, adsorption technology has
low operating cost and low requirement of energy, as well as it is more suitable to be use for CO\textsubscript{2}
separation from low concentration stream [15]. Besides, the solid adsorbents are cheap due to the
energy consumed during regeneration process is low, no fouling will occur, as well as the toxic level is
minimized as the amines are impregnated onto the surfaces of adsorbents instead of mixing in the
liquid solvent [16]. Adsorption technology is a process where the gas (solute) accumulates on the
surface of solid adsorbents and forms a layer of adsorbate in the form of atoms or molecules [15, 17].

In order to achieve excellent CO\textsubscript{2} adsorption capacity, the physical adsorbents need to possess the
characteristics such as large and tunable pore sizes, large specific surface area and pore volume, aside
from multi-dimensional porous structure [18]. The most commonly used physical adsorbents are
activated carbons, zeolites, metal organic frameworks (MOFs), mesoporous silicas [19], metal oxides
[14], and microporous organic polymers (MOPs) [20]. Among all the porous supports, mesoporous
silica is found to be one of the potential adsorbents for CO\textsubscript{2} adsorption due to its excellent properties
such as large and uniform pores, adjustable pore sizes, in addition to large specific surface area [13].
Extensive research works on CO\textsubscript{2} adsorption have been carried out using mesoporous silicas including
Mobil Composition Matter (MCM) [21], Santa Barbara Amorphous type materials (SBA) [22] and
folded sheet mesoporous material (FSM) [18]. However, the most critical challenge faced by these
mesoporous silicas is due to the characteristic of their pore structure which will affect their
performance in gas adsorption. For instance, MCM-41 and SBA-15 have 1D and 2D cylindrical pore
structure [23]. Although both of their pore structures are well ordered, 1D and 2D arrangement impose
gas diffusion difficulty, hence the problem of diffusion resistance in the pore framework still exist.
These will then lead to low adsorption capacity and low adsorption rate. Therefore, the research gap is
that it is still remain challenge to search for an alternative CO\textsubscript{2} adsorbent with minimum diffusion
resistance. With this, one of the possible solutions is by proposing the utilization of Michigan State
University (MSU), especially MSU-2 as an alternative CO\textsubscript{2} adsorbent. This is due to it has 3D
wormhole-like pore structure which is highly interconnected. This excellent mesopores connectivity
possessed by MSU-2 minimizes the diffusion resistance of CO\textsubscript{2} through the MSU-2 pore structure and
thus contributes to high diffusion in CO\textsubscript{2} adsorption process [24]. Besides, this characteristic also
enhance stability in hydrothermal conditions and ease the molecular diffusion within the pore
channels. Hence, with these properties, MSU-2 is a promising candidate for CO\textsubscript{2} adsorption with its
performance can be further enhanced by post modification on MSU-2.

In this paper, the synthesis of MSU-2 by using TEOS and Triton X-100 under 55 °C for 48 hours
were reported. TEOS acts as the silica precursor which is the source of silica whereas Triton X-100
acts as the surfactant which is also being called as template. The main function of surfactant is to
reduce the interfacial tension at the boundary of the interface when it is mixed with chemicals which
are not miscible with it. Besides, the function of the surfactant in the mixture is to form a micelles bulk
in order to react with silica precursor to produce MSU-2. The method used for synthesized is solution
precipitation method. The synthesized MSU-2 was characterized for yield, morphology, crystallinity, functional groups, thermal and pore characteristics in order to observe its reproducibility.

2. Experimental

2.1. Materials
Tetraethylorthosilicate (TEOS, MW = 208.33, density = 0.934 g/mL) and hydrochloric acid fuming 37% (HCl, EMSURE®, ACS, ISO, Reag. Ph Eur) were purchased from Merck. Octylphenol ethoxylate ethers Triton X-100 (density at 20 ºC = 1.065 g/mL) was obtained from Sigma Aldrich. Sodium fluoride 98.5% (NaF, MW = 41.99) was purchased from R&M Chemicals.

2.2. Synthesis of MSU-2
MSU-2 was synthesized by solution precipitation method where there are two important processes, which include self-assembly of silica-surfactant and silica condensation in order to make up a colloidal solution [25, 26, 27]. TEOS was mixed into 100 mL of 0.02 M Triton X-100 solution with a TEOS/Triton X-100 molar ratio of 8 and the mixture was stirred at room temperature. 0.25 M of HCl was added dropwise to the mixture of TEOS and Triton X-100 until the pH became 2. The resulting solution was then sonicated for 30 minutes at room temperature, stirred moderately for 5 hours and aged without stirring for 10 hours for the formation of a clear solution. NaF powder was added slowly under vigorous stirring to the resulting solution with a NaF/TEOS molar ratio of 4/1 for the silica condensation to occur. Then, the beaker was placed into an oven at 55 ºC for 48 hours in a static condition to allow the precipitation of the product. The product synthesized was filtered off, washed with deionized water and dried in the oven at 100 ºC for 4 hours. The framework-bound Triton X-100 was then removed by calcination in air at firstly 200 ºC for 6 hours and followed by 620 ºC for 6 hours.

2.3. Characterization
Field Emission Scanning Electron Microscope (FESEM, Zeiss Supra55 VP) operated under an acceleration voltage of 5.00 kV was used to investigate the size and morphology of the MSU-2. Before placing on the sample holder for FESEM analysis, MSU-2 was firstly coated with gold using a sputter coater in vacuum. An integrated feature of FESEM which called Energy Dispersion X-Ray (EDX) Spectrometry (Oxford Instruments INCA x-act) was used to identify the elemental composition of MSU-2. The crystallographic structure of the MSU-2 was studied by using high resolution X-ray diffraction, XRD (X'Pert3 Powder and Empyrean, PANalytical) diffractometer. The XRD diffraction patterns were obtained by using Cu Kα X-ray radiation at room temperature. Fourier Transform Infrared Spectroscopy (Thermo Scientific Nicolet iS50 FT-IR) was used to determine the surface functional groups and chemical bonding of the MSU-2 by using the KBr wafer technique. The scans conducted were from 4000 cm⁻¹ to 400 cm⁻¹ to obtain the adsorption band. Brunauer-Emmett-Teller (BET, Belsorp Mini-II) was used to study the pore characteristic of the MSU-2 which include pore size, specific surface area and total pore volume by N₂ adsorption/desorption at -196 ºC. Prior to the BET measurement, the MSU-2 was degassed under vacuum condition at 200 ºC for 15 hours in order to remove the moisture and the pre-adsorbed gases on the surface of the samples. Barrett-Joyner-Halenda (BJH) method was used to obtain the pore size distribution of MSU-2. Besides, the CO₂ adsorption capacity of MSU-2 also can obtained from BET by CO₂ adsorption/desorption at 25 ºC.

3. Results and discussion

3.1. Yield of MSU-2
A total of four MSU-2 samples were synthesized under the operating temperature of 55 ºC for 48 hours. Table 1 summarizes the yield of the four samples. The percentage yield of MSU-2 samples
were consistent with an average percentage yield of 16.66% ± 1.70%. This indicate that the MSU-2 powder are reproducible.

Table 1. Percentage yield of the MSU-2

| Sample | Percentage Yield (%) |
|--------|----------------------|
| 1      | 16.73                |
| 2      | 17.23                |
| 3      | 16.65                |
| 4      | 16.04                |

3.2. Morphology analysis
Figure 1 shows the FESEM image of the MSU-2.

![FESEM image of the MSU-2](image)

From Figure 1, the synthesized MSU-2 possesses uniform monodisperse microspherical morphology which is in good agreement with the results reported by Beaudet, Hossain & Mercier [25], and by Boissiere et al. and Perez-Quintanilla et al. [26]. This morphology is due to the hydrolysis conditions used during the synthesis process which is at synthesis temperature of 55 ºC. In the beginning of the procedures, Triton X-100 was added as the surfactant and spherical micelles started to form. The silica precursor, which is TEOS was then added and acts as a co-structure-directing agent for the formation of spherical structure [28]. Besides, the acidic TEOS/Triton X-100 solution used, which is at pH 2 plays an important role on the microstructure formation of the MSU-2 synthesized. In order to synthesize MSU-2, polymerization of silica precursor takes place via hydrolysis reaction followed by condensation reaction. During hydrolysis process, the alkoxide groups (OR) are replaced by hydroxyl groups (OH) to produce silanol groups (≡Si–OH). Subsequently, during condensation process, siloxane bonds (≡Si–O–Si≡) are formed between silanol groups. The pH value of the mixture plays an important role in controlling the polymerization rate of alkoxysilanes [28]. In neutral condition, the synthesis of MSU-2 is due to the H-bonding occurs between TEOS and Triton X-100. However, in acidic condition, due to protonation, both TEOS and the surface of micelles become positively charged. Therefore, besides H-bonding, an additional electrostatic interaction takes place between TEOS and Triton X-100 when the pH is acidic [29]. Furthermore, acidic condition promotes rapid hydrolysis reaction but causes slow condensation reaction. This is due to the proton has the ability to withdraw the electron from the silicon, thus making it more electrophilic [28]. This leads to the formation of oligomers which can remain stable for a long time and thus resulting in MSU-2 with
definite particle morphology [29]. The mean diameter of the synthesized MSU-2 is approximately 5 μm.

Besides, Table 2 shows the weight composition of carbon (C), oxygen (O) and silica (Si) elements in the MSU-2 synthesized obtained from the EDX analysis.

Table 2. Weight composition of C, O and Si mapped in EDX for the MSU-2

| Element | Weight Composition (wt %) |
|---------|--------------------------|
| C       | 22.31                    |
| O       | 51.12                    |
| Si      | 26.58                    |
| Total   | 100.00                   |

3.3. Crystallinity

The XRD diffraction pattern of the MSU-2 is shown in Figure 2. It exhibits a well-resolved pattern at low 2θ value of 2.14° that is in agreement with the XRD pattern reported in the literature [25]. MSU-2 shows single narrow XRD diffraction peak, which can be assigned to (100) crystal face diffraction. The results obtained show that the MSU-2 was successfully synthesized under the operating temperature of 55 ºC through the solution precipitation method for 48 hours. The XRD diffraction pattern is displaying a high intensity and narrow peak which indicates that the MSU-2 synthesized in this current work possesses high crystallinity and also indicates that the mesostructure frameworks of the MSU-2 produced are highly ordered [25]. This result can be interpreted as MSU-2 possesses a three-dimensional, wormhole-like structure.

Figure 2. XRD diffraction pattern of the MSU-2
3.4. Functional groups

Figure 3 shows the FTIR spectrum of the MSU-2.

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

**Figure 3.** FTIR spectrum of the MSU-2

The significant features of the FTIR spectrum for the MSU-2 include a large broad band between 3600 cm\(^{-1}\) and 3250 cm\(^{-1}\), and a few peaks at wavenumber of 1630 cm\(^{-1}\), 1100 cm\(^{-1}\) and 800 cm\(^{-1}\), which is similar to the FTIR spectrum reported in the previous study [26].

According to the FTIR spectrum shown in Figure 3, the O-H stretching of the surface silanol groups and the remaining adsorbed water molecules was characterized by the large broad band between 3600 cm\(^{-1}\) and 3250 cm\(^{-1}\) with a peak at 3467 cm\(^{-1}\). Besides, the broad strong peak centred at 1100 cm\(^{-1}\) was attributed to the siloxane (-Si-O-Si-) band whereas the Si-O bond stretching of the silanol groups was represented by the peaks exhibited at 800 cm\(^{-1}\). Furthermore, the deformation vibrations of adsorbed water molecules was illustrated by the absorption peak exhibited at 1630 cm\(^{-1}\).

3.5. Pore characteristics of the MSU-2

The pore characteristics of the MSU-2 obtained from BET measurement were shown in Table 3.

| BET specific surface area, \(S_{\text{BET}}\) (m\(^2\)/g) | Total pore volume, \(V_p\) (cm\(^3\)/g) | Pore size, \(d_p\) (nm) | Reference |
|---|---|---|---|
| 964 | 0.98 | 4.1 | This study |
| 1110 | 1.00 | 4.1 | [25] |

The analyzed BET specific surface area \(S_{\text{BET}}\), total pore volume \(V_p\) and pore size \(d_p\) of the MSU-2 were 964 m\(^2\)/g, 0.98 cm\(^3\)/g and 4.1 nm, respectively. These results of pore characteristic of the MSU-2 are comparable with the data obtained from the past researchers work [25]. However, current paper was able to synthesize smaller particle size of MSU-2, which is around 3.0 µm as compared to the results reported by previous literature [25].

Besides, the nitrogen adsorption/desorption isotherms of the MSU-2 was plotted as shown in Figure 4. By referring to the IUPAC classification of adsorption isotherms, the \(N_2\) adsorption isotherm of the MSU-2 synthesized shown in Figure 4 resembles the Type IV adsorption isotherm and is similar to the results reported by Perez-Quintanilla, Sanchez, Hierro, Fajardo, and Sierra [30]. Besides, there was an increase in the amount of nitrogen adsorbed on the MSU-2 around relative pressure of
approximately 0.46 to 0.66. This indicates that within the uniform mesoporous structure, there is the occurrence of capillary condensation [26]. This Type IV adsorption isotherm shows that the MSU-2 sample consist of mesopore [31]. The framework-confined mesoporosity also can be indicated by the H2(b) hysteresis loop present in the N2 adsorption/desorption isotherm. This type of loop is usually associated with pore blocking [32]. The mesopore of the MSU-2 sample is further confirmed by the pore size of 4.1 nm obtained for MSU-2 in the current study.

Next, BJH pore size distribution of the MSU-2 was also plotted as shown in Figure 5. As shown in Figure 5, the BJH pore size distribution of the MSU-2 was narrow, indicating that the range of the pore size for the MSU-2 was small. The pore size of the MSU-2 synthesized ranged from 2.4 nm to 4.2 nm, which centre at 3.3 nm. This result has showed that the framework mesoporosity of the the MSU-2 was highly uniform, which is similar to the result obtained by Perez-Quintanilla et al. [26].

Figure 4. Nitrogen adsorption/desorption isotherm of the MSU-2

Figure 5. Barrett-Joyner-Halenda (BJH) pore size distribution of the MSU-2
4. Conclusion
In the current study, MSU-2 has been successfully synthesized via solution precipitation method as verified by FESEM, XRD, FTIR, and BET analysis. The solution precipitation method was a two-step synthesis method which involve hydrolysis and condensation process. The silica precursor and surfactant used were TEOS and Triton X-100, respectively. In current study, the synthesis of MSU-2 was reproducible with consistent yield. FESEM analysis showed that MSU-2 synthesized displayed uniform monodisperse microspherical morphology. Besides, the peaks obtained from XRD analysis showed that the MSU-2 with high crystallinity was successfully synthesized. In addition, all the characteristic bands obtained in FTIR analysis had confirmed that the sample synthesized is MSU-2. Furthermore, BET results showed that the MSU-2 possesses analyzed BET surface area, total pore volume and pore size of 964 m²/g, 0.98 cm³/g and 4.1 nm. The N₂ adsorption isotherm and BJH pore size distribution of the MSU-2 also indicated that the MSU-2 has narrow mesopore size distribution. In conclusion, the high BET surface area of the synthesized MSU-2 shows that MSU-2 is a very potential candidate as a good adsorbent for gases.

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