Synthesis of activated carbon fiber from pyrolyzed cotton for adsorption of fume pollutants

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Abstract. In this study, we have synthesized and applied the activated carbon fibers from pyrolyzed cotton to adsorb fume pollutants. The activated carbon fibers from cotton were synthesized using an oven with simple heating method at low carbonization temperature. The cotton was successfully turned into carbon within four hours at carbonization temperature of 250°C. The results showed that activation process using KOH and NaOH significantly affected the functional groups, morphology, diameter, and porosity of the activated carbon fibers.

1. Introduction
Activated carbon is one of the materials which have an important characteristic, i.e. adsorption. Adsorption is a physics or chemical phenomenon on the surface of material between the adsorbent media and the adsorbed media. Compared to activated carbon powder or granule, the activated carbon fibers have higher porosity and its smooth fibers constitution will generate fast kinetic adsorption in liquid and gas phases [1,2]. Most of the activated carbon fibers have a size from 7 to 15 μm. Activated carbon fibers can be used as an adsorbent agent [3] and electrode [4].

Activated carbon fibers can be made from various materials which contain high enough carbon. Most of the activated carbon fibers have been synthesized from fossil-fuelled precursor [1]. However, the lack and rarity on the resources of fossil fuel will hinder the production of activated carbon fibers. Therefore, it is necessary to develop activated carbon fibers from some new kinds of raw materials which are abundant, obtainable and renewable sources.

In recent years, many researchers have developed activated carbon fibers from natural to produce activated carbon fibers with high adsorption capacity. Some natural fibers which have been used were hemp [5], cotton bar [6], coconut stem [7], bamboo [1,8], pineapple leaf [9], cocoa peel [10], durian peel [11], and cotton [12].

However, the number of researchers who were using cotton as the raw material to produce activated carbon fibers is still limited. In fact, a material containing a lot of cellulose is good enough to be used as the raw material since it contains activated groups of carbonyl, hydroxyl, and ether which are potential for adsorption process [11]. Cotton is abundant material on earth, easily available and also economical.
It belongs to a polymer of a natural product whose structure component is mainly cellulose [13]. However, the synthesis of activated carbon fibers usually needs high temperature [6,14].

In this study, we report the synthesis of activated carbon fibers using the oven as simple heating with low temperature. The activated carbon fibers have a potential for fume adsorption applications.

2. Methods
The synthesis of activated carbon fibers was used the oven as a simple heating method at 250°C within a certain period until the cotton turned into charcoal. The material used was facial cotton which contains 100% cotton. The carbonized cotton was then activated using KOH 10% and NaOH 10%. The volume ratio between NaOH/ KOH and a mass of cotton charcoal was 10: 1. After activation process, washing and drying were done at a temperature of 100°C. The carbon fibers then were used as the adsorbent to adsorb the fume pollutant. The adsorption media functioned as the place of flow for the mosquito repellent to reach the activated carbon as the fume pollutant adsorption. The carbon fibers were placed in the middle of the media and then passed by the fume for 30 minutes. The adsorption media was made using acrylic with length 30 cm, width 10 cm, and height 10 cm (Figure 1). The bottom part of the adsorption media was beam-shaped as the source of the fume while there was a fan at the upper end to pull up the fume through the activated carbon fibers.

![Figure 1. Scheme of fume adsorption media.](image-url)

The characterizations of the activated carbon fibers in this study included FTIR (Fourier Transform Infrared) and SEM (Scanning Electron Microscope). FTIR was used to detect any functional groups inside the activated carbon fibers. SEM was used to obtain the data regarding the diameter size and the morphology of the fibers as well as the porosity of the activated carbon fibers.

3. Result and Discussion

3.1. Synthesis of Activated Carbon Fibers
In the carbonization process, the cotton turned into black or charcoal within four hours. The chemical activation was done for 24 hours until the carbon fibers were settled down in the solution. Figure 2 shows the difference between the cotton before and after activation process. The activated carbon fibers have more solid surface structure.
3.2. Characterization of FTIR

The adsorption characteristic is strongly affected by chemical composition of the activated carbon. The capability of the activated carbon to adsorb was determined by its chemical structures. There were C, H, and O atoms which were chemically bound to form functional groups [14].

Figure 3 presents the FTIR spectra of cotton, carbon fiber, activated carbon fibers from cotton which were activated by KOH and NaOH. There are differences on absorption peak position for the cotton before and carbonization processes, and after activated by KOH and NaOH.

New absorption area at the wave numbers of 521.30 cm$^{-1}$ and 613.20 cm$^{-1}$ emerged after the carbonization, which indicated the existence of functional group of NO2 and C=O, respectively. The peaks at the wavenumbers between 3700 cm$^{-1}$ to 2800 cm$^{-1}$ became widened and there was absorption sharpening at the wave number from 1700 cm$^{-1}$ to 500 cm$^{-1}$.

Overall, most of the peaks of carbon fibers after the activation by KOH 10% and NaOH 10% shifted to higher wavenumber with higher intensity. The increase of intensity proved that the atomic structure of the carbon fibers after being activated became more compact and the number of atoms inside the carbon fibers had increased.

The absorption area which occurred around the wave number of 1700 cm$^{-1}$ showed the existence of C=O function group [15]. The process of carbonization and activation induce new functional group indicated by the appearance of C=O function cluster at the wave numbers of 1718.99 cm$^{-1}$ and 1673.93 cm$^{-1}$, respectively. This result can be caused by the chemical reaction due to heating and activation.
processes of the carbon. The carbons react with the activating agent (KOH and NaOH) during the activation process then form new pores and generated carbon dioxide that diffused to the surface of the carbon [8].

The spectra of FTIR (Figure 4) showed the difference on functional groups of the carbon fibers and activated carbon fiber before and after passing of the fume indicated by the presence of new functional groups. In particular, on the carbonized cotton fibers, there new peak at 514.98 cm\(^{-1}\) indicated the presence of C-C=O functional group after being passed by the fume. On the other cases, also after being passed by the fume, two new peaks appeared at 1204.03 cm\(^{-1}\) and 504.89 cm\(^{-1}\) that correspond to absorption peak of C-O-C. Additionally, there were three new peaks at 677.26 cm\(^{-1}\), 1264.18 cm\(^{-1}\), and 1095.07 cm\(^{-1}\) associated with C=O and C-O, functional groups, respectively.

![FTIR spectra of activated carbon fibers](image)

**Figure 4.** The FTIR spectra of the activated carbon fibers (a) using KOH (b) using NaOH, and (c) before and after the fume adsorption.

3.3. **SEM Characterization**

Figure 5 shows the SEM image of carbon fibers with and without activation processes. The carbon fibers, prior to activation, had smaller size, irregular and nonhomogeneous fiber structure. However, activation using NaOH 10%, induced the larger size, regular and homogeneous structure of carbon fiber.

Figure 6 shows the change in the size distribution of carbon fibers. Before activation, the average diameter of the carbon fibers was 6.80 μm. Nevertheless, the diameter of fibers increased with average diameter up 10.17 μm due to activation. The increase of diameter was related to the chemical reaction
of cotton fibers with NaOH solution. Activated carbon fibers have larger average diameter as well as surface areas compare to cotton fiber. On the other hand, the carbonization process does not change the fiber diameter size [12].

![Image](a) ![Image](b)

**Figure 5.** Surface structure of the activated carbon fibers from cotton. (a) Before activation and (b) after activation using NaOH 10%.

![Image](a) ![Image](b)

**Figure 6.** The diameter distribution of the activated carbon fibers. (a) Before activation and (b) after activation using NaOH 10%.

The morphological structure and size distribution of pores of carbon fibers before and after activation are shown in Figures 7 and 8. Figure 8 shows the pore size distribution was dominant at the range between 0 – 500 nm and only few pores had size between 500-1000 nm with the average pore size 179.332 nm prior to activation. The activation process enhances the pore size and forms the new pores. The activation process using NaOH 10% resulted in the average pore size of 390.3 nm.
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Figure 7. The structure of the pores on the surface of the activated carbon fibers. (a) before and (b) after activation using NaOH 10%.

Figure 8. Pore size distribution of the activated carbon fibers. (a) before and (b) after activation using NaOH 10%.

4. Conclusions
It has been successfully synthesized activated carbon fibers from pyrolyzed and carbonized cotton at low temperatures using the oven as simple heating. In the process of carbonization, cotton fibers were successfully converted to carbon or charcoal within 4 hours with a temperature of 250°C. The activation process produces a uniform fiber structure and increases the pore size. Activation of carbon fiber increase the average diameter size distribution from 6.8 to 10.17 μm. FTIR analysis showed that activated carbon fiber after the fume adsorption caused the emergence of several new peaks at wave number of 1254.18 cm\(^{-1}\) and 1095.07 cm\(^{-1}\) that corresponds to absorption region of O-C = O and C-O-C, respectively.

5. References
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