

Modification of Porous Materials by Saturated Fatty Amine as CO₂ Capturer

Maratun Najiha Abu Tahari, Azizul Hakim, Mohamed Wahab Mohamed Hisham, and Mohd. Ambar Yarmo

Abstract—Silica dioxide (SiO₂) and activated carbon (AC) are the common porous materials being used as CO₂ capturer. In this work, saturated fatty amine (octadecylamine, ODA) was evaluated as a potential source of basic sites for CO₂ capture. Both commercial SiO₂ and AC were used as preliminary supports in order to study the effect of loading. The amine-modified samples were characterized using x-ray diffractometer (XRD), attenuated total reflectance/fourier transform infrared spectroscopy (ATR/FTIR), and Brunauer-Emmet-Teller (BET). Reactivity of solid sorbents towards CO₂ was evaluated using isothermal CO₂ adsorption by BET technique. This study shows that the order of CO₂ adsorption capacity by SiO₂ type adsorbents are 25 ODA/SiO₂ > SiO₂ > 15 ODA/SiO₂ > 5 ODA/SiO₂ whereas AC > 5 ODA/AC > 15 ODA/AC > 25 ODA/AC for AC type adsorbents. However, the promotion of ODA compounds on the AC was not improving the CO₂ uptake significantly due to pore blockage and drastic reduction in microporous surface area and pore volume of AC.

Index Terms—Adsorption, amine impregnation, CO₂ capture.

I. INTRODUCTION

Carbon dioxide is one of green house gases, known as main anthropogenic contributors to climate change which has drawn significant attention to researchers in looking to develop strategies for reducing the CO₂ emissions and concentration. Increasing concentration of CO₂ contributes to global warming and climate changes that give critical effects to environment, infrastructures and human. There were several works reported in reducing CO₂ concentration by capture and separate the CO₂ gas. There are different unit operations such as liquid absorption [1], [2], solid adsorption [3]-[5], cryogenic techniques [6], and selective diffusion through polymer, metallic or ceramic membranes [7].

The development of solid sorbents has focused on the modification of solid surfaces with usually having amine compounds. The solid sorbents should have a porous structure to be filled with amine compounds to enable the retention of CO₂ molecules. The commonly used porous solid

supports are silica fume [8], zeolite [9], mesoporous silica [2], [7], [10], high-carbon fly ash [11], activated carbon (AC) [12], and hydrotalcite [13]. However, application of solid sorbents together with octadecylamine (ODA) as CO₂ capturer is still limited. Selected porous materials for this study are silica dioxide (SiO₂) and activated carbon (AC). SiO₂ and AC surfaces will be modified with ODA compounds by impregnation method to develop high dispersion of amine and thus will give better CO₂ adsorption performance. Hence, the objective of this study is to characterize and explore the effect of ODA compounds supported on the surface of SiO₂ and AC towards CO₂ adsorption capacity.

II. PROCEDURES AND METHODS

Silica-amine adsorbent was prepared by use of wet impregnation. Firstly, commercial silica dioxide, SiO₂ received from Fluka was calcined at 400 °C for 4 hours in air before impregnated with octadecylamine, ODA (ALDRICH®, 97%). Chloroform, CHCl₃ (SYSTEM®, 99-99.4%) was added to dissolve the ODA. After stirring for 4 hours in a close system, the mixture was rested for 1 hour before sonicate (S 4000-010 Sonicator 4000). The mixture was sonicated for 10 minutes, 10 amplitudes, 3 W and 1600 J. Evaporation of the used solvent was done in air for overnight and followed by drying at 65 °C for 1 hour. Total amount of each type of adsorbent is 5.0 g. The weight percent (wt %) of guest was determined by the amount of amine added per total weight of synthesized adsorbent which is 5.0 g. Example, in the making of 5 wt % ODA/SiO₂, 0.25 g of octadecylamine, ODA (aldrich®, 97 %) was dissolved in 20.0 g of CHCl₃ and 4.75 g SiO₂ added for further 4 hours stirring. This method was repeated for production of carbon-amine adsorbents by replacing the SiO₂ with activated carbon, AC (Fluka). Each porous material was loaded with three different weight percent (wt %) of ODA which are 5, 15 and 25. The modified SiO₂ and unmodified SiO₂ were denoted as 5 ODA/SiO₂, 15 ODA/SiO₂, 25 ODA/SiO₂ and SiO₂ whereas 5 ODA/AC, 15 ODA/AC, 25 ODA/AC and AC for modified and unmodified AC.

The adsorbents were characterized by FTIR (Perkin Elmer Precisely FTIR Spectrometer), BET (Micromeritics ASAP 2010) and XRD (Bruker AXS D8 Advance). FTIR supported with attenuated total reflectance (ATR) was used to measure functional groups of organic and inorganic compounds without further preparation by using infrared light. Adsorbents were examined on Perkin Elmer Precisely FTIR Spectrometer under pressure on the diamond ATR top plate

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The authors are with the School of Chemical Science and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia (e-mail: maratunnajiha@gmail.com, azizulhakim2442@gmail.com, hisham5011@gmail.com, and ambar@ukm.edu.my).

and scanned from 4000 to 650 cm^{-1} .

The XRD diffraction patterns obtained using a Bruker AXS D8 Advance type with x-ray radiation source of Cu K α (40 kV, 40 mA) to record the 2θ diffraction angle from 3° - 60° . The synthesized adsorbents were analyzed without further treatment.

The Brunauer-Emmet-Teller (BET) surface areas, pores width and pores volume of the adsorbents were measured from adsorption isotherm. These physical characterizations were carried out by N_2 adsorption-desorption at 77 K (liquid nitrogen) using a Micromeritics ASAP 2010 instrument. Samples were degassed at 75°C for 4 hours before analysis.

CO_2 adsorption capacity was measured by BET. 0.3 g of adsorbent was loaded into special round bottom glass and placed into BET port for degassing under N_2 atmosphere and heated up to 75°C . This temperature was maintained for about 4 hours in order to remove all gasses and moisture on the surface of adsorbents. After degassing, the adsorbents were analyzed by using CO_2 atmosphere at 25°C .

III. RESULTS AND DISCUSSIONS

A. Characterization of Synthesized Adsorbents

FTIR with attenuated total reflectance is an excellent technique to obtain structural information of the synthesized adsorbents as shown in Fig. 1. IR spectra for SiO_2 , 25 ODA/ SiO_2 and ODA are displayed on Fig 1a. Modified SiO_2 denoted as 25 ODA/ SiO_2 shows vibration of asymmetric (ν_a) and symmetric (ν_s) N-H stretches of primary amine at 3360 and 3325 cm^{-1} in Fig. 1b. These vibration bands have much weaker intensity compared to the bulk ODA and this is due to the small amount of ODA compounds being impregnated on the SiO_2 .

Referring to the Fig. 1c, the main peaks around 2930 and 2850 cm^{-1} are respectively assigned to the asymmetric (ν_a) and symmetric (ν_s) $-\text{CH}_2-$ stretching of ODA alkyl skeleton [14]. Besides, much weaker bands at 3000 and 2875 cm^{-1} are corresponding to vibrations of asymmetric (ν_a) and symmetric (ν_s) $-\text{CH}_3$ stretches end groups. These bands are very close to those of $-\text{CH}_3$ absorption bands as stated by [14]. A shoulder roughly at 2895 cm^{-1} is due to Fermi resonance between the symmetric $-\text{CH}_2-$ stretching and the overtones of the methylene scissoring [15].

Moreover, methylene rocking vibration on Fig. 1d appears as sharp and strong band at 718 cm^{-1} could be attributed to both ODA and modified SiO_2 . However, an out-of-plane (oop) N-H bending vibration of 25 ODA/ SiO_2 shows as broad band instead of sharp band as in ODA spectrum. This is due to overlapping of N-H (oop) with Si-O-Si symmetric stretching (ν_s) of SiO_2 . The C-N stretching absorption occurs at 1160 cm^{-1} as strong band with lower intensity in ODA spectrum. The presence of this group in the modified SiO_2 sample causes the broad absorption band around 1250 - 860 cm^{-1} became intense. This is owing to the overlapping of the C-N stretching absorption with Si-O-Si (ν_s). The existence of $-\text{CH}_2$ deformation of alkyl chain structural arrangement at 1473 and 1465 cm^{-1} are further confirm the presence of dispersed ODA on SiO_2 surfaces [16]. A strong and broad band at 1567 cm^{-1} is associated to $-\text{NH}_2$ bending vibration of

primary amine [17]. Besides, two absorption bands of asymmetric and symmetric deformation of $-\text{NH}^{3+}$ exist at 1643 and 1485 cm^{-1} on the ODA spectrum.

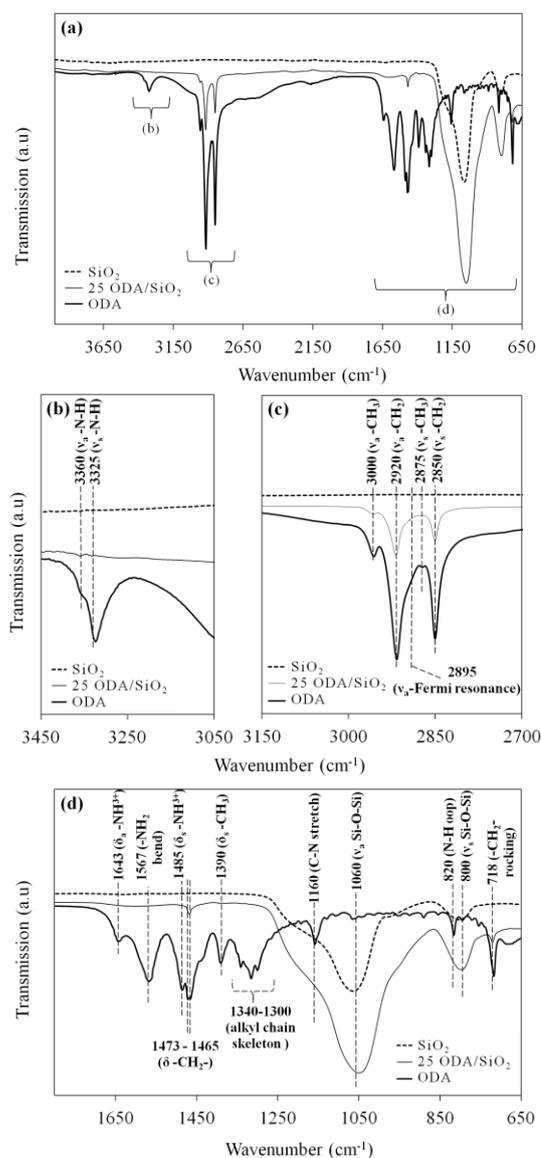


Fig. 1. ATR/FTIR spectra of ODA, SiO_2 , and 25 ODA/ SiO_2 in (a) overall spectra, (b) at range 3450 - 3050 cm^{-1} , (c) 3150 - 2700 cm^{-1} , and (d) 1800 - 650 cm^{-1} .

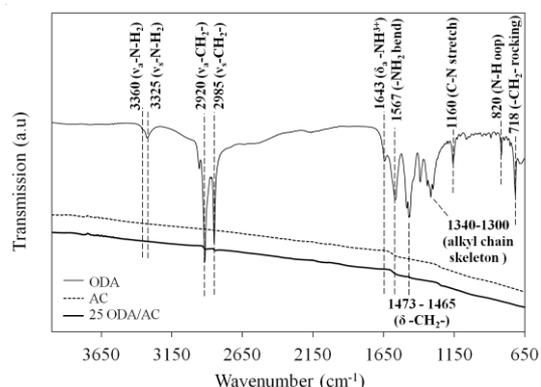


Fig. 2. ATR/FTIR spectra of ODA, AC, and 25 ODA/AC in the range of 4000 - 650 cm^{-1} .

Fig. 2 shows ATR/FTIR spectra for ODA, AC and 25 ODA/AC. There is no peak detected on the AC spectrum,

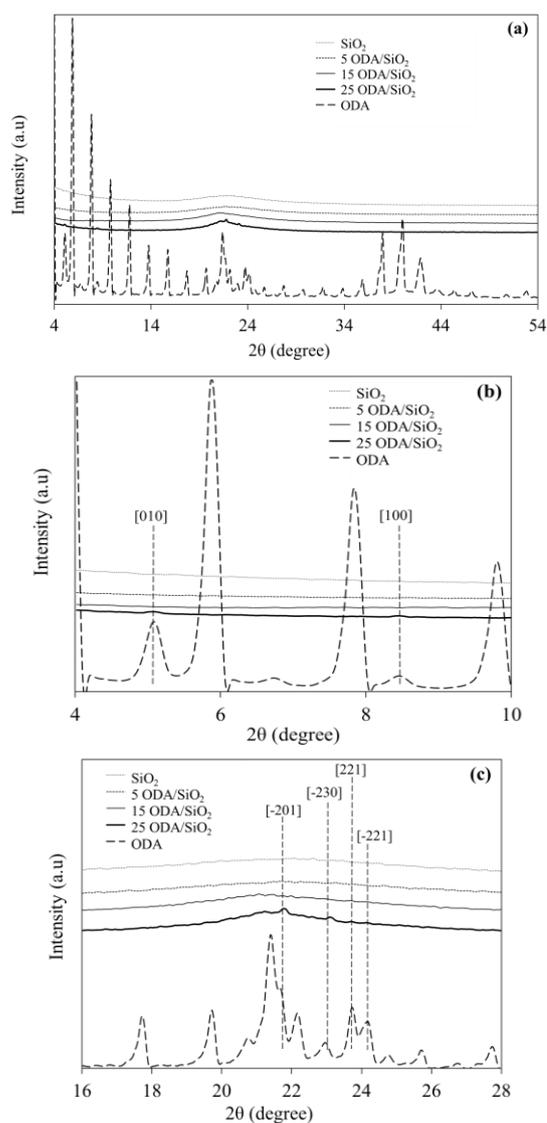
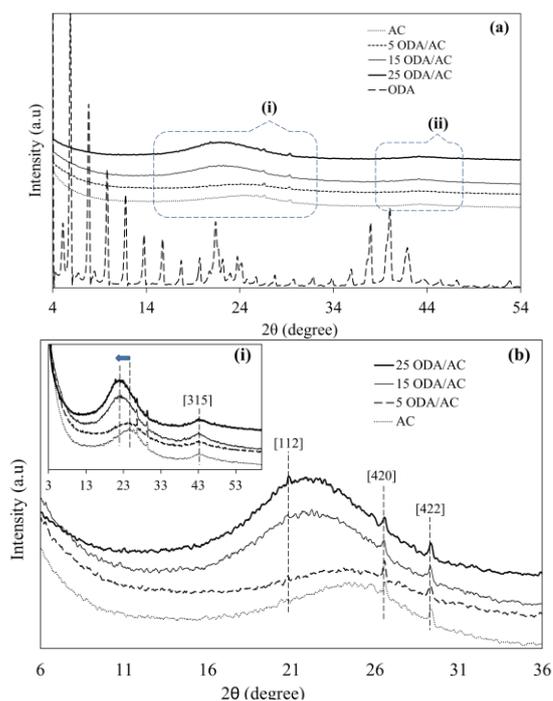
which is explained that the AC material is absence from any foreign molecules. In the case of 25 ODA/AC spectrum, asymmetric and symmetric vibrations of $-\text{CH}_2-$ stretching were detected at 2920 and 2850 cm^{-1} associated to the aliphatic of the amines incorporated onto the AC [18]. The presence of these bands referring to the alkyl skeleton of ODA compounds that dispersed on the AC surfaces. Unfortunately, the absorption bands for primary amine around 3360 and 3325 cm^{-1} on the AC were not appeared. It is due to the difficulties in detecting amine groups as the amount of the $-\text{NH}_2$ to the alkyl groups and AC is too small (1:15.8 and 1:16.8). Furthermore, the ability of the ODA compounds to enter the AC pores also contributing to this poor absorption bands detection. Therefore, XRD analysis has been used in order to confirm the presence of ODA on the AC surfaces.

The XRD patterns of SiO_2 type adsorbents are shown in Fig. 3a. The calcined SiO_2 shows a relatively broad diffraction peak at $2\theta = 21.9^\circ$. Addition of ODA on the SiO_2 surfaces, denoted as 5 and 15 ODA/ SiO_2 had little effect on their mesoporous structure as summarized in Table I, as the diffraction patterns remain unchanged after the introduction of small amount amine [19]. However, the broad peak of SiO_2 around $2\theta = 16^\circ - 30^\circ$ become weaker and shrink a bit especially for adsorbent with higher ODA loadings. In the case of 25 wt % ODA loadings, the 2θ range of broad peak become smaller ($18^\circ - 25^\circ$) and the presence of new diffraction peaks with their lattice [010], [100], [-201], [-230], [-221] and [201] in Fig. 3b and 3c proven that the impregnation of ODA on the SiO_2 surfaces was successful. These peaks belong to semi crystalline of existence ODA which is closely similar with JCPDS NO. 062-1586.

 TABLE I: TEXTURAL PROPERTIES OF SiO_2 , AC AND ODA-CONTAINING ADSORBENTS

Adsorbents	Denotes	S_{BET} [m^2/g]	V_t [cm^3/g]	W_A [\AA]	t -plot _{mic} [m^2/g]	t -plot _{ext} [m^2/g]
SiO_2 only	SiO_2	252.4 6	1.11	170.37	20.11	232.3 4
5 wt % ODA/ SiO_2	5 ODA/ SiO_2	211.8 2	0.97	155.8 9	ND	218.2 4
15 wt % ODA/ SiO_2	15 ODA/ SiO_2	137.3 1	0.65	138.2 8	ND	190.3 0
25 wt % ODA/ SiO_2	25 ODA/ SiO_2	114.6 7	0.41	103.2 7	ND	168.8 3
AC only	AC	862.7 2	0.30	40.85	483.2 8	379.4 4
5 wt % ODA/AC	5 ODA/AC	521.1 1	0.25	41.74	228.7 3	292.3 8
15 wt % ODA/AC	15 ODA/AC	266.6 3	0.21	45.92	37.98	228.6 5
25 wt % ODA/AC	25 ODA/AC	36.92	0.11	97.20	ND	48.05

S_{BET} = BET surface area. V_t = Total pore volume. W_A = BJH Adsorption average pore width. t -plot_{mic} = t -plot micropore area. t -plot_{ext} = t -plot external surface area. ND = Not detected.


 Fig. 3. XRD patterns of ODA, SiO_2 , and modified SiO_2 with different range of degree (2θ) as (a) at range $4^\circ - 54^\circ$, (b) $4^\circ - 10^\circ$, and (c) $16^\circ - 28^\circ$.

 Fig. 4. XRD patterns of ODA, AC, and modified AC with different range of degree (2θ) as (a) at range $4^\circ - 54^\circ$, and (b) $6^\circ - 36^\circ$, and (i) $3^\circ - 53^\circ$.

XRD patterns for AC with different weight percent (wt %) of ODA loadings are shown on Fig. 4a. Two broad peaks in the rounded rectangular callouts, i.e. (i) and (ii) centered at $2\theta = 26^\circ$ and 44° were detected on the AC diffractogram which characterizing the carbon structure. Intensity of another two sharp peaks ($2\theta = 26.7^\circ$ and 29.4°) with their lattice around [002] and [422] were reduced with the increasing ODA loadings as illustrated in Fig. 4b. The diffraction peaks intensities can be correlated with the scattering contract between the solid walls and the pores [19]. The higher amount of amine incorporated into the pore channels, the lower the peak intensity will be produced [20], [21]. These proved by the reduction in BET surface area, total pore volume and micropores area of modified AC as summarized in Table I. Additionally, one peak ascribed to ODA compounds that dispersed on the AC also detected at $2\theta = 20.8^\circ$ as revealed in Fig. 4b. Referring to inset in Fig. 4bi, the broad peak of AC centered at 26.7° was shifted to the left and increased intensity for the adsorbents containing 15 and 25 wt % of ODA. These diffraction changes are might be due to the promotion of higher ODA amount and interaction between then ODA and support.

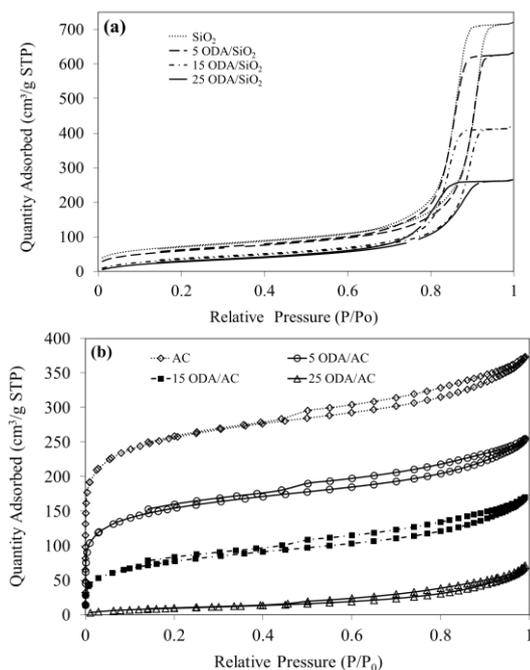


Fig. 5. BET analysis of N_2 adsorption-desorption isotherms of (a) SiO_2 and modified SiO_2 , and (b) AC and modified AC.

B. Nitrogen Adsorption – Desorption Isotherms

The influence of the impregnation on the textural characteristics of the SiO_2 and AC were evaluated through the analysis of the N_2 adsorption-desorption isotherm at 77 K. The N_2 isotherms of SiO_2 and ODA loaded SiO_2 were classified as type IV adsorption-desorption with hysteresis loops as illustrated in Fig. 5a. Type IV isotherm exhibit a hysteresis loop, i.e., the adsorption and desorption isotherms do not overlap over a certain region of external pressure [22]. The isotherms show that SiO_2 and amine loaded SiO_2 are classified as mesopores adsorbents due to the type IV isotherm. Fig. 5b is the N_2 adsorption-desorption isotherm of AC and ODA loaded on AC. The features of these isotherms represent the type I curve according to the IUPAC

classification. The physical properties of modified SiO_2 such as the BET surface area, total pore volume, average pore width, micropore and external pore surface area are given in Table I. This studies show that the physical properties of modified SiO_2 decreases with increases amount of ODA loaded. The modified AC exhibited similar surface properties as modified SiO_2 . Unfortunately, the average pore width of porous AC was contradictive with that trend.

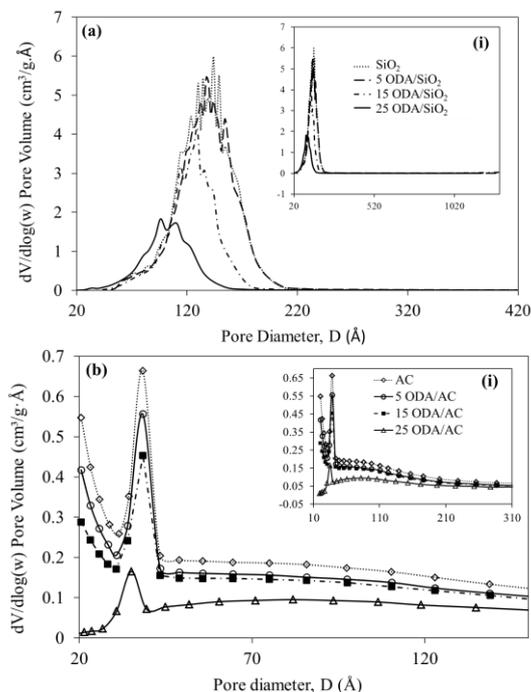


Fig. 6. Pore size distributions of SiO_2 and modified SiO_2 with (a) at range 20 – 420 Å, and (i) 20 – 1270 Å, followed by AC and modified AC with (b) 20 – 80 Å, and (i) 10 – 310 Å. Pore size distribution.

Fig. 6a and 6b show the pore size distributions of modified and unmodified SiO_2 and AC in which their pore diameter was measured in amstrong unit, Å. The mesopores of SiO_2 are mainly centered at 170.37 Å whereas the modified SiO_2 measured around 103.27 up to 155.89 Å. These prove that the decreasing pore size of all modified SiO_2 is due to the present of ODA layer on the pore surfaces. Therefore, SiO_2 adsorbents loaded with higher percentage of ODA (wt %) possess the smaller pore size. Meanwhile, the pore size distributions for AC adsorbents were different from the SiO_2 type adsorbents. The pore size of AC was slightly increased (refer to Table I) with increasing amount of ODA loaded. This proves that the impregnation produces a drastic decrease in the surface area and pore volume of AC due to the blockage of smaller pores by fatty amine as stated by [23]. Thus, the average pore diameter for 25 ODA/AC is referring to the bigger pores of AC that remained after the impregnation.

The cumulative pore volume curves in Fig. 7 show that the total pore volume is about 1.2 cm^3/g and 0.33 cm^3/g for SiO_2 and AC. The pore size distributions show 1 peak for SiO_2 (170.37 Å) and three peaks for AC centered at 97.20 Å, 40.85 Å and 20.00 Å which are corresponding to mesopores and large micropores respectively. This shows that the large micropores and mesopores are establish the porosity characteristics of AC whereas only mesopores could be ascertained the porosity of the SiO_2 .

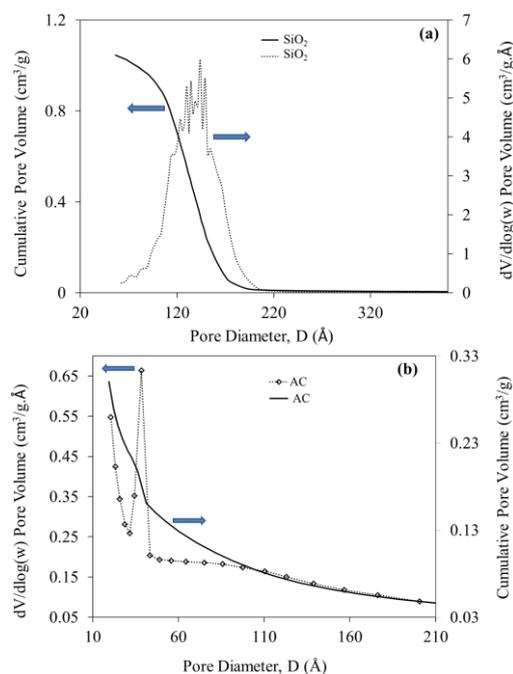


Fig. 7. Cumulative pore volume and pore size distribution of (a) SiO₂ and (b) AC.

C. CO₂ Adsorption Capacity

The effect of the impregnation on the adsorption capacity of CO₂ of each sample was studied by using CO₂ adsorption isotherms at 25 °C and 99.9% CO₂ as revealed in Fig. 8. In particular, silica modified with highest ODA showed high uptake of CO₂ at low pressures (see Fig. 8). The high uptake values and weak temperature dependencies suggested chemisorptions of CO₂ at low pressures [24]. Nonetheless, the isotherms were basically linear at high pressure suggestive of physisorption on both modified and unmodified.

Table II shows the CO₂ adsorption capacity by the raw and modified porous materials. SiO₂ with the highest ODA loading possess the best adsorption performance (2.45 wt % CO₂/adsorbent) compared to other loading. This could be attributed to the presence of 25 wt % ODA which gave lowest pore area, pore volume, pore width of the SiO₂ which provide numerous chemical adsorption sites for CO₂ molecules and causes the formation of stronger CO₂-ODA interaction in the SiO₂ pore surfaces [23]. Modification of SiO₂ surfaces by 5 wt % and 15 wt % ODA were not improving the CO₂ uptake significantly (11.04 and 17.93 mg CO₂/g adsorbent) compared to SiO₂ only (18.46 mg CO₂/g adsorbent). One can be concluded that the 25 wt % ODA/SiO₂ is the best adsorbent among SiO₂ type adsorbents.

The piece of evidence that the raw AC presents the highest CO₂ uptake capacity at room temperature is due to the higher involvement of physisorption. Conversely, this physisorption is restricted in the case of modified sorbents due to the empty large micropores being filled with the ODA molecules which influencing surface properties of AC. As a result, there is drastically decrease of their BET surface area and total pore volume as can be seen in Table I.

Modified AC showed lower CO₂ adsorption capacity because of ODA molecules in the large micropores were difficult to interact with CO₂ since the diameter of pores is too small and the long chain fatty amine producing steric

hindrance effects. Consequently, the CO₂ molecules were difficult to chemically interact with amine groups of the ODA compounds. Taken as a whole, we can summarize that the CO₂ captured by the AC type adsorbents are basically via physisorption on the AC surfaces. Chemisorptions between ODA and CO₂ molecules were not involved since the addition of ODA was significantly reduced the amount of CO₂ captured.

TABLE II: COMPARISON OF CO₂ ADSORPTION CAPACITY WITH VARIOUS ADSORBENTS

Adsorbents	Denotes	Max. CO ₂ Ads. [cm ³ /g]	Max. CO ₂ Ads. [mg/g]	Wt % CO ₂ Ads.
SiO ₂ only	SiO ₂	9.40	18.46	1.85
5 wt % ODA/SiO ₂	5 ODA/SiO ₂	5.62	11.04	1.10
15 wt % ODA/SiO ₂	15 ODA/SiO ₂	9.13	17.93	1.79
25 wt % ODA/SiO ₂	25 ODA/SiO ₂	12.47	24.49	2.45
AC only	AC	58.67	115.24	11.52
5 wt % ODA/AC	5 ODA/AC	21.06	41.37	4.14
15 wt % ODA/AC	15 ODA/AC	11.52	22.63	2.26
25 wt % ODA/AC	25 ODA/AC	6.85	13.46	1.34

Max.CO₂ Ads. = Maximum CO₂ uptake is given per gram adsorbent. Wt % CO₂ Ads. = Weight % CO₂ uptake per gram adsorbent.

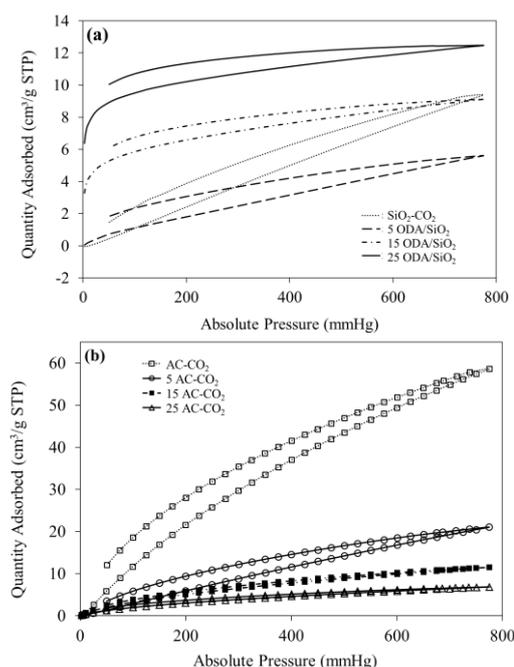


Fig. 8. CO₂ adsorption capacity of (a) SiO₂ and (b) AC with different ODA loading.

IV. CONCLUSION

The addition of 25 wt % ODA on the SiO₂ surfaces affected the CO₂ adsorption capacity by increasing 2.45 % CO₂/g adsorbent compared to SiO₂ which only adsorbed 1.85 % CO₂/g adsorbent and this is might due to chemisorptions between CO₂ and ODA molecules. Meanwhile, ODA loaded on the AC were not improving the CO₂ uptake capacity since the ODA addition causes the BET surface area, pore volume and micropore area of the AC were extremely reduced. It is clearly explain that addition of ODA was obviously constrained the physisorption between CO₂

and AC. Hence, amine compounds with less bulkiness should be used as basic sites for CO₂ capturing, minimizing the steric hindrance effect and reducing the pore blockage of the supporters.

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Maratun Najiha Abu Tahari obtained her BSc. in science (oleochemistry) from Universiti Kebangsaan Malaysia on 2012. She is currently doing master degree in chemistry at School of Chemical Sciences and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia (UKM) from 2012 to 2015.

She experienced works in synthesis biolubricant based on diesters from 2011 to 2012. She was a laboratory demonstrator at UKM before being offered as a part timer chemistry teacher at Pusat Permata Pintar, UKM in 2015. Her research interests are in the field of fatty amine development for CO₂ capturer application.

Maratun Najiha Abu Tahari was awarded with Public Service Department (JPA) Scholarship from Malaysian Government during her bachelor study. She had won silver medal award for poster presentation during International Conference of Postgraduate Education ICPE-6 2014. She also owned several publications related to the CO₂ capturing study.



Azizul Hakim Bin Lahuri is a Ph.D candidate of second year in chemistry from Universiti Kebangsaan Malaysia. He received BSc. in chemistry from Universiti Kebangsaan Malaysia in 2009 and master degree in environment from Universiti Putra Malaysia in 2012. He was born on July 28th 1987 at Pasir Gudang, Johor Bahru, Johor.

His research interests are reduces anthropogenic gas of carbon dioxide (CO₂) by investigate the adsorption and desorption properties. He is conducting the research by using commercial and synthesized solid adsorbents that have potential in CO₂ capturing. His worked experiences in academic nor industry as a level chemistry lecturer in Kolej Teknologi Timur and environmental executive in environmental science (M) Sdn. Bhd., were the advantages in his research. He is currently working as part time tutor in Universiti Putra Malaysia and laboratory tutor in Inti International College Subang.



Mohd. Ambar Yarmo received his BSc in chemistry from Universiti Kebangsaan Malaysia (UKM) and his Ph.D in analytical chemistry from University Of Wales, Cardiff, U.K. He was born at Johor, Malaysia.

His research interests are in conversion of CO₂ to Fuel, bio-ethanol derivatives and biofuel applications, upgrading of natural gas and palm oil to higher added value speciality chemicals using combinatorial technologies and catalysis. He has attended to Japanese scientific exchange programme under JSPS-VCC programme in 1988. He was a visiting scientist at Petronas Research and scientific services in 1995. He has research collaboration with Fritz Haber Institute, Max Planck Society, Berlin, Germany in 2002.

He was the outstanding UKM lecturer in research and teaching in 2000, 2002 and 2005. He is the chairman of Xapp-MNS (Xapp Application Society), Malaysian Nuclear Society. He is a senior member of International Zeolite Association (IZA, USA), Malaysian Analytical Member Society (ANALIST) and Malaysian Nuclear Society.