SHORT COMMUNICATION

CRYSTAL PHASE AND SURFACE MORPHOLOGY OF ZEOLITE-Y TEMPLATED CARBON WITH K_2CO_3 AND $ZnCl_2$ ACTIVATION

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ABSTRACT

Zeolite-Y templated carbon (ZTC) has been activated with K_2CO_3 and $ZnCl_2$. This research aims to compare the characteristics of ZTC with K_2CO_3 and $ZnCl_2$ activation. ZTC was synthesized via impregnation method followed by carbonization. In this research, activation process was conducted at variation of activator/carbon weight ratio of 1 and 1.50. The activation was carried out by heating up impregnated carbon at 800 °C for 1 h followed by washing to remove inorganic salt. XRD and SEM results indicate that the use of different activator produce ZTC with varied structure and morphology. Diffractogram results showed that the graphitic peak decreased due to K_2CO_3 activation, whereas graphitic peak increased owing to $ZnCl_2$ activation. Decreasing the graphitic peak indicates that ZTCs have higher pore structure formation. In addition, surface formation of ZTC activated by K_2CO_3 was higher than by $ZnCl_2$ activation. The higher pore structure formation means the amount of carbon deposited on external surface material was decreased.

Keywords: *zeolite-Y templated carbon; effect of K_2CO_3 and $ZnCl_2$ activator; structure and morphology*

ABSTRAK

Karbon Tertemplat Zeolit-Y (KTZ) telah diaktivasi dengan K_2CO_3 dan $ZnCl_2$. Penelitian ini bertujuan untuk membandingkan karakteristik KTZ yang diaktivasi dengan K_2CO_3 dan $ZnCl_2$. KTZ disintesis dengan metode impregnasi yang disertai dengan karbonisasi. Dalam penelitian ini, proses aktivasi dilakukan dengan variasi rasio berat aktivator/karbon = 1 dan 1,50. Aktivasi dilakukan melalui pemanasan karbon yang telah diimpregnasi pada 800 °C selama 1 jam diikuti dengan pencucian untuk menghilangkan garam anorganik. Hasil XRD dan SEM menunjukkan bahwa penggunaan aktivator yang berbeda menghasilkan KTZ dengan struktur dan morfologi yang bervariasi. Hasil difraktogram menunjukkan puncak grafitik menurun pada aktivasi menggunakan K_2CO_3, sedangkan puncak grafitik meningkat pada aktivasi menggunakan $ZnCl_2$. Penurunan puncak grafitik menunjukkan bahwa KTZ yang dihasilkan memiliki pembentukan struktur pori yang lebih baik. Selain itu, pembentukan permukaan KTZ yang diaktivasi dengan K_2CO_3 lebih baik dibandingkan dengan $ZnCl_2$. Pembentukan struktur pori yang lebih baik ditandai dari jumlah karbon yang terdeposit di permukaan luar material semakin sedikit.

Kata Kunci: *Karbon Tertemplat Zeolit-Y (KTZ); efek aktivator K_2CO_3 dan $ZnCl_2$; struktur dan morfologi*

INTRODUCTION

The use of porous materials currently being attention, relates to the ability of this materials for gas adsorption, gas separation and reversible gas storage. The advantages of porous materials are low density [1] and high thermodynamic stability [2]. Strobel et al. (2006) stated that many research focused on the development and modification structure of materials such as zeolite and carbon [3]. Zeolite templated carbon (ZTC) modification was conducted to produce a material with large specific surface area, high porosity and pore regularity. However, ZTC still has a number of mesopore sizes, which is less suitable for gas storage application and other applications related to gas. An alternative route to vary the porosity of ZTC is post synthesis activation via chemical activation. This step will reduce the amount of mesoporous and increase the amount of micropores [4]. In this study ZTC was activated by using K_2CO_3 and $ZnCl_2$ as activators with weight ratio variation. Activation was conducted to form new micropores from the existing mesopore. The structure and morphology formation was observed with XRD and SEM, respectively.

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EXPERIMENTAL SECTION

Materials

The materials used in this experiment were sodium aluminate, sodium silicate solution, NaOH pellet (99% pa), sucrose (98% Fluka), H_2SO_4 (98% pa), High purity of N_2 gas (99.99% N_2), distilled water, K_2CO_3 (99.99% pa), $ZnCl_2$ (99% pa) HCl (37% SAP), HF (48% pa). ZTC was synthesized according to the method published by Kayadoe [5].

Instrumentation

Powder X-Ray Diffraction (XRD) patterns were collected using CuKα radiation to identify crystal phase and crystallinity. Scanning Electron Microscope images were collected with an accelerating voltage at 200 kV to identify ZTC surface morphology before and after activation.

Procedure

Activated zeolite templated carbon with K_2CO_3 and $ZnCl_2$

ZTC were mixed with K_2CO_3 and $ZnCl_2$ at variation of activator/ carbon weight ratio of 1 and 1.50. Each mixture with weight variation of activator dissolved in 20 mL distilled water and stirred for 5 h. The following relationship show impregnated ratio is:

$$\text{Impregnation ratio} = \frac{\text{activator weight (g)}}{\text{ZTC weight (g)}}$$

After mixing, solutions were dried at 110 °C for several hours and samples were ready for the carbonization with activation under N_2 atmosphere. After being cooled, all samples were washed with HCl, hot distilled water and cold distilled water until pH neutral. The washed samples were dried at 110 °C for 12 h [3].

RESULT AND DISCUSSION

Effect of K_2CO_3 and $ZnCl_2$ towards the Structure of ZTC

ZTC was successfully synthesized from sucrose as carbon source and zeolite-NaY as template according to research methods published by Kayadoe (2013). X-ray diffraction patterns of ZTC shown in Fig. 1(a) shows the presence of peak at 2θ = 6°, which indicates that ZTC can replicate the regularity of zeolite-NaY. In addition, ZTC has amorphous and graphitic peaks at 2θ = 25°. Guan et al. (2009) suggested the presence of graphitic carbon obstructed the developing of pore structure [6].

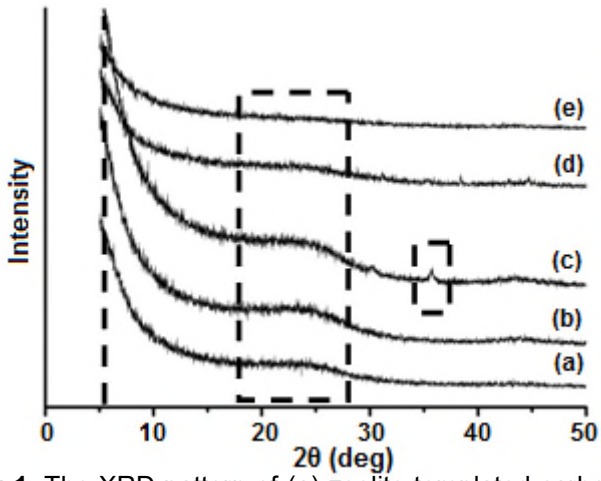

Fig 1. The XRD pattern of (a) zeolite templated carbon (ZTC), (b) $ZnCl_2$/ZTC = 1, (c) $ZnCl_2$/ZTC = 1.50, (d) K_2CO_3/ZTC = 1, (e) K_2CO_3/ZTC = 1.50

ZTC after activation with K_2CO_3 and $ZnCl_2$ were characterized using XRD to compare the effect of activators to the formation of carbon structure. Fig. 1(b) and 1(d) shows the diffractogram of ZTC activated with K_2CO_3 and $ZnCl_2$ at weight ratio of 1, respectively. Activation with $ZnCl_2$ in this ratio shows the formation of peak at 2θ = 6°. This peak appeared during activation process. It indicates that carbon could maintain the replication of zeolite-NaY framework. However, at this weight ratio activator of $ZnCl_2$, there is an increase intensity peak around 2θ = 25°, which shows more graphitic peak. The graphitic peaks generate poor pore structure development. The activation of ZTC with K_2CO_3 weight ratio of 1 showed the presence of peak around 2θ = 6°, with low intensity. This pattern indicated by using K_2CO_3 activation, there is little damage to the structure of carbon. However this material still able to maintain the replication of zeolite-NaY structures. It suggests that during the activation process, K_2CO_3 chemically reacts with carbon at high temperature and simultaneously form new pores from graphitic carbon. Powder X-Ray Diffraction pattern shows there was no other peak, which means that K_2CO_3 could be completely removed after washing process.

Fig. 1(c) and 1(e) shows the diffractogram of ZTC activated with K_2CO_3 and $ZnCl_2$ at weight ratio of 1.50, respectively. When $ZnCl_2$ at this weight ratio was used, the diffractogram peak at 2θ = 6° is shifted. There are graphitic peak which indicated poor pore structure development at 2θ = 25°. In addition, there is Zn peak at 2θ = 36°. These results suggest some Zn were left behind after activation process. In this case, Zn causes blocking on ZTC and also decreases the surface area of ZTC. The diffractogram peak intensity around 2θ = 6°

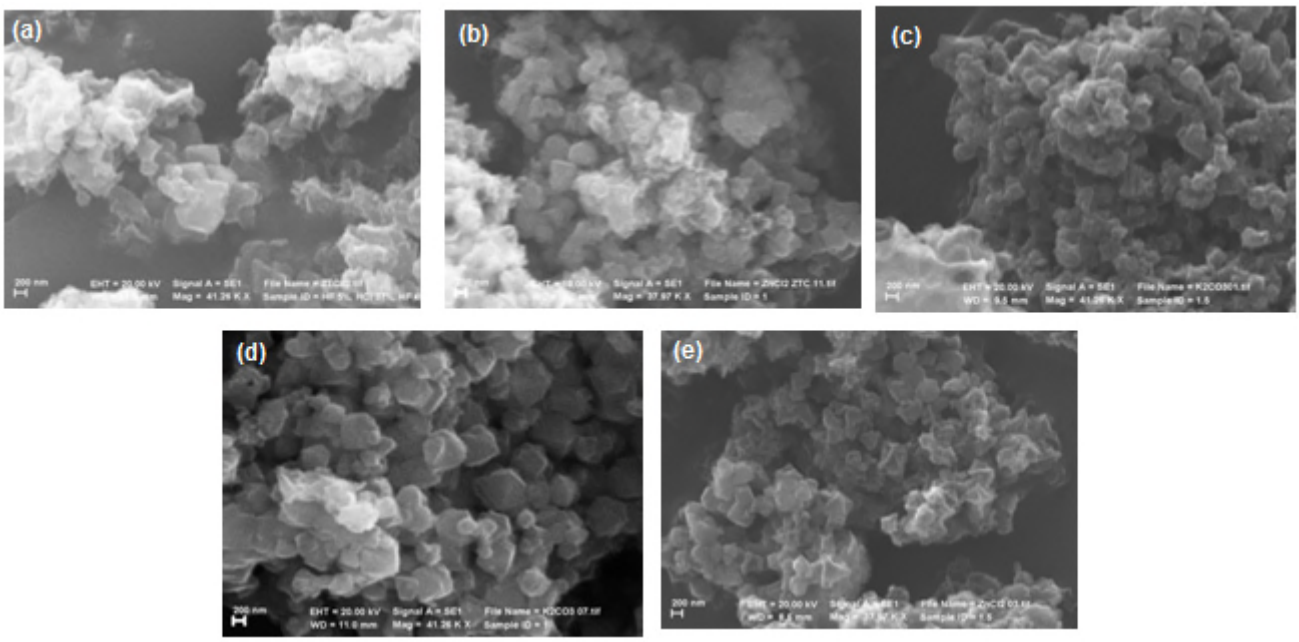

Fig 2. SEM image of (a) ZTC, (b) $ZnCl_2$/ZTC = 1, (c) $ZnCl_2$/ZTC = 1.50, (d) K_2CO_3/ZTC = 1, (e) K_2CO_3/ZTC = 1.50

of ZTC at K_2CO_3 activation at weight ratio of 1.50 was low, but graphitic peak at 2θ = 25° was hardly noticeable. This means that carbon with poor pore structure has been converted into porous carbon during activation process.

Effect of K_2CO_3 and $ZnCl_2$ towards the Morphology of ZTC

Fig. 2 shows the SEM images of ZTC before and after activation using K_2CO_3 and $ZnCl_2$ with variation of weight ratio. The morphology of ZTC resembles the morphology of zeolite-NaY. ZTC has crystal box shape with a few aggregate showing formation of amorphous carbon due to imperfect carbon filling in the zeolite pores and the formation of turbostatic carbon with poor pore structure development.

ZTC morphology after K_2CO_3 and $ZnCl_2$ activation at weight ratio of 1 as shown in Fig. 2(b) and 2(d), respectively. ZTC with $ZnCl_2$ activation at weight ratio of 1 indicates formation of octahedral crystals and amorphorous carbon. The damage of ZTC morphology during activation process confirms the result from XRD pattern. Activation with K_2CO_3 at this weight ratio showed the formation of octahedral crystals with little amorphous carbon on the surface. Based on morphological description, it seems that the use of K_2CO_3 could reduce the formation of amorphous carbon on the surface of ZTC.

ZTC morphology after activation with $ZnCl_2$ and K_2CO_3 at weight ratio of 1.50 was shown in Fig. 2(c) and 2(e). The formation of amorphous carbon using K_2CO_3 activator at this weight ratio was less than using $ZnCl_2$ activator at same ratio. This result appropriates with XRD result which shows the use of K_2CO_3 for activation could reduce graphitic carbon (2θ = 25°), whereas activation using $ZnCl_2$ increase graphitic peak. The increase of graphitic carbon will enhance the amount of poor pore carbon development. Based on this fact, activation with $ZnCl_2$ could produce a material with smaller porosity.

CONCLUSION

ZTC has been activated with K_2CO_3 and $ZnCl_2$ as activator at weight ratio of 1 and 1.50. The use of $ZnCl_2$ as activator increased graphitic peak intensity, indicated the increase of poor pore structure development. On the other hand, activation with K_2CO_3 reduced the intensity of graphitic peak. This means that the development of ZTC pore structure was improved.

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