

RESEARCH ARTICLE

Activation of zeolite-Y templated carbon with KOH to enhance the CO₂ adsorption capacity

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Graphical abstract



Abstract

This research was aimed to activate zeolite-Y templated carbon (ZTC-Y) with potassium hydroxide (KOH) at variation weight ratios of KOH(g)/KTZ-Y(g) at 0.5, 1, 1.5 and 2, in order to enhance the carbon dioxide (CO₂) adsorption capacity on the material. ZTC-Y was synthesized via impregnation method by carbonization. The activation was performed by heating of impregnated ZTC-Y with KOH at 800°C for 1 hour, followed by acid washing to remove inorganic salt. ZTC-Y before and after activation were characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM) and Brunauer–Emmett–Teller (BET) methods. The surface area and average pore size of activated ZTC-Y were 557.41 m²/g and 2.36 nm, respectively. The CO₂ adsorption capacity was determined by gravimetric method. The result showed that activation of ZTC-Y with KOH could increase CO₂ adsorption capacity on ZTC-Y. CO₂ adsorption capacity was enhanced from 1.07% (wt) to 2.72% (wt) for ZTC-Y after activation with KOH at weight ratio of KOH(g)/ZTC-Y(g) 1.5.

Keywords: Zeolite-Y templated carbon, activation, KOH, CO2 adsorption capacity

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INTRODUCTION

Adsorption using porous materials such as carbon (Zhang et al., 2010) and zeolite (Siriwardane et al, 2001) is an alternative method to reduce CO₂ in air. Although carbon has high surface area, economically wise and good recyclability (Chung et al., 2010; Weitkamp, 2000), the pore structure of carbon is less uniform and porous (Alam and Mokaya, 2011). On the other hand, zeolite exhibits low surface area, but pore structure of zeolite resembles cage like, with molecular size of 0.5-1.2 nm (Kelut et al., 2014). Although zeolite surface pore can be arranged to micropore (<2 nm) or mesoporous (2-50 nm), zeolite surface area is generally lower than carbon (Chung, 2010). The idea was to combine the zeolite and carbon properties by creating zeolite templated carbon (ZTC), which can provide high surface area and uniform pore structure. It has been reported that CO2 adsorption capacity of ZTC-Y was 1.07 % wt at temperature of 30°C and pressure of 1 atm (Gunawan, 2015), indicating that CO2 adsorption capacity still did not achieve the adsorption of CO₂ requirements.

In order to further enhance the CO₂ adsorption capacity of the ZTC, chemical activation of ZTC using KOH is a potential method. Reaction between KOH and carbon produces K_2CO_3 (Im *et al.*, 2008). Then, K_2CO_3 reduction produces potassium and carbon monoxide (CO) gas. The potassium is intercalated between graphitic type layers to make a new form pore. The formed pore size is in accordance with radius of the potassium atom, 0.470 nm (Viswanathan *et al.*, 2009) and it is suitable with CO₂ molecular size, 0.33 nm (Deng *et al.*, 2012). However, there is still no research that reported on the ZTC activation using KOH in order to enhance the CO₂ adsorption capacity.

This research aims to activate the ZTC using KOH to enhance the CO_2 adsorption capacity. The appropriate amount of activator can

improve the adsorption capacity. However, increasing the amount of activator can also reduce CO₂ adsorption capacity. In this study, weight ratio of KOH(g)/ZTC-Y(g) was in accordance with the previous research by Anggraini (2013), who worked on the activation of carbon with K₂CO₃. It is caused by the activation mechanism of carbon with KOH is similar to K₂CO₃, but with a longer step (Ramm *et al.*, 1982; Yang *et al.*, 2006) and refers to several studies that determine the optimum ratio for adsoption capacity, ie. 0.5, 1, 1.5, and 2 (Abechi *et al.*, 2013; Li *et al.*, 2011; Mopoung *et al.*, 2015).

EXPERIMENTAL

Materials

The materials used in this research were natrium aluminate (Sigma-Aldrich, 13404-5KG-R), natrium silicate (Sigma-Aldrich, 338443-3L), sodium hydroxide (99% p.a, Merck, 011-002-00-6), sulfuric acid (98% p.a), hydrogen chloride (37% SAP), hydrogen fluoride (48% p.a), sucrose (98%, Fluka), nitrogen gas (UHP) (99.99% N₂), demineralized water, potassium hydroxide (99.99%, p.a). and carbon dioxide gas (UHP) (99.99% CO₂).

Synthesis of zeolite-Y

Zeolite-Y was synthesized by using the molar ratio of $Na_2O:Al_2O_3:SO_2:H_2O$ at 10.67:1:10:180, followed by three steps, namely as seed gel, feedstock gel and overall gel. Seed gel was prepared by mixing 23.991g DM water, 3.469g NaOH, 1.924g natrium aluminate and 17.518g natrium silicate, then being closed and allowed to stand at room temperature for 24 hours. Feedstock gel was prepared by mixing 157.518g DM water, 0.100g NaOH, 13.851g natrium aluminate, and 107.212g natrium silicate. Overall gel was blended of seed gel and feedstock gel at room temperature for 24 hours, then heated at 100°C for 7 hours. Zeolite was filtered and

washed with DM water until achieving pH under 9. Then, zeolite was dried at 110° C for 12 hours. The synthesized zeolite was then used as a template to syntesize ZTC.

Synthesis of zeolite-Y templated carbon (ZTC-Y)

Fixed mass of 10 g zeolite-Y was blended with 12.5 g sucrose, before being dissolved in 50 mL of 0.35M sulfate acid for 72 hours, follwed by carbonization at temperature of 800°C for 4 hours under nitrogen. Zeolite-Y/carbon composite was soaked in the mixture of HF 5%, HCl 37%, and HF 48% for 1 hour, and washed by deionized water until pH become neutral. ZTC was dried at 120°C for 12 hours and then was activated by KOH.



Fig. 1 Schematic diagram for synthesis of Zeolite-Y Templated Carbon (ZTC-Y) (Nishihata *et al.*, 2009).

Activation of zeolite-Y templated carbon (ZTC-Y) with KOH

KOH and ZTC-Y at weight ratios of 0.5, 1, 1.5 and 2 were dissolved in 20mL water and stirred at \pm 150 rpm for 5 hours. The mixture was filtered and dried at 110°C for 12 hours. Then it was carbonized at 800°C for 1 hour under nitrogen at flow rate of 3°C /min. ZTC-Y was soaked in hydrogen chloride 2 M for 1 hour, then washed with DM water until pH neutral and dried at 110°C for 12 hours. The activation process was referred to previous work (Sevilla *et al.*, 2010).

CO₂ adsorption

 CO_2 adsorption test was performed on ZTC-Y before and after activation. Before adsorption process, sample was degassed at 350°C for 3 hours. CO_2 adsorption was conducted at temperature of 30°C with CO_2 flow rate of 20 mL/s. Data was taken every 5 minutes until constant measurement was achieved. CO_2 adsorption capacity was calculated using Equation (1).

$$\%CO_2 = \frac{(m_t - m_o)}{m_o} \times 100\% \tag{1}$$

RESULTS AND DISCUSSION

Structural characterization of zeolite-Y and zeolite-Y templated carbon (ZTC-Y)

The structural properties of the zeolite-Y and zeolite-Y templated carbon (ZTC-Y) were investigated by XRD and SEM. XRD patterns of zeolite-Y and ZTC-Y were shown in Fig. 2. It could be seen from the Fig. 2(a) that synthesized zeolite-Y showed the peak at $2\theta = -6^{\circ}$. It was indicated for the high crystalinity and corresponded to the zeolite-Y peak in JCPDS standard No. 39-1380. Morever, XRD pattern of synthesized zeolite-Y also showed that there was no other phase except zeolite-Y. Reaction for the zeolite-Y synthesis process could be described in Eq. 2. k and j are the value index for zeolite, which is specific for each type of zeolite. Zeolite-Y in this research has k value of 56 and j value of 250.

ZTC-Y was prepared by impregnating sucrose as a carbon precursor on the zeolite-Y template. Before impregnation process, zeolite-Y was degassed at 200°C (N₂ flow was used to remove gasses and impurities in zeolite-Y pore). As a result, sucrose molecule was easier to enter into the zeolite -Y pores. The sucrose:zeolite-Y ratio in this research was fixed at 12.5:10, which referred to the optimum result from previous work by Kayadoe (2013), who performed ZTC-Y synthesis at various ratios of sucrose:zeolite-Y. In the impregnation

process, sucrose with molecular diameter of 0.7-0.9 nm (Ramm *et al.*, 1982; Gunawan, 2015) would enter the zeolite-Y window pore of 0.74nm (Weitkamp, 2000). The impregnated material was then carbonized at 800°C to get zeolite-Y/carbon composite. During carbonization process, carbon was decomposed in the zeolite-Y pore channel to replicate the zeolite-Y pore structure. Then the template was removed by HF and HCl washings to break the Si-O-Al-O-Si zeolite-Y framework, (Su *et al.*, 2004; Yang *et al.*, 2006). HF and HCl washings were used to break Si-O (Su *et al.*, 2004), and Al-O (Yang *et al.*, 2006), respectively. ZTC-Y was then characterized by XRD and SEM.

$$Na_{2}SiO_{3(s)} + 3H_{2}O_{(1)} \rightarrow Na_{2}SiO_{3(aq)}$$

$$NaAlO_{2(s)} + H_{2}O_{(1)} \rightarrow NaAl(OH)_{4(aq)}$$

$$NaOH_{(aq)} + NaAl(OH)_{4(aq)} + NaSiO_{3(aq)} \xrightarrow{\text{room temperature}}$$

$$[Na_{x}(AlO_{2})_{y}(SiO_{2})_{z}.NaOH.H_{2}O]_{(gel)} \xrightarrow{Hydrothermal at 100^{\circ}C}$$

$$Na_{j}[(AlO_{2})_{j}(SiO_{2})_{192-j}].KH_{2}O_{(s)} \text{ (zeolite chyrstal)}$$

$$Na_{56}[(AlO_{2})_{56}(SiO_{2})_{136}].250H_{2}O_{(s)} \text{ (zeolite-NaY chrystal)} \qquad (2)$$

Zeolite-Y/carbon composite and ZTC-Y were characterized by XRD to see the arrangement of carbon in entering and replicating of zeolite-Y pore as shown in Fig. 2(b) and 2(c), respectively. Fig. 2(b) was indicated for the special peaks of zeolite-Y in the zeolite-Y/carbon composite. It was confirmed that sucrose has successfully entered into the zeolite-Y pore and after carbonization, the sample was turned into carbon with the pore size that similar to template without damaging the crystal pore of zeolite-Y. From Fig. 2(c), it was indicated that special peaks of zeolite-Y were not found, showing that zeolite framework was removed. The residual of carbon was similar to zeolite-Y, which was shown at peak of $2\theta \sim 6^{\circ}$ (Chen *et al.*, 2007). In addition, there was weak peak at $2\theta \sim 25^{\circ}$ (002) plane, indicating for graphitic carbon. It was indicated that there were minor formation of graphite carbon (Guan *et al.*, 2009) and dominant formation of porous carbon.



Fig. 2 XRD pattern of (a) Zeolite-Y; (b) Zeolite-Y/Carbon composite and (c) Zeolite-Y Templated Carbon (ZTC-Y).



Fig. 3 SEM image of (a) zeolite-Y and (b) zeolite-Y templated carbon.

SEM image of zeolite-Y as shown in Fig. 3(a) was indicated for the synthesized zeolite-Y that has an octahedral crystal form order with an average particle size of less than 0.5 μ m. This fact was confirmed the formation of zeolite-NaY crystals.

Figure 3(b) shows the SEM image of ZTC-Y. It could be seen that the formation of carbon particles was similar to zeolite-Y. The formation of octahedral crystals was observed by the appearance of a box shape that resembling a zeolite crystal form. However, in some sections, coagulated carbon was appeared and did not form perfect octahedral crystals.



Fig. 4 TEM image of zeolite-Y templated carbon.

The TEM image of ZTC-Y shows that the carbon particles have a morphology that was similar to zeolite-Y. However, it could be seen that the carbon graphitic on the ZTC-Y was the carbon that did not template by the zeolite. This carbon graphitic would cause a large number of mesopores in ZTC-Y. It was confirmed that the total pore volume of ZTC-Y was bigger than the micropore volume (as shown in Table 3), which indicating that ZTC-Y has a large number of mesopore.

Activation of ZTC-Y with KOH

The activation process of ZTC-Y with KOH was referred to following mechanisms as shown in Equations (3)-(10). When KOH and carbon were heated at the high temperature under nitrogen atmosphere, KOH would react with carbon to form K_2CO_3 , K_2O , and hydrogen gas (Apriani *et al.*, 2013).

$$4 KOH + C \leftrightarrow K_2 CO_3 + K_2 O + 2H_2 \tag{3}$$

Beside the decomposition of KOH into K_2O , the decomposition of KOH could also produce water vapor (Erlina *et al.*, 2015). The water vapor would cause the changing of carbon into CO (Apriani *et al.*, 2013).

$$2 KOH \leftrightarrow K_2O + H_2O \tag{4}$$

$$C + H_2O(uap) \rightarrow H_2 + CO$$
 (5)

The CO would cause the formation of pores by producing CO_2 and H_2 gases as seen in the Eq. 6, in which these gases would then react with K_2O .

$$CO + H_2O \rightarrow H_2 + CO_2 \tag{6}$$

$$K_2O + CO_2 \rightarrow K_2CO_3 \tag{7}$$

$$K_2O + H_2 \rightarrow 2K + H_2O \tag{8}$$

When the temperature reaction was above 700 °C, the amount of K⁺ ion in K₂O and K₂CO₃ could be converted into potassium atom (Pinero *et al.*, 2005). K₂CO₃ was reduced by carbon to form K atom and carbon was oxidized to produce CO gas (McKee, 1983) as shown in Eq. (9).

$$K_2CO_3 + 2C \leftrightarrow 2K + 3CO$$
 (9)

Meanwhile, K_2O was reduced by carbon to form K atoms (Apriani *et al.*, 2013) and carbon was oxidized to produce CO gas as shown in Eq. 10.

$$K_2O + C \leftrightarrow 2K + CO \tag{10}$$

Potassium with an atomic radius of 0.470 nm (Viswanathan *et al.*, 2009) has a good intercalation to develop and form micropores of ZTC-Y. The usage of potassium to form this micropore was suitable for adsorption of CO₂ gas, which has a molecular size of 0.33 nm (Deng *et al.*, 2012).

The activated ZTC-Y with KOH was then washed to remove residual inorganic salt. There were three steps of ZTC-Y washing. The first washing was conducted by immersing the activated carbon of KOH with HCl 2 M. Washing with HCl was aimed to remove inorganic salts (Sevilla *et al.*, 2010).

Subsequently, the sample was washed by using hot and cold demineralized water until neutral pH was achieved. The purpose of washing with hot demineralized water was to reduce the residual KOH that has been converted into potassium ions, whereas washing with cool demineralized water was to remove residual HCl in the initial washing. After washing process, ZTC-Y then was dried in the oven to remove the water content on the carbon.



Fig. 5 XRD pattern of (a) ZTC-Y before activation; (b) activation by weight ratio of KOH/ZTC-Y=0.5; (c) KOH/ZTC-Y=1; (d) KOH/ZTC-Y=1.5 and (e) KOH/ZTC-Y=2.

XRD pattern of ZTC-Y after activation with KOH was shown in Fig. 5. It could be seen that highest peak at $2\theta = 6^{\circ}$ (111) plane indicated that activation did not affect the structure of ZTC-Y, thus enabling the maintenance of replica of zeolite-Y. Peak intensity at 2θ = 6° was decreased with the increase of weight ratio of KOH/ZTC-Y. In addition, peak at $2\theta = 25^{\circ}$ (002) plane was represented for the grafitic carbon, indicating that the carbon did not replicate the zeolite-Y structure (Houghton et al., 2001; Siriwardane et al., 2001). The grafitic peak was decreased with the increase of activator ratio due to the formation of new pores in the grafitic carbon layer (Su et al., 2004; Zhang et al., 2010). Nevertheless, at the weight ratio of KOH/ZTC-Y= 2, it could be seen that there was almost the same peak that was found at ratio of KOH/ZTC-Y=0.5 and it was greater than at ratio of KOH/ZTC-Y=1 and 1.5. It was due to the number of activator that was too little or because of the excess of the activator that could destroy the formed pore (Anggraini, 2013). The XRD pattern also showed that good pore of carbon structure was found at the weight ratio of KOH(g)/ZTC-Y(g)=1.5 with grafitic peak that was lower than in the ratio of KOH/ZTC-Y =0.5, 1, and 2.

Fig. 6 shows SEM image of activated ZTC-Y at the various weight ratios of KOH. Activated ZTC-Y showed similar regularity of zeolite-Y structure, which characterized by octahedral structure, and there were parts of amorphous carbon found on the ZTC-Y surface. Based on Fig. 6, the formation of amorphous carbon on the ZTC-Y surface after activation could enhanced with the increase of KOH ratio. It could be seen that at the ratio KOH/ZTC-Y = 0.5 and 1, the samples were found to be almost octahedral, whereas at ratio KOH/ZTC-Y = 1.5 and 2, the samples were found to be octahedral covered by amorphous carbon due to deposition of carbon on the external material surface. Activator worked strongly at ratio of KOH/ZTC-Y = 1.5 and 2, thus resulting in the massive formation of granules on the carbon surface.



Fig. 6 SEM image of ZTC-Y after KOH activation at weight ratio KOH/ZTC-Y of (a) 0.5 (b) 1 (c) 1.5 and (d) 2.

SEM images were in good agreement with XRD results, which peak intensity at $2\theta = 6^{\circ}$ that showed the regularity replica of zeolite-Y was decreased with the increase of KOH/ZTC-Y ratio. The result of SEM-EDX as shown in Table 1 summarized that KOH activation of ZTC-Y was dominated by carbon and oxigen. It was corresponded to the research reported by Anggraini (2013). However, the Al and Si elements were indicated for the residue of zeolite-Y template, while K element was indicated for the residue of KOH activator. ZTC-Y before and after KOH activation have the same color physically. However, ZTC-Y after KOH activation has a finer texture than ZTC-Y before activation. The result of characterization using SEM-EDX showed that ZTC-Y after KOH activation was dominated by carbon and oxygen content, which were 87.89 % wt and 9.94 % wt, respectively.

Table 1 Elemental analysis by SEM-EDX for activated ZTC-Y with KOH.

Element	wt %
С	87.89
AI	9.94 0.87
Si K	0.33 0.97

The percentage of carbon yield in ZTC-Y was the amount of carbon produced after the activation process with KOH, which shown in Table 2. Based on the data in Table 2, the increasing number of KOH used for activation has resulted in the increasing percentage of carbon burnt off. This burnt-off percentage was expressed as a percentage of carbon loss after heating at high temperatures (Ioannidou *et al.*, 2007). The greater amount of KOH, the lower percentage of carbon yield. It could be happened because the more activators were used, the amount of carbon oxidized into CO or CO₂ gas was increased. As a result, it would decrease the percentage of carbon yield.

The textural properties of zeolite-Y, ZTC-Y and activated ZTC-Y were listed in Table 3. The zeolite-Y has micropore with volume of 0.341 cc/g and there was a slight difference in amount by total volume pore of 0.404 cc/g, which was found from interpore. On the other hand ZTC-Y has micropore volume and total pore volume of 0.624 cc/g and 1.134 cc/g, respectively. This indicated that ZTC Y has

micropore and mesopore. These micropore and mesopore could cause the surface area of ZTC-Y to be larger than zeolite-Y, which were $1359.25 \text{ m}^2/\text{g}$ and $686.82 \text{ m}^2/\text{g}$, respectively.

Table 2	Precentage of	carbon yield ir	n activated ZTC-Y	with KOH.
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Weight Ratio of KOH(g)/ZTC-Y(g)	% burnt off	% Yield
0.5	23.7573	76.2427
1	50.1879	49.8121
1.5	51.1158	48.8841
2	55.6173	44.3827

The average pore of activated ZTC-Y after activation was smaller than before activation, which were 2.356 nm and 3.338 nm, respectively. The difference amount of volume pore between micropore and total pore volume indicated that activated ZTC-Y has micropore and mesopore, which micropore volume and total pore volume of activated ZTC-Y were 0.200 cc/g and 0.657 cc/g, respectively. The pore volume of activated ZTC-Y was lesser than ZTC-Y. It could be due to the presence of residual KOH activators which has covered activated ZTC-Y pore. In addition, the activation process might lead to agglomeration of the material. Thus, the particle size would become larger than before activation and the surface area of activated ZTC-Y could decrease.

CO₂ adsorption capacity measurements

CO₂ adsorption test performed on ZTC-Y before and after activation were conducted to study the effect of KOH activation on CO₂ adsorption capacity. Adsorption test was conducted at temperature of 30°C using gravimetry method. The result of CO₂ adsorption test was presented in Figure 7. It showed that CO₂ adsorption capacity of ZTC-Y was increased after KOH activation. CO₂ adsorption capacity was increased from 1.07 %wt. of ZTC-Y before activation to 1.47, 1.89, 2.72, and 2.10 %wt. of KOH/ZTC-Y 0.5, 1, 1.5, and 2, respectively. The optimum CO₂ adsorption capacity was found at the weight ratio of KOH/ZTC-Y 1.5, which was 2.72 %wt.



Fig. 7 CO_2 adsorption capacity graph of (a) ZTC-Y before activation (b) after KOH activation with KOH/ZTC-Y 0.5 (c) 1 (d) 1.5 and (e) 2.

Table 3 ⁻	Textural	properties	of the	material.
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Material	Surface Area (m²/g)	Average pore size (nm)	Micropore Volume (cc/g)	Total Volume Pore (cc/g)	References
Zeolite-Y ZTC-Y KOH Activated ZTC- Y	686.82 1359.25 557.41	2.352 3.338 2.356	0.341 0.624 0.200	0.404 1.134 0.657	Kayadoe, 2013 Anggraini, 2013 This research

CONCLUSION

Activator KOH could increase the CO₂ adsorption capacity from 1.07 % wt of ZTC-Y before activation to 1.47, 1.89 and 2.10 % wt of the weight ratio KOH(g)/ZTC-Y(g) 0.5, 1, and 2, respectively. The optimum CO₂ adsorption capacity was obtained at the KOH(g)/ZTC-Y(g) ratio 1.5, which was 2.72 % wt with surface area and average pore size of 557.41 m²/g and 2.36 nm, respectively.

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