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Synthesis and characterization of activated carbon produced from kenaf core fiber using H₃PO₄ activation

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Abstract

Kenaf core fiber (KF) which is an agricultural biomass was used to produce low-cost activated carbon using acidic chemical activating agent. The aim of this study is to find out the changes occuring in kenaf core fiber during activation with phosphoric acid (H₃PO₄). The surface area of the formed phosphoric acid treated kenaf core fiber activated carbon (KFAC) was determined by physical adsorption of N₂ gas. Brunauer, Emmett and Teller (BET) surface area, and micropore volume values were 299.02 m^2/g and 0.12 cm^3/g , respectively. Fourier transform infrared (FT-IR) spectroscope analysis identified the present of carbonyls, alkenes and hydroxyls. Field Emission Scanning Electron Microscope (FESEM) image showed gradual formation of pores due to elimination of volatiles and contaminants. Powder X-ray diffraction (XRD) analysis indicated the appearance of broad diffraction background revealed predominantly amorphous structure. The proximate and ultimate analysis showed high percentage of carbon and low percentage of ash which is an indication of a good material for production of porous carbon.

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1. Introduction

Activated carbons (AC) are made from materials rich in carbon through carbonization and an activation process. A porous structure and its adsorption properties can be obtained in carbonaceous materials via either chemical or physical¹ activation. Physical activation involves pyrolysis of the source material at 800°C-1000°C to produce charcoal. This is then followed by activation using steam, carbon dioxide (CO₂) or oxygen (O₂).

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Chemical activation involves the impregnation of the precursor material with a chemical activating agent followed by activation at temperatures of 400°C-700°C under nitrogen atmosphere². Zinc chloride (ZnCl₂), potassium hydroxide (KOH) and phosphoric acid (H₃PO₄) are widely used as chemical activating agents. The activating chemical agents influence the pyrolytic decomposition and inhibiting formation of tar and volatile matter, thus enhancing the yield of AC³. The dehydration and oxidation characteristics of chemical activation agent require much lower activation temperature compare to physical activation⁴. A comparative study of chemical (H₃PO₄) and physical activation using biomass fiber as AC has been reported⁵. The research found that the surface area recorded by physical activation was slightly higher than chemical activation but lower in term of yield production. The carbonization of biomass at high temperature would trigger the emission of higher greenhouse gaseous (GHG) such as carbon monoxide (CO) and methane (CH₄)⁶ which is an unfriendly approach.

There has been remarkable interest in the production of AC due to its adsorptive, environmental, thermal, electrical and mechanical characteristics^{7,8}. It is proof from previous study that AC is one of the leading industrial materials due to its well developed pore structure and adsorption properties^{9,10,11}. Recent developments in modern technologies have resulted in various novel applications of AC. Coal-based AC is the most common adsorbent used, however, it is costly and not sustainable. Abundant low cost agriculture biomass, which contain high amounts of natural polymers such as cellulose, hemicellulose, and lignin, are suitable alternative materials to produce low-cost and green adsorbents with necessary modification to enhance its efficiency¹². Various studies on the production of AC have been done by applying different agricultural biomass including rice husk¹³, grape seed¹⁴, rambutan peel¹⁵, kenaf fiber¹⁶, maize cob¹⁷ and date pit¹⁸.

Kenaf (*Hibiscus cannabinus*, L) is herbaceous annual plant that belongs to the family of Malvaceae. Kenaf possess two kind of fibers that may be explored to produce AC. The long fiber contain approximately 30% of the total volume of the plant whereas the short fiber represents the remaining of $70\%^{19}$. The stem of kenaf plants consists of the outer bark (bast) and inner core, both contain fibrous components. Recent studies have demonstrated the capability of kenaf core and bast fibres to enhance bioremediation²⁰ and adsorption²¹ process. There were earlier studies have been made to produce ACs from the inner cores of kenaf where the first study utilized KOH and CO₂ through physicochemical activation²², whereas the other study utilized potassium oxalate (K₂C₂O₄) under different impregnation ratios²³. Both studies have significantly increased the evolution porosity in the carbon matrix. Hence, the objectives of the present study were to synthesis the AC from kenaf core fiber using H₃PO₄ as activating agent and conducted the characterization of elemental analysis, surface area, surface chemistry and morphological properties.

2. Experimental methodology

2.1. Raw materials

The raw kenaf core fiber (KF) sample was obtained from Lembaga Kenaf dan Tembakau Negara. Phosphoric acid (H_3PO_4) (~85% acid) and Sodium Hydroxide (NaOH) were obtained from Merck (M) Sdn, Bhd, Malaysia. All chemicals used in the investigation were of analytical grade.

2.2. Sample preparation

Preliminary washing of the raw materials with hot distilled water were conducted and dried in an oven at 105° C for 24 h to remove all moisture. The dried samples were cut into small pieces, sieved to the size of 500-600 µm and stored in air-tight containers to prevent moisture build up and fungi infections. Chemical activation was carried out by initially carbonizing the precursor in muffle furnace under inert atmosphere from room temperature to carbonization temperature of 400°C for 1 hour under a closed system. The resulting char was labelled as KFC. The KFC was subsequently impregnated with 30% ortho-phosphoric acid (H₃PO₄) with impregnation ratios of 1:4 (w/w) for 24 h of soaking duration. Next, the sample was filtered using a vacuum pump and dehydrated overnight in an oven at 105°C. The dried sample was then pyrolysed for activation at temperatures of 500°C. After the activation

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