Properties of Activated Carbon from Lignite

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Abstract

The research aimed to study the properties of carbonized and activated lignite. The lignite was carbonized at 300-700 °C. The activated lignite was prepared from lignite carbonized at 500 °C and activated by using phosphoric acid with a ratio of 1:0.5 (w/v), then pyrolyzed at 600-800 °C and demineralized with 12 N HCl and 22 N HF. The surface properties of carbonized and activated lignite were studied using FTIR, SEM and XRD. The FTIR spectra of carbonized lignite showed a shift to a lower wavenumber for the (C=C) at 1603.08 cm^{-1} when increasing the carbonized temperature from 300 to 700 °C. The broad bands at 1032-1034 cm^{-1} [ν(C-O)] appeared after being carbonized at 300 °C and decreased when increasing carbonized temperature from 300 to 700 °C. The bands between 1136.86 and 1031.98 cm^{-1} were observed in all spectra of activated lignite. These bands indicated the presence of phosphorous and oxygen compounds. The SEM images of the lignite when carbonized at 500 °C had quite regular particles and little pores. With phosphoric acid activation at 800 °C, activated lignite showed the creation of more pores and were quite irregular. The XRD pattern for activated lignite at 800 °C corresponded to graphite.

Keywords: Activated carbon; Lignite; Phosphoric acid; Surface properties

INTRODUCTION

The lignite found in the Mae Moh Valley has a high carbon content. It is burned to generate electricity (Mouloud et al., 2002). The main minerals in Mae Moh lignite are quartz, pyrite, and a clay fraction consisting of kaolinite and illite (Ward, 1991). The preliminary experiment showed that the Mae Moh lignite contains 8.12, 22.86, 69.02 and 36.63 percent by weight of moisture, volatile matter, fixed carbon and ash, respectively. Previous research has involved the effect of γ-irradiation from a ^{60}Co source on the structure of activated carbon prepared form lignite. γ-Irradiation caused remarkable erosion on the surface with the loss of some carbonaceous matter by probable bond cleavage reactions (Erçin et al., 2005). Chemical activation of lignite or bituminous coal was studied using $ZnCl_2$, H_3PO_4, and KOH (Hsu & Teng, 2000; Toles et al., 1996). A thermogravimetric study showed that these reagents were capable of suppressing the evolution of tarry substances during carbonization. In this research, we chose phosphoric acid for activation of banana peel.

Activation of lignite, obtained from the Mae Moh Basin, Lampang Province, Thailand, was studied using phosphoric acid activation followed by pyrolysis at 600-800 °C.

MATERIALS AND METHODS

Preparation of carbonized lignite

Lignite obtained from the Mae Moh Basin, Lampang province, Thailand, was used in this study. It was dried in an oven at 110 °C for 3 h. Thereafter, it was carbonized at 300, 400, 500, 600, and 700 °C for 1 h under closed system in a porcelain crucible, and then it was cooled to room temperature. The raw lignite and carbonized samples were characterized using FTIR (Spectrum GX, Perkin Elmer), SEM (LEO 1455 VP electron microscopy) and XRD (PW 3040/60, X' Pert Pro MPD) to determine functional groups, surface morphology, and the type of carbon, respectively.

Preparation of activated lignite

The lignite carbonized at 500 °C was agitated in 85% (w/w) of H_3PO_4 (UNIVAR, AR) at a ratio of 1:0.5 (lignite : H_3PO_4) on a prepared weight by volume basis. Thereafter, the slurry was left overnight at room temperature, and then dried at 110 °C for 24 h. The samples were then activated in a closed system. The samples were pyrolysed at three consecutive temperatures from 600, 700 and 800 °C to optimize the heat treatment. They were then maintained at a constant temperature for 1 h before cooling. After cooling down, the activated charcoal was washed several times successively with 5 N HCl (BDH, AR.), 22N HF (MERCK, pro analysi), then with hot water until the pH became neutral, and finally with cold water to remove the excess phosphorous compounds. The washed samples were dried at 110 °C to get the final product. The final products were analysed using FTIR, SEM and XRD.

RESULTS AND DISCUSSION

FTIR spectra

From Figure 1, FTIR spectra of carbonized lignite shows the loss of ν(C-H) vibration in alkanes or alkyl groups (peak of 2923.77 cm^{-1}) when carbonized at 400 °C. The FTIR spectra shows a shift to a lower wavenumber for the ν(C=C) at 1603.08 cm^{-1} when increasing the carbonized temperature from 300 to 700 °C. The broad bands at 1032-1034 cm^{-1} are present after carbonizing at 300 °C. It has been assigned to C-O stretching in acids, alcohols, phenols, ethers and esters (Budinova et al., 2006). The C-O peak decreases with increasing carbonized temperature from 300 to 700 °C. This indicates that the degree of stable C-O functions decrease with increasing carbonized temperature. Therefore, it is assumed that the increased temperature has released acidic and phenol groups. According to this phenomenon, dehydration of OH groups in samples to an enol and reduction of carbonyl group to alcohol and then the hydroxyl ion reacts with the addition of carbon monoxide to regenerate the ormate ion (Demirbas, 2000).

Figure 2 shows the FTIR spectra of the activated lignite with temperatures from 600 to 800 °C. The spectrum of activated lignite with a temperature of 800 °C is quite smooth, evidencing a low amount of functional groups. It may also be observed that increasing the temperature reduces the intensity of all bands. The bands between 1136.86 and 1031.98 cm^{-1} are observed in all spectra. These bands indicate the presence of phosphorous and oxygen compounds in the activated lignite. The bands in this region are usually found in oxidized carbons and carbons activated with phosphoric acid. Due to an overlap of the adsorption bands from C-O in acids, alcohols, phenols, ethers and esters in this region, an unambiguous assignment is difficult (Budinova et al., 2006). These bands, in general, become reduced with an increase in activated temperature from 600 to 800 °C.

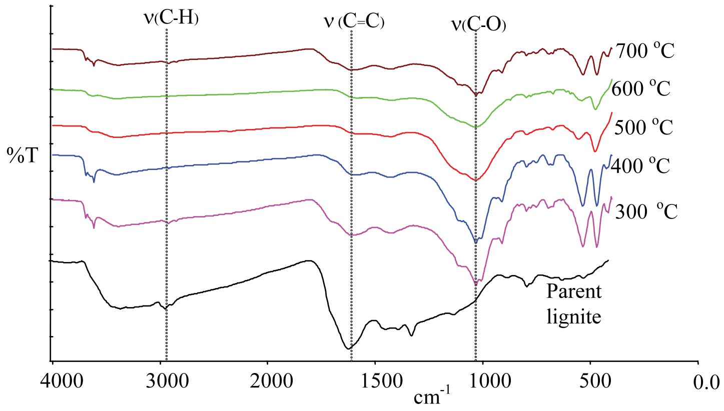


Figure 1. FTIR spectra of lignite with carbonized at 300-700 °C.

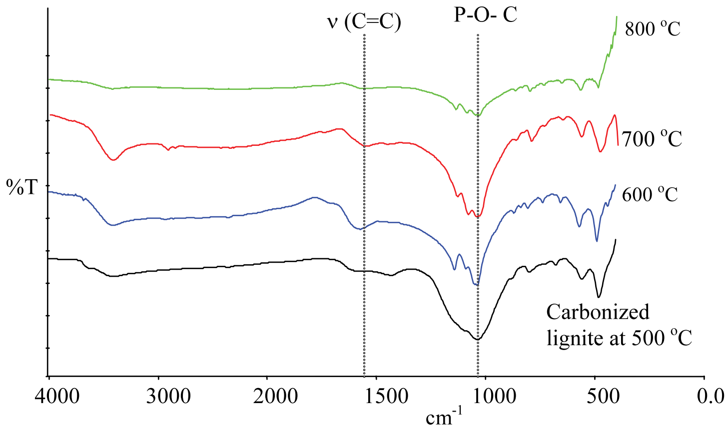


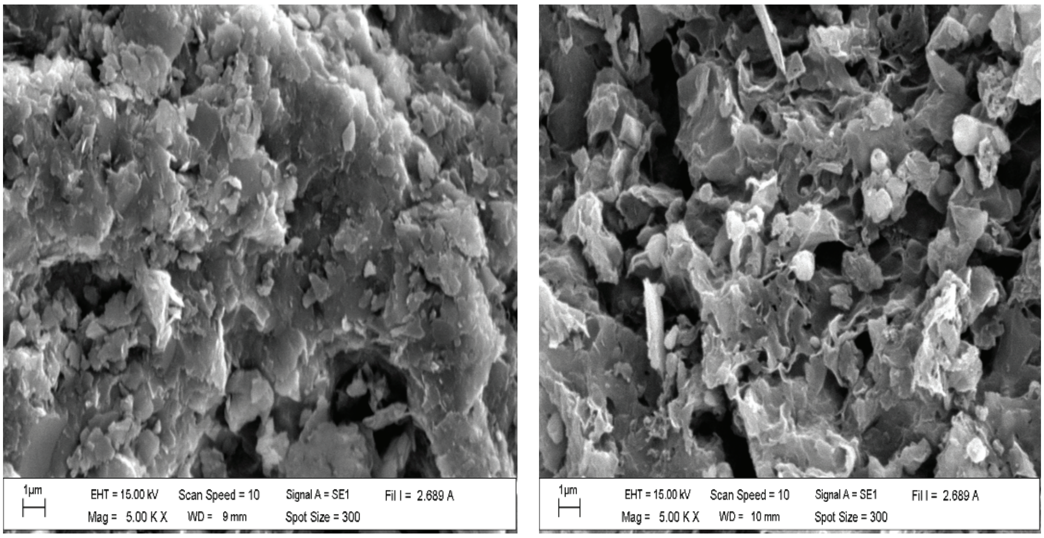
Figure 2. FTIR spectra of activated lignite at 600-800 °C.

Scanning electron micrography

Figure 3 illustrates the SEM images of the carbonized lignite (500 °C) and activated lignite (800 °C). From Figure 3 (a), carbonized lignite has quite regular particles and small pores. Phosphoric acid activation at 800 °C results in the creation of more irregular pores (Figure 3 (b)). This is assumed to be due to the diffusion of the phosphoric acid into the carbonized lignite (Toles et al., 1996). It can be seen that the external surface of activated lignite is full of cavities. This results from the evaporation of phosphoric acid during pyrolysis, leaving the space previously occupied by the phosphoric acid (Hsu & Teng, 2000).

XRD analysis

The XRD pattern for activated lignite is shown in Figure 4. The activated carbon exhibits two peaks at around $2\theta = 26^\circ$ and 44° which corresponds to the peak of graphite. At the same time, it exhibits noise of the powder XRD signals. This reveals a predominantly amorphous structure of carbon. It has been explained that the pyrolytic reaction of organic compounds consists of the breaking of chemical bonds with temperature and repolymerization of radicals condensing further into active compounds. These compounds form typical graphitic layers and stacks of planes during carbonization (Kurosaki et al., 2003).



(a) **(b)**

Figure 3. Surface morphology of lignite when carbonized at 500 °C (a) and after activation at 800 °C (b).

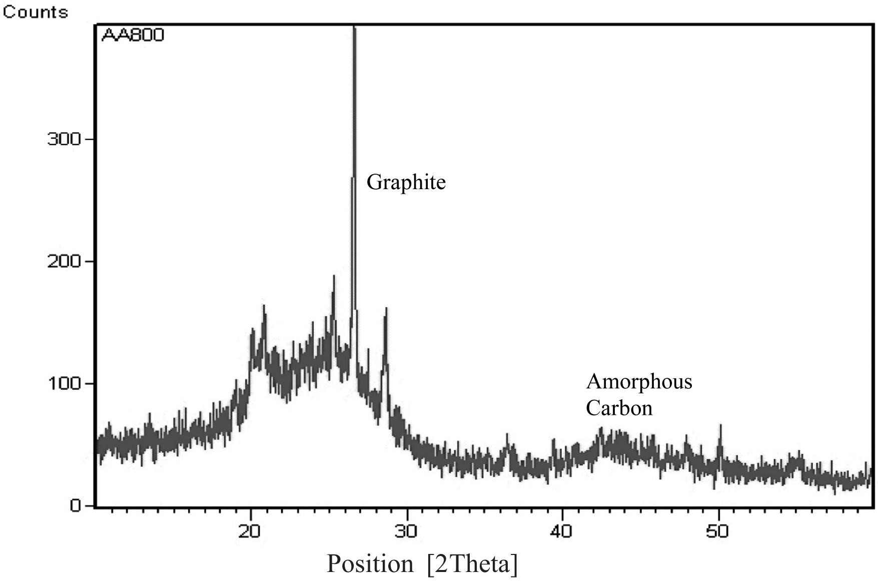


Figure 4. XRD pattern of activated lignite at 800 °C.

CONCLUSIONS

The C-H functional group of carbonized lignite disappears after being carbonized at 400 °C. The C=C functional groups decrease when increasing the carbonized temperature from 300 to 700 °C. The broad bands of C-O are present after being carbonized at 300 °C and decrease after increasing carbonized temperature from 300 to 700 °C. The FTIR spectra of the activated lignite when increasing the temperature from 600 to 800 °C show bands of phosphorous and oxygen compounds. These bands are generally reduced as the temperature is increased from 600 to 800 °C. The SEM images of the lignite when carbonized at 500 °C has quite regular particles and small pores. With phosphoric acid activation at 800 °C, the activated lignite shows more irregular pores. The XRD pattern for activated lignite corresponds to graphite. It indicates that graphene sheets are the main carbon phases.

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