

Investigation of ordered mesoporous carbon potential as CO₂ adsorbent

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Abstract

In this study, an investigation was carried out in order to determine the potential of ordered mesoporous carbon including high ratio sucrose in its composition as a CO2 adsorbent. Structural characterizations of silica and carbon material were determined by using a scanning electron microscope (SEM), N2 adsorption/desorption and X-ray diffraction (XRD) analysis. CO2 adsorption capacities of developed adsorbent at various temperatures (25 °C, 35 °C and 50 °C) were determined by using a thermogravimetric analyzer under CO₂ flow. The maximum adsorption capacity obtained as approximately 2.27 mmol g CO₂ g⁻¹ adsorbent which was remained almost the same in the desorption process. The first order reaction kinetic model was well fitted with the experimental data. Arrhenius equation was used to calculate activation energy which was obtained as -2.86 kJ mol⁻¹ indicating barrier-less type adsorption. Results showed that ordered mesoporous carbon could be a potential source for an efficient and lowcost adsorbent for CO₂ capture.

Keywords: Ordered mesoporous carbon, CO₂ adsorption/desorption, kinetics.

1. Introduction

In recent years, greenhouse gases have become one of the most concern issues (Huang and Shen, The main factors of increasing greenhouse gas emission are economic and population growth. The environment has been affected negatively by the gas emissions such as increased global temperatures (0.12 °C per decade since 1951), ocean acidification (26% increase in acidity since 1750), and rising average sea levels (0.19 m since 1901) (Lashaki and Sayari, 2018). At the same time, if it doesn't take precaution for this, it is estimated that the global temperature will increase by 1-5 °C in 2050 (Liu et al., 2017a; Villoria-Saez et al., 2016). One of the main components of greenhouse gases is CO2 and it is significantly produced from anthropogenic sources and fossil fuel combustion (Creamer et al., 2016; Youn et al., 2011). Also, it is reported that CO2 contributes 60% of the total global warming caused by all greenhouse gases (Lakhi et al., 2016). For all these reasons, researchers pay attention to CO₂ capture. Solvent absorption, pressure and temperature swing adsorption, membrane separation and cryogenic distillation have also been used to capture CO₂ (Pham et al., 2016; Huang et al., 2018; Yousef et al., 2018). Nevertheless, some CO₂ capture processes have serious problems, such as energy penalties (Padurean et al., 2013), economic and environmental drawbacks (Jing et al., 2014; Ullah et al., 2015) and amine waste during operation (Rochelle, 2009). To overcome such problems, the development of solid adsorbents to CO₂ capture has come to the prominence (Drage et al., 2012). A large variety of adsorbents may be used for CO2 captures, such as zeolites, microporous silicates, aluminum phosphates, carbons, other porous oxides, metal-organic frameworks (MOFs), amine-grafted or impregnated silica (Lashaki and Sayari, 2018; Ma et al., 2018).

Solid adsorbents are very suitible for adsorption of CO₂ owing to high adsorption capacity, fast adsorption rate, and good stability (Liu et al., 2017c). One of the solid adsorbents is porous carbon materials have been attracting lots of awareness as potential large-scale adsorbents because of the many advantages such as well-ordered structure, high surface area, large tunable and uniform mesoporous size, high mechanical strength and good hydrothermal stability at extensive temperature ranges (Zhao et al., 2012; Deng et al., 2012; Singh et al., 2018; Liu et al., 2017b). Carbons must have open pores to be accessible for the CO₂ molecules. Ryoo and co-workers (Ryoo et al., 1999) prepared a new family of mesoporous carbons to represent as the CMK series. These carbon materials have a well-ordered pore structure with the larger surface area (Chandrasekar et al., 2009). All of this properties made CMK series excellent applicant for hydrogen storage, gas separation, electrochemistry, catalysis, and adsorption (Juárez et al., 2017; Yang et al., 2018; Xu et al., 2017). Li and et al. (2017) studied on CO₂ sorption over activated mesoporous carbon (AMC) and metal oxides (CeO₂, CuO, Mn₃O₄ and NiO) modified AMC with different pressure. The best result of CO2 adsorption was 4.66 mmol/g at 273 K and 1 bar by using pure AMC as an adsorbent (Li et al., 2017). Chomiak and et al. (2017) investigated that CO₂ adsorption on microporous granular

activated carbons (GACs) that has been prepared by KOH activation of walnut shell-based carbons. Carbons showed very high CO₂ adsorption, at 273 K up to 7.2 and 18.2 mmol/g under 1 and 30 bar, respectively (Chomiak et al., 2017). Ma et al. (2018) studied CO2 capture with nanoporous carbons materials which was prepared by using a metal-organic framework (MOF-5) as a template. The highest CO₂ adsorption capacity was obtained by nanoporous carbon that carbonized at 900 °C (3.70 mmol/g at 0 °C (1 atm)) (Ma et al., 2018). In the study of Pham et al. (2016) nano-zeolite was evaluated for its potential use in CO₂ capture via temperature swing adsorption. As a result of the experiments, the best adsorption capacity of CO₂ by nano-zeolite was 4.81 mmol/g at a temperature of 20 °C and at a pressure of 1 atm (Pham et al., 2016). Lashaki et al. (2018) examined the impact of the support pore structure on the CO₂ adsorption performance of triaminetethered SBA-15 silica. Large-pore supports exhibited the highest surface density of amine groups (up to 35 μmol/m²) and highest CO2 uptakes (up to 1.88 mmol CO2/g) (Lashaki and Sayari, 2018).

In most of the previous studies, adsorption isotherms of CO₂ adsorbents have been investigated and their adsorption capacities have been examined by various characterizing technics. However, there are few studies on CO₂ adsorption kinetics of such adsorbents. It is critical to examine adsorption kinetics, to better understand the events that occur during the adsorbent-goal material reaction, and also to estimate the time required to complete the adsorption process. Also, information on adsorbent characteristics and reaction kinetics is important for proper adsorbent selection, reactor sizing and process optimization. Thus, in this work we have carried out a comprehensive study both on CO₂ adsorption on to CMK-3 including characterization and kinetics.

In this study, it was carried out for the potential of the ordered mesoporous carbon to be used in carbon dioxide removal. SBA-15 was used as a template to synthesize ordered mesoporous carbon material, CMK-3, for CO₂ capture. Characterization of the adsorbent was carried out with different analytical instruments such as BET (Brunauer–Emmett–Teller), SEM (Scanning Electron Microscope), XRD (X-ray powder diffraction) and TGA (Thermogravimetric Analyzer). In addition, the mechanism of adsorption reaction was investigated and related kinetic parameters were calculated.

2. Materials and methods

2.1. Materials

Sucrose ($C_{12}H_{22}O_{11}$), pluronic 123 (P123), sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) was supplied by Sigma-Aldrich. Tetraethylorthosilicate (TEOS) was purchased from Sigma-Aldrich with 97% purity. Hydrofluoric acid (HF) was supplied by Merck. The purity of hydrofluoric acid is 40%.

2.2. Synthesis of adsorbent

SBA-15 was synthesized according to Zhao *et al.* (1998). For the synthesis of CMK-3, sucrose ($C_{12}H_{22}O_{11}$), sulfuric acid and deionized water were used as a carbon source, acid

solution, and solvent, respectively. CMK-3 was synthesized according to Jun $et\ al.$ (2000). In briefly, 1.25 g sucrose, 5 g of deionized water and 0.14 g H_2SO_4 were stirred until a homogeneous solution was formed. 1 g of SBA-15 was added to this solution and continued to stir. The mixture was placed aged in a furnace at 100 °C for 6 hours and the furnace temperature was increased to 160 °C and maintained for 6 hours. 0.8 g sucrose, 0.08 g H_2SO_4 and 5 g of deionized water were added to this solution and were treated again for the same process (step 2). The resulting material was milled and carbonized in an inert atmosphere at 900 °C for 3 hours at 7 °C/min as a heating rate. After carbonization, the material was washed with HF and ionized water and dried overnight.

2.3. Characterization of adsorbent

The surface area, pore size, pore size distribution and pore volume values of synthesis samples were determined by using nitrogen adsorption/desorption isotherms. Nitrogen adsorption/desorption isotherms were studied by using Quantachrome Corporation, Autosorb.

Silica and carbon structures were investigated to use STOE IPDS II Model X-ray diffraction device. X-ray diffraction pattern of the synthesized sample, the step in the range of 0.02 and 0.025 ($2\theta/s$) CuK α beam having a wavelength of 0.15406 nm were obtained. The characteristic peaks of silica and carbon were determined at 2 θ between 1°-10°. Unit cell parameter (a_0), d100 spacing values (d_{100}) and pore wall thickness (δ) for SBA-15 and CMK-3 were calculated by using equation (1), equation (2) and equation (3), respectively. In equation (3), dp is pore diameter which is calculated by using the BJH method.

$$d_{100} = \frac{n\lambda}{2\sin\theta} \tag{1}$$

$$a_0 = \frac{2d100}{\sqrt{3}}$$
 (2)

$$\delta = a - 0.95 \times dp \tag{3}$$

The morphologies of all the materials were observed using a scanning electron microscopy (SEM). The surfaces of silica structures were plated with a thin layer of gold-palladium before inspection under SEM. The SEM analysis was conducted with JEOL JSM-7001FTTLS LV SEM machine.

2.4. CO₂ adsorption experiments

CO₂ adsorption capacity was determined by DTA-TG Analysis unit (Shimadzu, Japan) (Creamer *et al.*, 2016). A sample weight of carbon approximately 20 mg was placed into an alumina sample pan. The placed sample was dried at 393 K for 1 h in a nitrogen atmosphere to potential volatile components out of the sample and then cooled down to room temperature. Nitrogen gas flow rate was 50 mL/min for both heating and cooling. When the system was at equilibrium, the CO₂ gas stream was switched on at a rate of 50 mL/min for 3 h. The adsorption capacity in percent was computed from the weight change of the sample in the TG adsorption process.

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Total pore volume

(cm3/g)

1.285

1.167

2.5. Kinetic studies

The CO₂ adsorption kinetics onto carbon adsorbent can be described using various kinetic model such as pseudo-first order (Eq. 4) and pseudo-second order (Eq. 5) model (Simonin, 2016).

$$\frac{dq_t}{dt} = k_1 \times (q_e - q_t) \tag{4}$$

$$\frac{dq_t}{dt} = k_2 \times (q_e - q_t)^2 \tag{5}$$

Where q_t (mg g⁻¹) and q_e (mg g⁻¹) are the concentrations of CO₂ adsorption at time t and at equilibrium, respectively, k_1 and k_2 (g mg⁻¹ s⁻¹) is the first and second-order adsorption rate constants.

The activation energy must be minimum value in order to facilitate the adsorption process. The activation energy can be calculated using the Arrhenius equation shown in equation (6) (Shahrom *et al.*, 2018).

$$\ln k = \ln A - \frac{E_o}{RT} \tag{6}$$

BET surface area (m²/g)

753

1360

Table 1. Textural property of SBA-15 and CMK-3

Adsorbents

SBA-15

CMK-3

(a)

According to equation (6), k_1 is the rate constant, A is the temperature impact factor, E_a is the activation energy for adsorption (J/mol), R is the gas constant (8.314 J/mol K) and T is the adsorption temperature in K.

3. Result and discussion

3.1. Characterizations of carbon materials

The ordered pore structure, surface area and morphological structure of the synthesized adsorbent were determined by XRD, nitrogen adsorption/desorption isotherm and SEM, respectively.

3.1.1. N₂ adsorption/desorption

The textural parameters of SBA-15 and CMK-3 samples are investigated using N_2 adsorption/desorption technique. The textural parameters of these samples including the specific surface area, specific pore volume, and average pore size are summarized in Table 1. The specific surface area and total pore volume of the CMK-3 are 1360 m²/g and 1.167 cm³/g, respectively. These results are consistent with the literature (Huang and Shen, 2013; Ghani *et al.*, 2015).

SF method average

pore size (nm)

0.47

0.46

100			CMK-3 Pore	Distrubition, A	
200 -		(b)	1 SBA-15 10	100	1000
		0.00			
300 -		0.05		↓	
400	•	€ 0.10		1	
7 500 - 500 Ages, 21b	CMK-3, Adsorption CMK-3, Desorption	(pgo ₁), p 0.10			
	∘ CMK-3,	0.15	1		
600	• CMK-3,	0.20			
700	◇ SBA-15, Desorption	0.25	-	•	
800	• SBA-15, Adsorption	.			
900 -		\neg			

Figure 1. (a) N₂ adsorption/desorption isotherms. (b) Pore distributions of SBA-15 and CMK-3

Figure 1 shows the N₂ adsorption/desorption isotherms and pore distributions of samples. The synthesized SBA-15 and CMK-3 have been shown to exhibit Type IV isotherm behavior in the BDDT (Brunauer-Deming-Deming-Teller) classification as in the literature (Araujo *et al.*, 2016; Tang *et al.*, 2017). According to Figure 1a, SBA-15 and CMK-3 samples prove to have a mesopore structure. It also shows that in addition to mesoporosity, the structures also have microporosity. From the adsorption/desorption isotherms, the hysteresis loop for the SBA-15 and CMK-3 samples are

observed at partial pressure values in the range of 0.65-0.80 and 0.45-0.6 for P/P_0 , respectively. This type of hysteresis (H1 hysteresis) shows that the structure has a cylindrical structure (Alothman, 2012). The sharp increase and decrease in the hysteresis region is indicative of the narrow pore size distribution in accordance with the pore size distribution shown in Figure 1b. The pore size distributions shown in Figure 1b are performed by the BJH method for mesopore and SF method for micropore. The average pore size of CMK-3 is 2.03 nm.

3.1.2. Small angle X-ray diffraction

BJH method average pore size

(nm)

3.9

2.03

Figure 2 shows the small angle powder XRD patterns of SBA-15 and CMK-3 samples. The SAXS patterns of SBA-15 show the dominant signal peak corresponding to the (100) plane and two small diffraction peaks at higher angles (110) and (200) plane, characteristic of a well-defined hexagonal 2D structures of P6mm symmetry (Sareen *et al.*, 2016; Ochoa-Hernández *et al.*, 2013). CMK-3 was obtained from pore filling of SBA-15 and uniform hexagonal carbon rods were formed. The CMK-3 sample provides a diffractogram with a dominant peak corresponding to the (100) plane, characteristic of these kinds of ordered mesoporous carbons (Joo *et al.*, 2002). This shows that CMK-3 succesfully replicate SBA-15 templates. The patterns for both SBA-15 and CMK-3 indicate that they are ordered mesoporous materials with well-defined pore geometry.

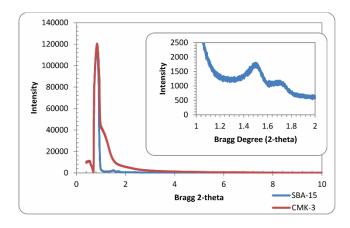


Figure 2. XRD Patterns of SBA-15 and CMK-3

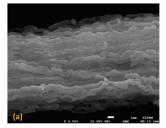
Unit cell parameter (a_0), d100 spacing values (d100) and pore wall thickness (δ) of all materials are given in Table 2. The wall thickness of the SBA-15 and CMK-3 sample was calculated at 8.91 nm and 10.38 nm, respectively. It was found to be consistent with the wall thickness (3-9 nm) specified in the literature (Zhao *et al.*, 1998; Meynen *et al.*, 2009).

Table 2. Unit cell parameter (a_0) , d100 spacing values (d100) and pore wall thickness (δ)

Adsorbents	d (nm)	a (nm)	δ (nm)
SBA-15	10.63	12.61	8.91
CMK-3	10.64	12.28	10.38

3.1.3. SEM images

The morphology of SBA-15 and CMK-3 are illustrated in Figure 3. SBA-15 structures are formed homogeneously. SEM images have proven that CMK-3 is synthesized without change of the surface morphology of SBA-15. SBA-15 and CMK-3 structures have rod-type morphology. The obtained SEM images of SBA-15 and CMK-3 are good agreement with literature (Radhakrishnan *et al.*, 2017; Zhu *et al.*, 2019). EDX analysis of these materials shows that the template has only silica for SBA-15 and the CMK-3 sample has only carbon, confirming the success of the synthesis.



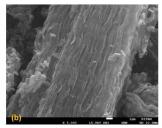


Figure 3 SEM images of (a) SBA-15 and (b) CMK-3

3.2. Kinetic studies of CO_2 adsorption on the carbon material

The TGA data on the CO_2 adsorption capacity of CMK-3 at 25 °C, 35 °C and 50 °C are shown in Figure 4. It has been found that different CO_2 adsorption capacities at various temperatures. Because of CO_2 adsorption is an exothermic reaction, lower temperature is most suitable for CO_2 adsorption. The highest adsorption capacity is obtained at 25 °C as approximately 2.27 mmol CO_2/g adsorbent. This indicates that the higher driving force at 25 °C. As seen from Table 3, the adsorption capacity of the produced adsorbent is within the range of previously reported ones. Thus it can be said that this type of adsorbent have a potential for CO_2 capture processes.

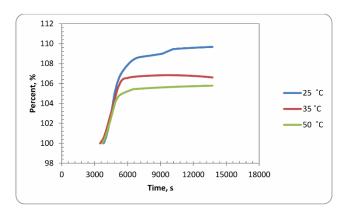


Figure 4. CO₂ adsorption capacity of CMK-3 with various temperatures

Table 3. Comparison of adsorption capacities with literature

Material	Condition	Capacity (mmol/g)	Reference
Activated carbon	273 K and 1 bar	4.66	Li <i>et al.</i> (2017)
PEHA-loaded SBA-16	343 K and 1 atm	2.1	Wu et al. (2018)
KOH activated walnut shell-based carbons	273 K and 1 bar	7.2	Chomiak et al. (2017)
Metal-organic framework	273 K and 1 atm	3.70	Ma et al. (2018)
Nano zeolite	293 K and 1 atm	4.81	Pham et al. (2016)
Nano structured carbon	303 K and 1 atm	1.5	Tiwari <i>et al.</i> (2018)
Amine activated kenaf		2.086	Kamarudin et al. (2018)
Palm activated char	303 K and 1 bar	1.66	Nasri <i>et al.</i> (2014)
Al salt activated cottonwood	298 K and 1 bar	1.61	Creamer et al. (2016)
CMK-3	298 K and 1 atm	2.27	This study

CO₂ adsorption kinetics is determined by using different adsorption kinetic models such as pseudo-first-order and pseudo-second-order. The pseudo-second-order model poorly fits into the kinetic data in which low *R*² values were obtained. For this reason, the pseudo-first-order model can

describe the adsorption process of CO_2 on carbon adsorbent. Kinetic parameters at various temperatures are given in Figure 5a. Pseudo-first order fits the CO_2 adsorption at all temperatures with high regression coefficient values (>0.90). With the increase in

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temperature, decrease in rate constant which indicates an exothermic process. The rate constant at 25 °C, 35 °C and 50 °C are 0.0030103 s⁻¹, 0.0028958 s⁻¹ and 0.0027798 s⁻¹, respectively.

The *Ink* has been plotted versus the inverse of temperature (1/T) to determining the activation energy at Figure 5b. The value of activation energy is found -2.86 kJ/mol. The activation energy is negative because the reaction rate decreases as the temperature increases. CO_2

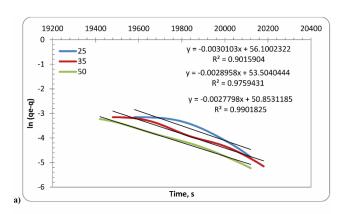
Table 4. Activation energy of CO₂ adsorption for various adsorbent

adsorption with negative activation energy is categorized as barrier-less adsorption. This adsorption process depends on the capture of CO_2 molecules in a potential well.

In this case, the probability of collision of CO_2 molecules decreases as the temperature increases (Liu *et al.*, 2014). The activation energy value of different adsorbents is given in Table 4. The activation energy of CMK-3 for CO_2 adsorption showed similarity with literature.

Adsorbent	Activation energy (kJ/mol)	References
Activated carbon	-2.28	Wei <i>et al.</i> (2017)
Thermally treated graphene nanosheets	-7.21	Kudahi <i>et al.</i> (2017)
Amine functionalized KIT-6*	-6.56	Liu and Yu (2018)
CMK-3	-2.86	In this study

*CO2 concentration (vol %60)



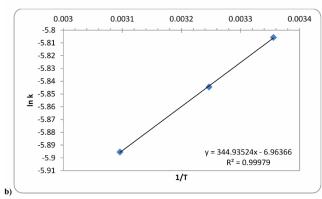


Figure 5. a) Kinetic plot of pseudo first order model with various temperatures. b) Arrhenius plot for activation energy calculation

3.3. CO₂ desorption on the carbon material

Reusability is one of the most important parameters for adsorbents. For this reason, the reusability of CMK-3 for CO₂ adsorption is also studied at 25 °C. Desorption process carried out an inert atmosphere (N₂ flow) after CO₂ adsorption. This adsorption/desorption cycle was repeated two times and distinguished variety are not observed in the CO₂ desorption or adsorption. As seen in Figure 6, similar curves for both fresh and regenerated adsorbents and this indicates that the CO₂ adsorption capacity is the almost the same. Therefore, CMK-3 can be reported as fast, easily, and completely regenerated over multiple cycles without noticeable loss of adsorption capacity.

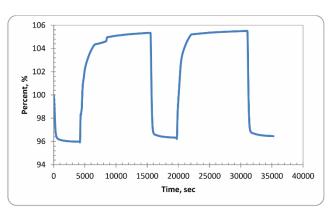


Figure 6. CO₂ adsorption and desorption capacity of CMK-3

4. Conclusion

In this study, CMK-3 has been synthesized and characterized in order to evaluate the potential of ordered mesoporous carbon as a CO2 adsorbent. According to the characterization, CMK-3 was successfully synthesis from pore filling of SBA-15. The adsorption capacity reduced with increase in temperature. The highest adsorption capacity is obtained at 25 °C as approximately 2.27 mmol CO₂/g adsorbent. Kinetic parameters were evaluated with pseudo-first order kinetics where a negative value of activation energy obtained indicating barrier-less type CO₂ capture process. The desorption performance of the adsorbent was almost same the adsorption capacity. Results showed that ordered mesoporous carbon, CMK-3, has a potential in efficient CO₂ adsorbent.

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