Preparation of creating active carbon from cigarette filter waste using microwave-induced KOH activation

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Abstract. For the first time, cigarette filter waste, which is an environmental hazardous material, is used as basic material prepared for creating activated carbon (AC) via KOH chemical activation using a microwave input power of 630 W and irradiation time of 20 minutes. Active carbon was characterized by TGA, x-ray diffraction, scanning electron microscopy, energy dispersive x-ray, nitrogen adsorption-desorption, and absorption of methylene blue (MB). The results of x-ray diffraction showed that active carbon has a semi-crystalline structure with peaks of 2θ of $22.87°$ and $43.70°$. Active carbon microstructure analysis showed that the layer height (L_c) is inversely proportional to the width of the layer (L_a), and the distance between the two layers is d_{002} and d_{100}, which depends significantly on the ratio of AC: KOH. It was found that the optimum BET surface area and adsorption capacity for MB were 328.13 m^2 / g and 88.76 m^2 / g, respectively. The results revealed the potential to prepare activated carbon from cigarette filter waste using microwave irradiation.

1. Introduction

There are 5.6 trillions of cigarette filter butts which have been produced by tobacco industries worldwide, and it is now the biggest challenge for global environment protection every year [1]. Cigarette filter butts (CF) are made of nearly 95% non-biodegradable cellulose acetate (plastic) which looks like cotton. CF is disposal, toxic, and hazardous material waste thrown to the environment after usage despite its extremely slow degradation process [2]. It has a microscopic-sized fibrous matter characterized with Y shape made of cellulose acetate. A plasticizer, glycerol triacetate, is applied to bond the fibers [3]. Cellulose acetate is a type of polymer made of paper and similar to cotton of fiber composites in thousands of fiber. For this research, the writer could not find any similar research using cellulose acetate precursor of cigarette butts waste as an active carbon.

The   CF waste has the potential to be used as raw material for active carbon. It has been reported that active carbon materials containing a meso-/micropore structure can be produced using carbonization process with 262 m^2/g of its surface area [4]. The porous carbon of CF has also been studied using pre-treatment sulfuric acid (H_2SO_4) which is carbonized at 500˚C temperature. Using the Braumauer Emet and Teller (BET) method, surface area was at 117.2 m^2 / g and the adsorption-desorption isotherm surface area decreased by around 72% [5]. These active carbons are especially suited to oil-water separation, methylene blue (MB) adsorption, and super capacitors [6-8]. The use of active carbon as alternative adsorbent material is because it has large surface area and structural diversity.

In this present work, we report for the first time the preparation of active carbon from CF butts waste by microwave induced KOH activation and a way of making use of a waste material. The CF butt which was a hazardous waste material was selected as promising alternative due to its abundance, thus reducing it as a disposal problem. Microwave heating method has been applied to prepare active carbons because it rapidly and uniformly heats [9]. In addition, it is an energy-efficient method compared to conventional methods by efficiently improving the quality of final products. Effect of the activation ratio of KOH to enlarge the absorption capacity and MB absorptions of the CF active carbon products were then investigated to determine the optimum preparation condition.

2. Materials and method

2.1 Experiment

A set up experiment has been done using microwave induce active carbon from biomass with KOH activation method as reported in [9]. In this research, the cigarette filters (CF) from Marlboro Red (Philip Morris, Indonesia) were collected and used for this purpose. The CBF were then washed with hot water and unwrapped from the cigarette paper. They were then washed again with 96% ethanol for three times, dried in open air for 72 hours, and placed into a vacuum oven for 3 hours at 230°C for heat treatment. The CF samples were then crushed into a powder size of about 100 μm. The CF powder was activated with potassium hydroxide (KOH) solution at ratio carbon-to-alkali from 1, 2 and 3 labeled as CF-1, CF-2, and CF-3, respectively. The activation step was induced in a tubular glass reactor with a microwave input power of 630 W at 2.45 GHz frequency and irradiation time for 20 minutes. The samples were washed with hot distilled water (85°C) until the pH of the filtrating solutions was neutral (pH = 7.0). Finally, the cleaned samples were immediately dried in a vacuum oven at 100°C for 60 hours to eliminate moisture.

2.2 Characterization Technique

Thermal decomposition analysis of the CF was determined using Thermal Gravimetric Analysis (TGA) of Shimadzu TGA-50 technique at the temperature range from 30°C up to 600°C at the heating rate 1°C/min in liquid nitrogen flow. Crystallographic properties of the prepared active carbon of the sample were characterized using x-ray diffractometer (XRD) of Siemens D5000m, with nickel-filtered Cu K K-α target (λ = 1,5418 Å) at the scan rate 0.5°/min in the 2θ range from 10-60°. The morphology and microstructural investigation of the prepared samples were then performed using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) (JEOL-JSM 6360LA) technique. The adsorption-desorption isotherm of surface area was analyzed using Quantachrome Instruments with liquid nitrogen flowing at 77K. Finally, all of the samples were studied on MB adsorption.

3. Results and discussion

The TGA analysis has shown the obtained thermal decomposition of CF in the first stage of heating. A mass loss was observed in 250°C to 400°C temperature. This stage was due to the dehydration of water content in the CF samples. The highest mass loss (74%) trend was observed from second stage starting at 260°C and was completed 350°C. At higher temperatures, the sample mass decreased slightly up to 600°C. The results of weight loss showed a CF carbon yield of 16% of the final mass, indicating the decomposition of cellulose which was a structure with higher stability.

The effect of pyrolysis of cellulosic acetate with a mass loss in the second stage, reported in [4], yielded 12% of final weight loss and started at 280°C to 370°C temperature. In [5], cellulose acetate filter with CF carbon yield of 10% to 15% using carbonization process was conducted at 280°C to 360°C temperature, and the result had good agreement with our results.
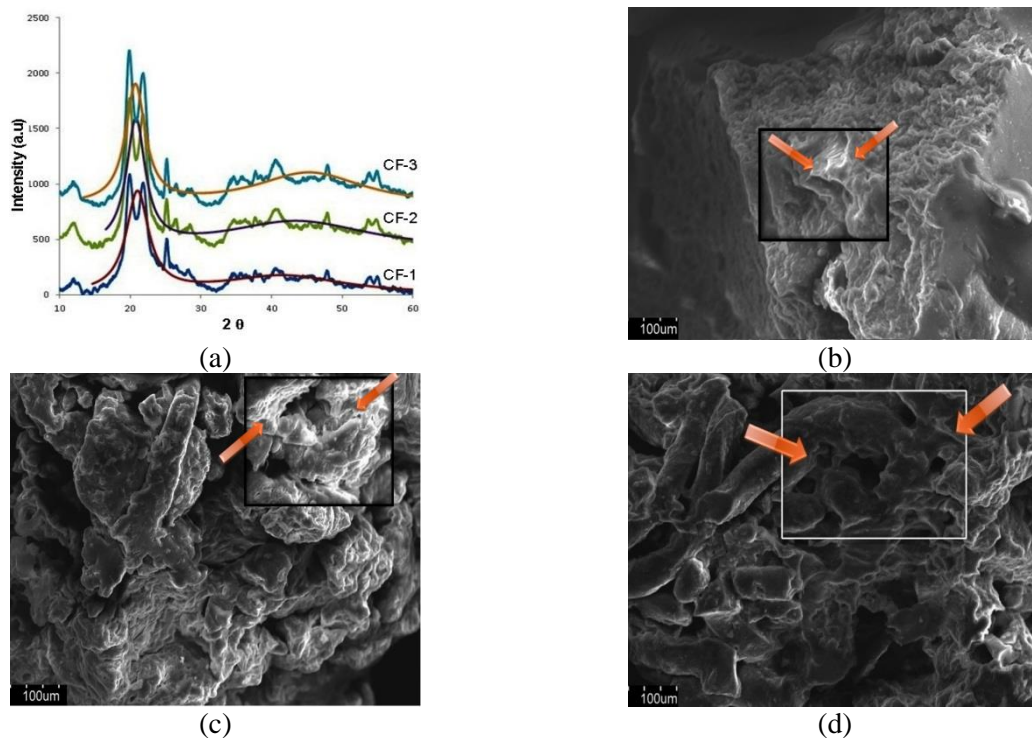
Figure 1. (a) XRD pattern and (b), (c), (d) SEM images of the active carbon CFs.

Figure 1 (a) shows the results of XRD pattern of all samples indicating two major reflections at the position 22.87° (002) and 43.70° (100). The XRD analysis shows a shift angle of reflection for each peak due to KOH activation ratio. The relative crystallinity all the sample (2θ) was quantified range from 10° to 60° and fit using the Origin software (Microcal Inc., Version 8). From the determination of the interlayer interferences, the average layer height L_c was 3.53 nm with the average width of the layer L_a was 0.59 nm for the intensity of peaks (002) and (100). The interlayer spacing was calculated for sample CF-3 and resulted in d_{002} (0.38 nm) and d_{100} (0.21 nm). These reflections were similar to non-graphitic carbons with significant degree of rotational and translational disorder in the stacking of the graphenes, resulting in better layer alignment [4]. Active carbon CF microstructure analysis showed that the layer's height (L_c) was inversely proportional to the width of the layer (L_a), and the distance between the two layers of d_{002} and d_{100} depends significantly on the ratio of CF and KOH.

Figure 1 (b), (c), and (d) show SEM image of samples CF-1, CF-2, and CF-3, respectively. The porous active carbon of all samples is meso-/micro porous-composed with the diameter of ~ 50 μm and the length of ~ 10 μm. Figure 1 (d) shows a magnified images depicting numerous micro porous structure on the surface of the active carbon CF. As comparison, the SEM reported that CF carbonized at 800°C for 7 hours has the same morphology with our result of CF-3 [5]. EDX analysis of all samples shows the presence of C and O elements with the following composition: 62.23% C, 37.77% O (CF-1); 65.50% C, 34.50% O (CF-2); and 68.68% C, 31.32% O respectively. The result shows that there is no other element observed which was composed in our active carbon CF.

To study the effect of active carbon CFs porosity after KOH activation, nitrogen adsorption-desorption isotherms measurement was then calculated using BET method, and it resulted specific surface area of 303.49 (m^2/g) with pore diameter of 3.01 nm for CF-1 and 328.13 (m^2/g) with pore diameter of 3.04 nm for CF-3 compared to 262 m^2/g for porous carbon from cellulose acetate fibers [5].
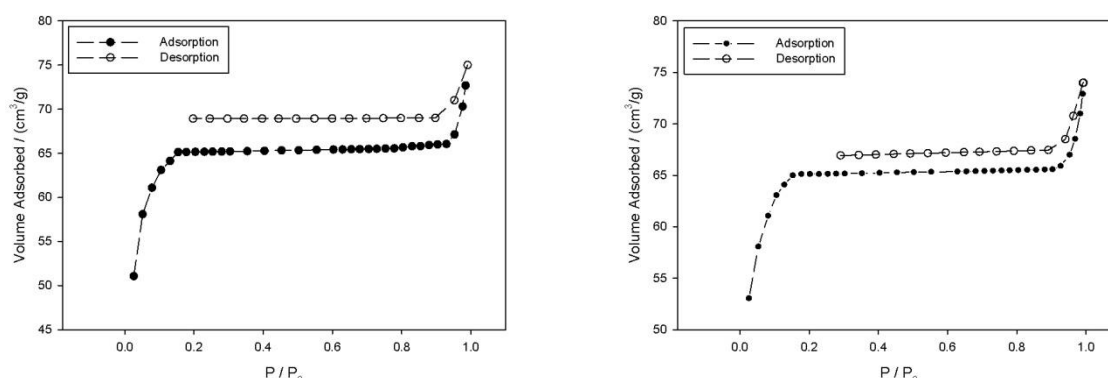
Figure 2. Nitrogen adsorption-desorption isotherms of active carbon CFs (a) CF-1 and (b) CF-3

The ability of active carbon to absorb the MB is a quality standard to absorb dye. The active carbon CFs prepared in this work showed relatively high MB adsorption capacity of 88.77 mg/g for CF-3, as compared to some previous works reported in the literature [10].
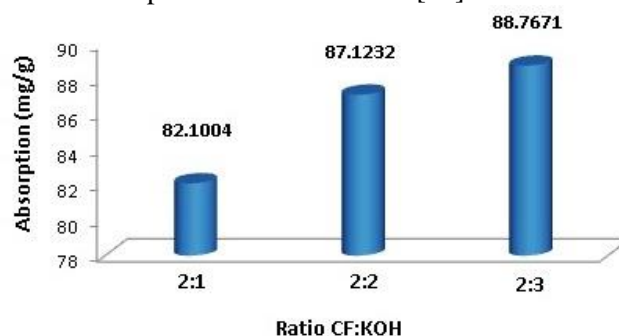

Figure 3. Absorption of methylene blue Ratio of CF:KOH

The observed result of MB active carbon is verified with the analysis of the microcrystalline dimension, the average layer height L_c, the average width of the layer L_a, as well as surface area of active carbon produced. The microcrystalline dimension of active carbon CFs L_c increased inversely proportional and showed a similar trend to that of the L_a micro crystalline dimension (ratios-to-alkali) ranging from 1, 2 and 3 due to enlarged surface area for absorbed MB.

4. Conclusion

The result shows the adaptability of microwave-induced in activating the KOH/cigarette filters waste with simple activation time for 20 minutes and 630 Watt at 2.45 GHz frequency. The two broads peak at (002) and (100), and shifted diffraction peaks in XRD curve due to variation on the mass ratio of CF/KOH. The BET surface area and pore diameter were 328.13 m^2 / g and 3.04 nm respectively. This indicates the optimum adsorption capacity of active carbon for MB of 88.77 mg / g, verified by the morphological and nitrogen adsorption-desorption isotherms studies.

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