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Research Article

A Study of CO2 Adsorption Behaviour and Kinetics on KIT-6

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Abstract

KIT-6 mesoporous silica has been used in catalysis, nano containers, adsorption and drug delivery applications due to its original three dimensional channel network. Typically, KIT-6 is prepared from triblock copolymers as the template directing agents under acidic conditions. In this article, KIT-6 was successfully synthesized via partitioned cooperative self-assembly method. The synthesized sample was characterized using X-ray diffraction (XRD), thermogravimetry/differential thermal analysis (TG/DTA) and N_2 adsorption/desorption. The characterization methods demonstrated that the synthesized sample is ordered KIT-6 mesoporous silica with a high surface area (605.93 m^2 g^{-1}), pore volume (0.58 cm^3 g^{-1}), and good thermal stability. The CO_2 adsorption studies of the sample were performed at different temperatures (25, 75 and 100°C). The maximum adsorption capacity (0.65 mmol g^{-1}) was observed at 25°C adsorption temperature. In addition, it was determined that the adsorption capacity of KIT-6 decreases with increasing adsorption temperature. This study also presents the investigation of the CO_2 adsorption kinetics on KIT-6 using the first order and the second order models. The kinetic data for the CO_2 adsorption on the sample conformed to the second order model. The activation energy (E_a) was calculated as 18.75 kJ mol^{-1} from Arrhenious plot for CO_2 adsorption on KIT-6 mesoporous silica. Moreover, the regenerability and cyclic stability of KIT-6 mesoporous silica was determined using TG/DTA analysis. From the analysis results, it was clearly seen that the ordered mesoporus silica has perfect cyclic stability of 94% after 4 adsorption/desorption cycle, which implies that the synthesized KIT-6 could possibly used as an adsorbent in the CO_2 adsorption.

Keywords: KIT-6, CO_2, Adsorption, Kinetics.

KIT-6 Üzerinde CO_2 Adsorpsiyon Davranışı ve Kinetiği Üzerine Bir Çalışma

Öz

KIT-6 mezogözenekli silika özgün üç boyutlu kanal ağı sayesinde kataliz, nano kaplar, adsorpsiyon ve ilaç taşıyıcı uygulamalarında kullanılmıştır. Tipik olarak KIT-6, asidik koşullar altında şablon yönlendirici ajanlar olarak triblok kopolimerlerinden hazırlanır. Bu makalede, KIT-6 bölümlenmiş kooperatif kendi kendine montaj yöntemi ile başarıyla sentezlenmiştir. Sentezlenen numune X-ışınları difraktometresi (XRD), termogravimetri/diferansiyel termal analiz (TG/DTA) ve N_2 adsorpsiyon/desorpsiyon kullanılarak karakterize edilmiştir. Karakterizasyon yöntemleri, sentezlenen numunenin yüksek yüzey alanlı (605.93 m^2 g^{-1}), gözenek hacimli (0.58 cm^3 g^{-1}) ve iyi termal stabiliteli düzenli KIT-6 mezoporöz silika olduğunu göstermiştir. Numunenin CO_2 adsorpsiyon çalışmaları farklı sıcaklıklarda (25, 75 ve 100°C) gerçekleştirilmiştir. Maksimum adsorpsiyon kapasitesi (0.65 mmol g^{-1}) 25°C adsorpsiyon sıcaklığında gözlenmiştir. Ayrıca, adsorpsiyon sıcaklığının artmasıyla KIT-6'nın adsorpsiyon kapasitesinin azaldığı belirlenmiştir. Bu çalışma aynı zamanda birinci ve ikinci derece modelleri kullanarak KIT-6 üzerindeki CO_2 adsorpsiyon kinetiğinin araştırılmasını sunmaktadır. Numune üzerine CO_2 adsorpsiyonu için kinetik veriler, ikinci dereceden modele uymuştur. Aktivasyon enerjisi (E_a), KIT-6 mezoporöz silika üzerine CO_2 adsorpsiyonu için Arrhenious grafiğinden 18.75 kJ mol^{-1} olarak hesaplanmıştır. Ayrıca, KIT-6 mezoporöz silikanın yenilenebilirliği ve döngüsel stabilitesi TG/DTA analizi kullanılarak belirlenmiştir. Analiz sonuçlarından, düzenli mezoporöz silikanın,

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4 adsorpsiyon/desorpsiyon döngüsünden sonra %94'lük mükemmel döngüsel stabiliteye sahip olduğu açıkça görülmüştür; bu, sentezlenen KIT-6'nın muhtemelen CO_2 adsorpsiyonunda bir adsorban olarak kullanılabileceğine işaret etmektedir.

Anahtar Kelimeler: KIT-6, CO_2, Adsorpsiyon, Kinetik.

1. Introduction

Porous materials has a wide usage in engineering applications due to its high heat resistance, low density of mass and thermal conductivity (Liu & Chen, 2014). According to the IUPAC definition, porous materials are classified into microporous, macroporous and mesoporous materials. The mesoporous materials have pores with diameters between 2 and 50 nm (Schneider, 1995). In recent years, the synthesis and applications of mesoporous materials have attracted the attention of many scientists (Zhao et al., 2012).

A huge family of the mesoporous silica materials has been reported so far including MCM-n (Mobil Composition of Matter), SBA-n (Santa Barbara), MSU (Michigan State University material), FSM-n (Folded Shet Materials), and KIT-n (Korean Advanced Institute of Science and Technology). These mesoporous samples possess high specific surface area, large pore volume, high chemical and thermal stabilities (Kruk et al., 2000; Wang, 2009). Ordered mesoporous silica are used as widely in the field of drug delivery (Yilmaz et al., 2016), catalysis (Lin et al., 2018), optics (Liu et al., 2013), photovoltaics (Adams et al., 2006), membranes (Sakamoto et al., 2007), gas sensing (Palaniappan et al., 2006) and adsorption (Yilmaz, 2017; Yilmaz & Karakas, 2018). They have recently attracted considerable attention of scientists as adsorbents with wide range of CO_2 gas adsorption applications.

The increasing amount of CO_2 in atmosphere due to the combustion of fossil fuels is harmful for human health and environment. For this reason, the capturing and separating technologies of CO_2 are very important. Various solid adsorbents such as zeolite (Hudson et al., 2012; Walton et al., 2006), carbon-based materials (An et al., 2011; Jribi et al., 2017), metal-based adsorbents (Bhatta et al., 2015; Shengping et al., 2011), organic-inorganic hybrids (Shanmugam et al., 2012) and amine solutions (Zhao et al., 2011) have been used for the capturing and separating technologies of CO_2. The main disadvantages of using amine solutions for CO_2 separation on an industrial scale are their high energy requirement for regeneration of amine solutions, being highly corrosive and degradation of absorption solution in the presence of oxygen. (Bello & Idem, 2006; Chakma, 1997; Veawab et al., 1999). A considerable amount of heat is also required to regenerate some zeolite adsorbents (Surblé et al., 2006). Recently, the mesoporous silica materials are used in the CO_2 adsorption and separation technologies due to their superior properties. However, there are a few studies in the literature about the usage of KIT-6 as an adsorbent in the CO_2 adsorption. KIT-6 possesses three dimensional la3d structure, high specific surface area and pore size adjustable from 4-12 nm depending on the synthesis conditions. Due to these properties, it is suitable to be used as an adsorbent in the CO_2 adsorption technologies. Accordingly, Kishor and Ghoshal studied the usage of 3-aminopropyl triethoxysilane (APTES) modified KIT-6 in CO_2 adsorption. They found that the uptake capacity depend on the accessible adsorption sites (amines) for CO_2 interaction (Kishor & Ghoshal, 2015). Tetraethylenepentamine (TEPA) loaded KIT-6 was synthesized by Liu et al. and applied in CO_2 adsorption. They obtained the highest CO_2 adsorption capacity of 2.9 mmol g^{-1} using 50 wt% TEPA loaded KIT-6. (Liu et al., 2010). KIT-6 functionalized with polyethylenimine was prepared for CO_2 separation. It was observed that the CO_2 sorption capacity increased by increasing the temperature (Kishor & Ghoshal, 2016).

In this study, the use of KIT-6 synthesized as an adsorbent in CO_2 adsorption was investigated. CO_2 adsorption capacity of the material was studied at different temperatures using TG/DTA. Also, CO_2 adsorption kinetics of the synthesized sample was carried out by using the first order kinetic model (FOKM) and the second order kinetic model (SOKM).

2. Material and Method

Pluronic P123 block copolymer surfactant and concentrated HCl (37 wt%) were supplied from Sigma-Aldrich. Tetraethyl orthosilicate (TEOS; 98 wt%) and 1-butanol (0.81 g cm^{-3}) were obtained from Merck.

X-ray diffraction (XRD) data was taken by a PANalytical X'Pert-Pro diffractometer with CuKα radiation in the scanning range of $2\theta=0.58–3.6°$. Thermal stability of the sample was investigated in the Perkin Elmer Pyris Diamond TG/DTA under N_2 flow at a heating rate of 10 °C min^{-1}. N_2 physisorption measurement of the sample was carried out at 77 K in Micromeritics ASAP 2020 BET (Brunauer, Emmett, and Teller) equipment. BET surface areas (S_{BET}, $m^2\ g^{-1}$) were calculated using adsorption data with relative equilibrium pressure ranging from $0.06 < P/Po < 0.30$. Pore size distribution of the sample was calculated according to Barrett –Joyner–Halenda (BJH) method using desorption data.

2.1. Preparation of KIT-6

KIT-6 was synthesized according to the partitioned cooperative self-assembly (PCSA) method as reported previously (W. Wang et al., 2014). Briefly, 4.65 g P123 was thoroughly dissolved in a mixture of distilled water and HCl at 35°C and subsequently, 4.65 g n-butanol was introduced in the homogeneous solution and the mixture stirred for 1 h. Then, the desired amount of TEOS was added slowly in the solution and after a while, the remaining amount of TEOS was dropped into the mixture. The final solution was stirred for one day at 35°C and it was introduced to an autoclave and heated at 100°C for one day. Then, the obtained solution was filtered and the resultant white solid product was dried at 50°C. The obtained material was calcined at 550°C for 4 hours to remove P123.

2.2. CO_2 adsorption

CO_2 gas adsorption analysis of the sample was performed in TG/DTA under different temperatures (25, 75 and 100°C). For adsorption process, the sample of mass about 10 mg was taken into an aluminum crucible and heated under a nitrogen from room temperature to 110°C with a heating rate of 10 °C min^{-1}. The sample was waited until the moisture in it was removed. Then, it was cooled to a desired temperature with a cooling rate of 15 °C min^{-1}. When the desired temperature was reached, it was switched to CO_2 and kept for 90 min. CO_2 adsorption capacity of the sample was determined based on the weight increase in the material.

In the adsorption/desorption analysis, the same amount of sample was loaded in the same TG/DTA equipment. Firstly, the material was heated to 110°C under nitrogen with a flow of 60 ml min^{-1} and it was waited until the moisture content was removed. Then, it was cooled to75°C with a cooling rate of 15 °C min^{-1}. The gas was altered to CO_2 and the sample was waited under CO_2 atmosphere for 1 h. To desorption process, the gas was switched to nitrogen and the sample was kept at 75°C. This adsorption/desorption analysis was repeated for 4 times. The temperature of the adsorption/desorption cycle was chosen according to previous works (Xu et al., 2003; Son et al., 2008; Zhao et al., 2013).

3. Results and Discussion

3.1. Characterization

XRD pattern of the synthesized sample is depicted in the Figure 1. It can be seen from the pattern, the diffraction peaks (211), (220), (420), and (332) corresponding characteristic of KIT-6 ordered mesoporous structure (Wang et al., 2014).

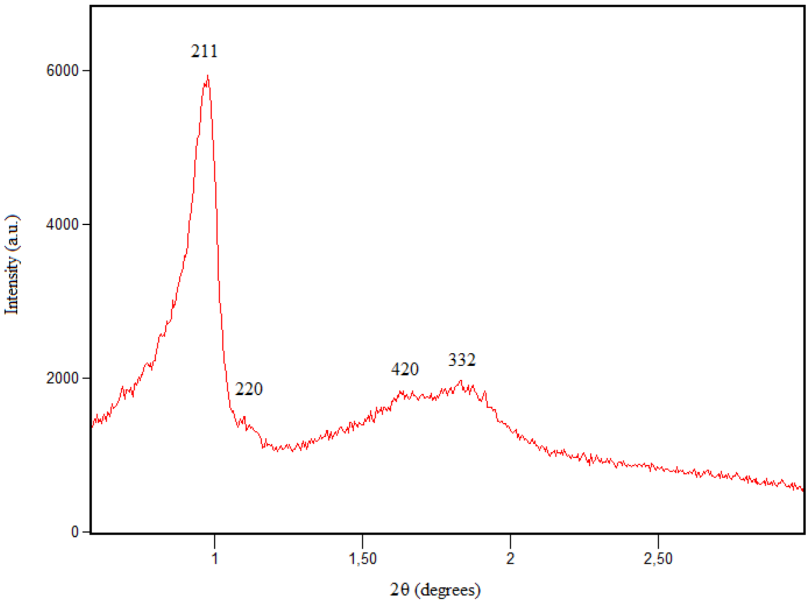


Figure 1. XRD pattern of KIT-6

The thermal behavior of KIT-6 is demonstrated in Figure 2. As it can be observed that a weight loss of 3.5% occurred between 30°C to 100°C, corresponding to removal of moisture. At temperatures above 100°C no weight loss occurred, showing that the synthesized sample has good thermal stability.

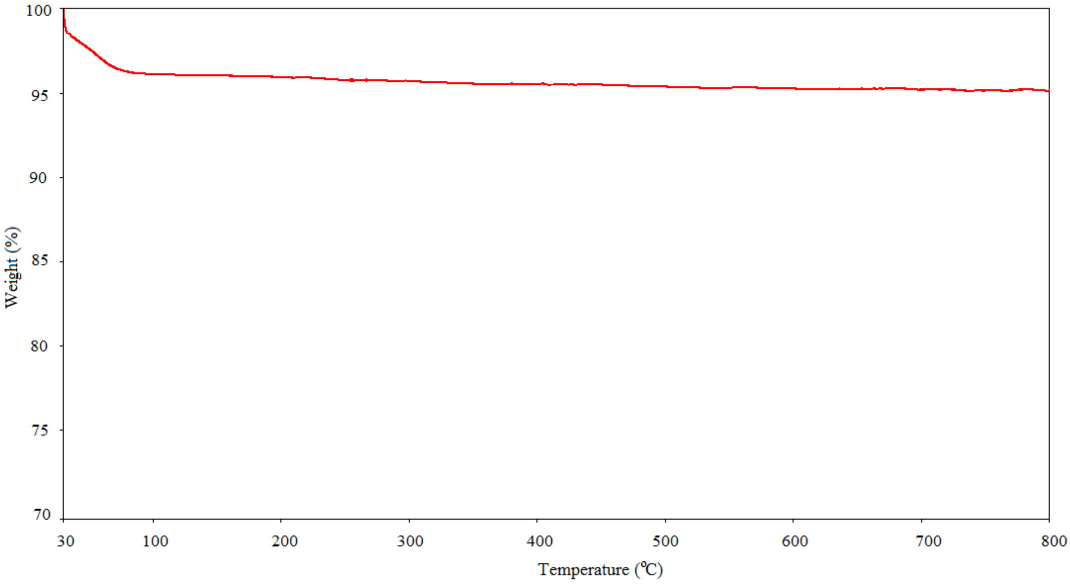


Figure 2. TG curve of KIT-6

Figure 3 illustrates N_2 adsorption/desorption isotherm of KIT-6. It was observed that the quantitiy of adsorption increased rapidly at P/Po between 0.5 and 0.8 as a result of capillary condensation. The isotherm shows the Type IV isotherm which is the characteristic for mesoporous materials and H1hysteresis loop, indicating that large channel-like mesopores were occurred. The specific surface area, pore volume and pore size of the sample were 605.93 m^2 g^{-1}, 0.58 cm^3 g^{-1} and 4.37 nm, respectively.

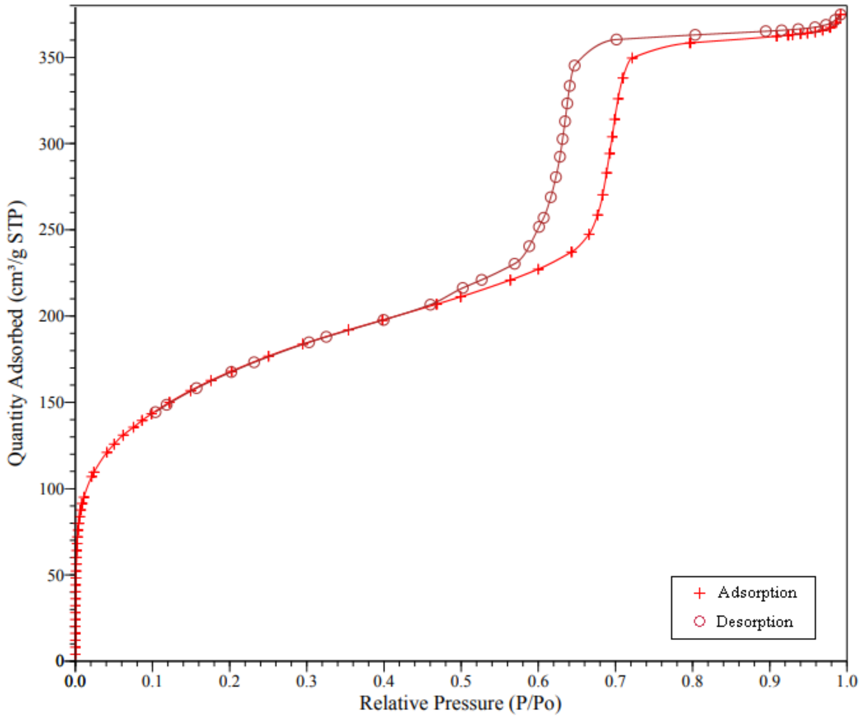


Figure 3. Nitrogen adsorption and desorption isotherms of KIT-6

3.1. CO_2 Adsorption Performance of KIT-6

The CO_2 adsorption behavior of KIT-6 mesoporous silica at different temperature was given in Figure 4. It was seen from the figure, adsorption capacity of the sample decreased by increasing of the temperature is a typical physisorption phenomena. The CO_2 uptake capacities of the sample increased rapidly and then it remained constant after 75 minute for all temperatures. The adsorption capacity of the sample was calculated considering the increase in sample mass in the TG curve. The maximum uptake capacity of KIT-6 was found to be 0.65 mmol g^{-1} at 25°C.

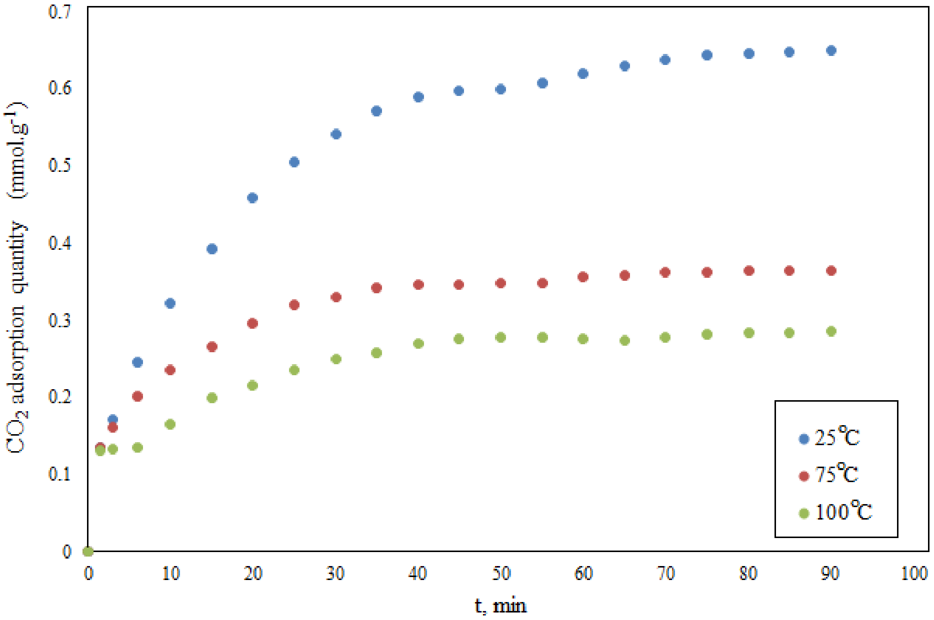


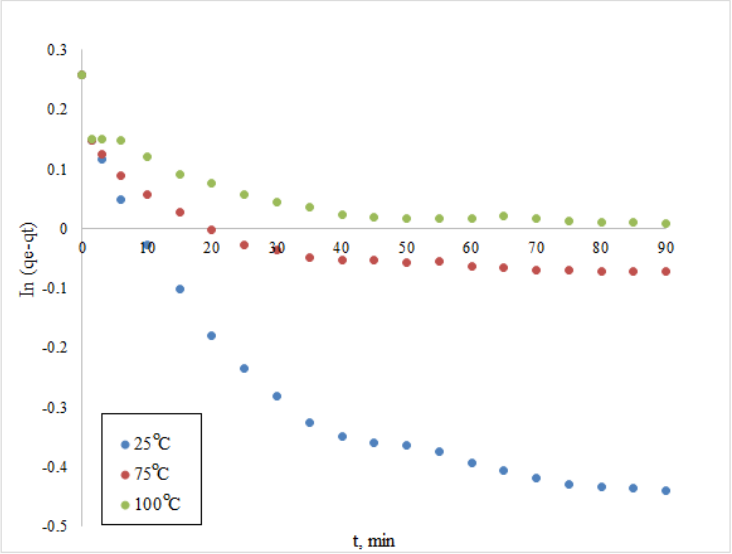
Figure 4. The adsorption capacities of CO_2 on KIT-6 at various temperature

The adsorption kinetics of the sample was investigated by using FOKM and SOKM. The adsorption kinetic models were listed in Table 1. FOKM adsorption rate expression, originally derived by Lagergren, is based on solid phase concentration in the adsorption processes occurring in solid-liquid phase systems. The resulting FOKM expression is shown in Table 1. In the SOKM, the rate-limiting step is chemical sorption that involves the forces generated by exchange or sharing of valence electrons between the adsorbent and the solute. The SOKM expression is given in the Table 1.

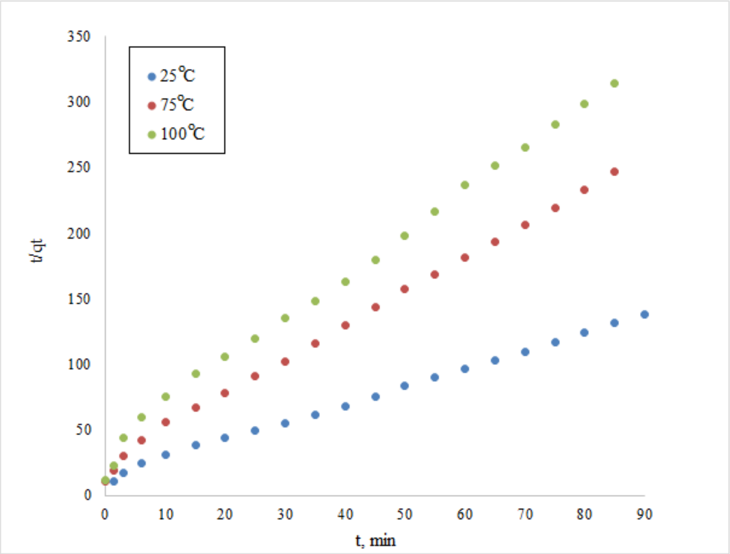
Table 1. Adsorption kinetic models

Model	**Equation**	**Explanations**	**Ref.**
FOKM	$\frac{dq}{dt} = k_f . (q_e - q_t)$	q_e Amount of adsorbed substance at equilibrium (mmol g^{-1}) q_t : Amount of adsorbed substance at *t* (mmol g^{-1}) t: Time (min) k_f : The FOKM rate constant (min^{-1})	(Yilmaz & Karakas, 2018)
SOKM	$\frac{dq}{dt} = k_s . (q_e - q_t)^2$	k_s: The SOKM rate constant (g^{-1} min^{-1}**)**	(Yilmaz & Karakas, 2018)

The plots of FOKM and SOKM for the CO_2 adsorption on KIT-6 are given in Figure 5 and the calculated kinetic parameters of the kinetic models are listed in Table 2. From the table, it is observed that the correlation coefficient for the SOKM was higher than the FOKM. Also, q_e value estimated by the SOKM and the q_e values found experimentally are much closer than the other model. Therefore, it was concluded that the kinetic mechanism of CO_2 adsorption on the synthesized KIT-6 can be better explained by the SOKM.



a)



b)

Figure 5. The kinetic model plots for CO_2 adsorption on KIT-6 a) FOKM and b) SOKM

Table 2. Kinetic model plots for adsorption of CO_2 on KIT-6 a) FOKM and b) SOKM

Model	Parameter	Temperature		
		25°C	75°C	100°C
FOKM	q_e (exp.) q_e k_f R^2	0.643 1.039 0.007 0.809	0.363 1.103 0.003 0.663	0.283 1.151 0.002 0.697
SOKM	q_e k_s R^2	0.730 0.129 0.998	0.386 0.507 0.999	0.305 0.538 0.997

The activation energy is an important factor used to determine the reaction rate. This value was found by the Arrhenius method, is written by the following equation:

$$\ln k = -\frac{E_a}{RT} + \ln A \tag{1}$$

where E_a is activation energy (J mol^{-1}), T is the temperature (K), k is the adsorption rate constant, and A is the frequency factor. Since the SOKM is the best method to define CO_2 adsorption on KIT-6, the activation energy of the adsorption process was found using the adsorption rate constants calculated from this kinetic model. Figure 6 shows that the Arrhenius plot obtained from second order k values calculated for the CO_2 adsorption on KIT-6. The activation energy value was calculated as 18.75 kJ mol^{-1}. The obtained E_a value is low due to weak adsorbate−adsorbent interaction and it is in the range of physisorption phenomena (Singh &Kumar, 2016). This result is compatible with the findings obtained from CO_2 adsorption capacity analysis.

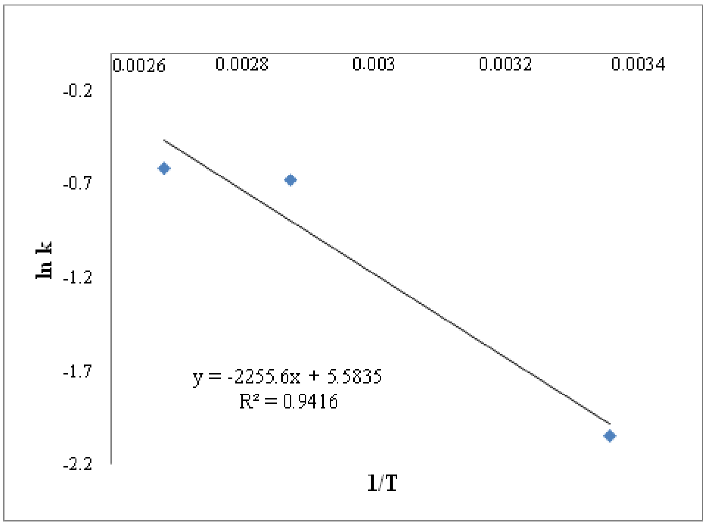


Figure 6. Arrhenius plot for the activation energy of CO_2 adsorption on KIT-6

In order to investigate the regenerability and cyclic stability of KIT-6, multistage CO_2 adsorption–desorption cycles were performed by DTA/TG at 75°C. The adsorption–desorption cycles were repeated four times. The CO_2 uptake capacity of the material is around 0.34 mmol g^{-1} for each cycle, demonstrating excellent cyclic stability (Figure 7).

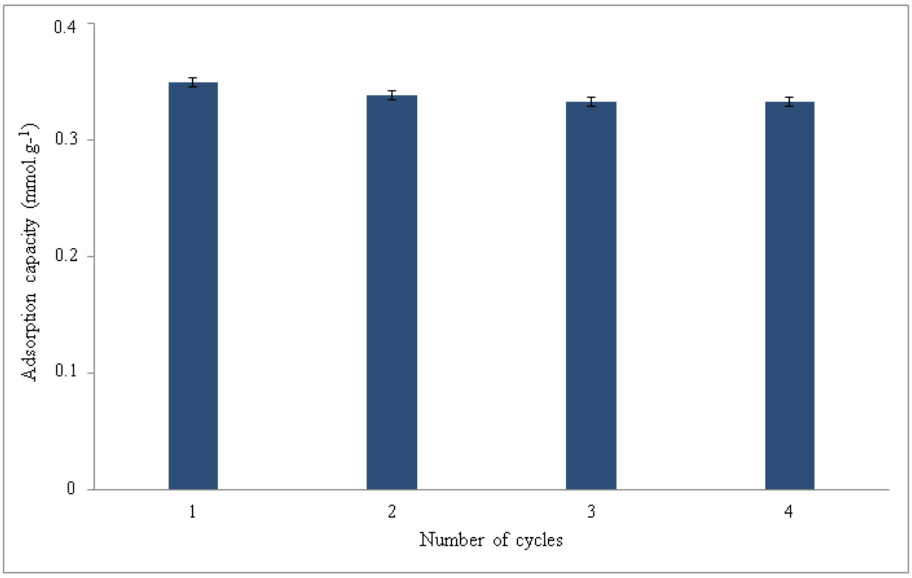


Figure 7. The cyclic adsorption capacity of the material

4. Conclusions and Recommendations

This study investigates the synthesis and usage of KIT-6 mesoporous silica as an adsorbent for CO_2 adsorption. The characterization results showed that ordered KIT-6 mesoporous silica was synthesized successfully. The surface area and pore volume of the material were found as 605.93 $m^2\ g^{-1}$ and 0.58 $cm^3\ g^{-1}$, respectively. The CO_2 adsorption results showed that adsorption capacity of KIT-6 decreased by increasing of the adsorption temperature is a typical physisorption phenomena. Its maximum adsorption capacity reaches 0.65 mmol g^{-1} at 25°C adsorption temperature. Also, CO_2 adsorption kinetics of KIT-6 was studied using the FOKM and SOKM. It was found that the SOKM describes the CO_2 adsorption on KIT-6. The E_a value was found as 18.75 kJ mol^{-1} and it is in the range of physisorption. Four adsorption desorption cycles revealed that KIT-6 exhibits excellent regenerability and cyclic stability, which shows that the synthesized KIT-6 mesoporous silica can be considered potential adsorbent for CO_2 adsorption.

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