

Preparation of activated carbon fabrics from cotton fabric precursor

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Abstract. The preparation of activated carbon fabrics (ACFs) from cotton fabric was performed by chemical activation with phosphoric acid (H_3PO_4). The operation conditions for obtaining the ACFs with the highest the adsorption capacity and process yield, proposed. Optimized conditions were: impregnation ratio of 2, the rate of temperature rising of $7.5\text{ }^\circ\text{C min}^{-1}$, the activation temperature of $500\text{ }^\circ\text{C}$ and the activation time of 30 min. The ACFs produced under optimized conditions was characterized by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The surface area and pore volume of carbon nanostructures was characterized by BET nitrogen adsorption isotherm at $77\text{ }^\circ\text{K}$. The pore size distribution calculated from the desorption branch according to BJH method. The iodine number of the prepared ACFs was determined by titration at $30\text{ }^\circ\text{C}$ based on the ASTM D4607-94. The results showed the improvement of porous structure, fabric shape, surface area ($690\text{ m}^2/\text{g}$), total pore volume ($0.3216\text{ cm}^3/\text{g}$), and well-preserved fibers integrity.

1. Introduction

Activated carbons are carbonaceous adsorbents with high specific surface area and developed porous structure in the forms of powder, granule, fibers, etc. Activated carbon fibers and cloths are commercially prepared from different precursors such as poly(acrylonitrile) (PAN) (1), Nomex (2), Kenaf, and viscous rayon (3). In the recent years activated carbon fabrics (ACFs) have gained increasing attention due to the comparative advantages over the traditional forms of activated carbon and potentialities for technological innovation in different fields. The advantages of activated carbon fabrics (ACFs) are faster adsorption kinetics, higher efficiency, and larger capacity for adsorption of different molecules and species. Powder and granule activated carbons generally have low flexibility, and it is difficult to use for preparation of activated carbon fabric without attaching to a web of polymer or similar materials (4). Studies in the literature concerned with preparation of activated carbon by physical and chemical activation processes. Wide variety of chemicals has been suggested for activation including phosphoric acid (the most popular agent), chloride salts of magnesium, ferric iron and aluminium, sodium carbonate, and sodium and calcium hydroxide. The common trait between all these chemicals is their strength as dehydrating agents (5). In order to produce activated carbon with desired properties, proper selection of experimental parameters such as impregnation ratio, the rate of temperature rising, activation temperature and activation time, etc. must be taken into



account. The aim of the present work was to study the effect of operational parameters on preparation of ACFs to maximize the adsorption capacity and yield using response surface methodology (RSM).

2. Material and methods

Cotton fabric composed of identical 100% cotton yard with a plain weave, a warp density of 19 yarns/cm⁻¹ and weft density of 13 yarns/cm⁻¹, H₃PO₄ solutions, solid iodine, hydrochloric acid, sodium thiosulfate and potassium iodide was used. The characterization of cotton fabric and 10 wt. % acid treated samples was carried out by non-isothermal thermogravimetric analysis (TGA).

The preparation of activated carbon fabrics (ACFs) from cotton fabric was performed by chemical activation with phosphoric acid (H₃PO₄). Experimental activation variables including; impregnation ratio, the rate of temperature rising, activation temperature and activation time was optimized based on the responses evaluated (adsorption capacity, yield of the produced ACFs). The operation conditions for obtaining the ACFs with the highest the adsorption capacity and process yield proposed. Optimized conditions were: impregnation ratio of 2, the rate of temperature rising of 7.5 °C min⁻¹, the activation temperature of 500 °C and the activation time of 30 min. The ACFs produced under optimized conditions was characterized by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The surface area and pore volume of carbon nanostructures was characterized by BET nitrogen adsorption isotherm at 77 °K. The pore size distribution calculated from the desorption branch according to BJH method. The iodine number of the prepared ACFs was determined by titration at 30 °C based on the ASTM D4607-94.

3. Results and discussion

3.1. Characterization of the ACFs prepared at optimized conditions

The TG curve of raw cotton fabric and H₃PO₄ impregnated cotton fabric presents in Figure 1. A weight loss of approximately 89 wt.% in 0-550 °C is observed for the raw cotton fabric, and the process can be divided into three steps: a little weight loss of 10 wt.% below 295 °C, due to the decrease of physically adsorbed water; a significant weight loss of 10- 72 wt.% from 295 to 370 °C, corresponding to the decomposition of hemicellulose, cellulose and lignin; a very slow weight loss of 72-89 wt.% at the third step in 370-550 °C, confirming the resultant char in this temperature range is thermally more stable.

Thermal degradation of the H₃PO₄ impregnated cotton fabric started at 50-150 °C for elimination of H₂O. After complete dehydration, another weight loss of materials performed between 160 and 200 °C, which carboxyl and methyl groups given off from hemicellulose. Higher weight fractions were reached in the range of 200-550 °C that relate to degradation of cellulose, hemicellulose and lignin at temperatures 295, 345, 375 °C respectively (6). It is believed that H₃PO₄ acts as a dehydrating catalyst, which can accelerate decomposition of the cellulosic precursors at lower heat treatment temperature (7). Furthermore, the presence of H₃PO₄ in the interior of the precursor prevents the formation of tar and chemicals by formation of cross-links, and stops the shrinkage of the precursor particle by engaging certain considerable volumes, resulting in the lower weight loss and higher. Using H₃PO₄ also proceeds activation through formation of phosphate and polyphosphate bridges which preserve crosslink biopolymer fragments and avoiding the shrinkage of the material by effect of temperature and removal of the activating material during the washing and finishing steps which lead to formation of matrix in an expanded state with an available pore structure (8).

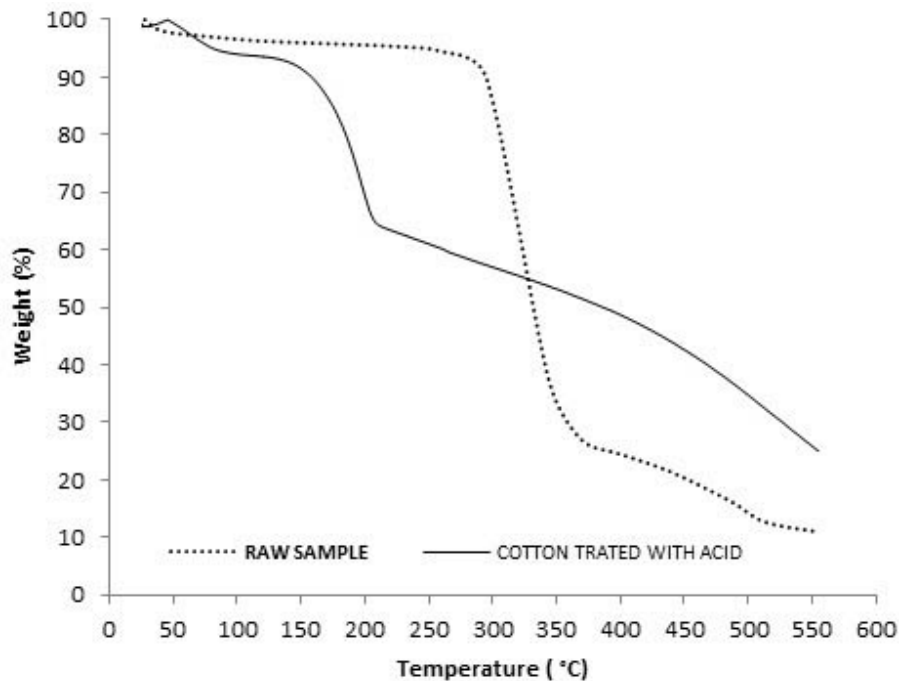


Figure 1. Thermogravimetric curve for the thermal degradation of raw cotton fabric and 10 wt % phosphoric acid treated sample under flowing N₂: variation of weight fraction (w) with temperature (T).

The SEM photographs are shown in Figure 2. demonstrate the preservation of fibers during carbonization and activation and retaining the structure and flexibility of AFCs.

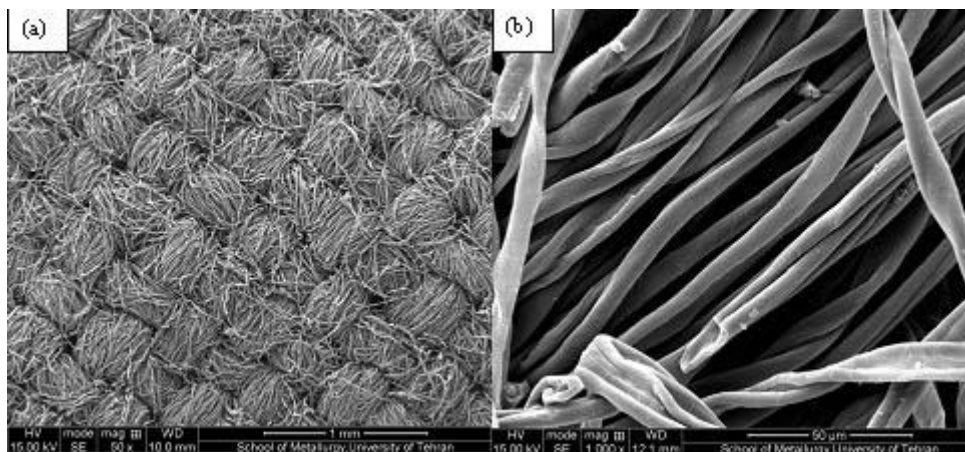


Figure 2. SEM images of ACF prepared at optimized conditions, bar length: 1mm (a); 500µm (b).

EDX analysis of the produced ACFs (Figure 3.) shows the sample mainly consist of carbon with an atomic fraction of 71 % followed by oxygen with an atomic fraction of 26.46 % and the carbonization of sample. The small amount of phosphorous (0.6 %) emphasis to the presence of phosphor groups on the surface of produced activated carbon.

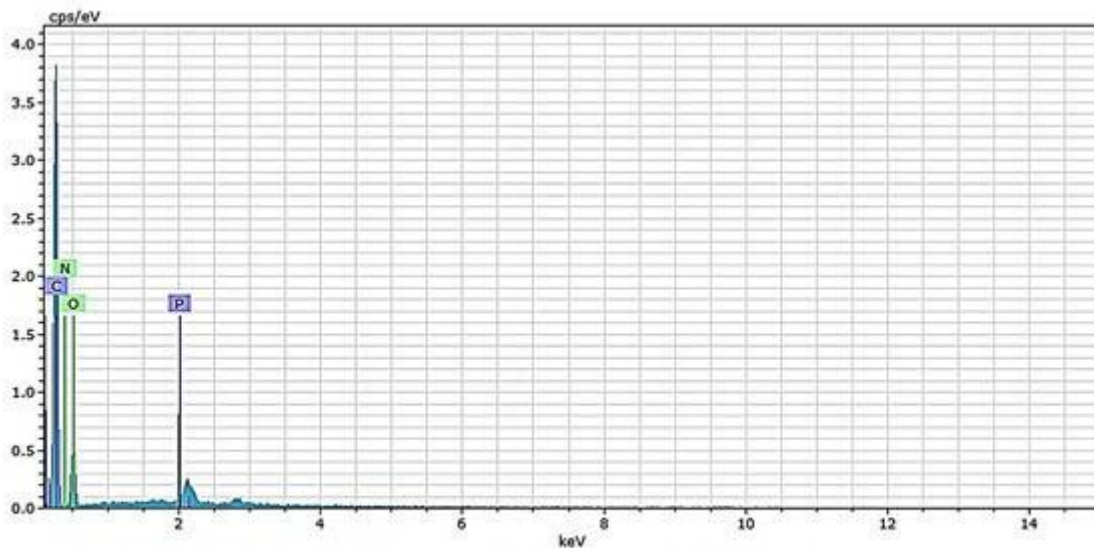


Figure 3. The EDX spectrum of produced ACFs at optimized conditions.

The Barrett-Joyner -Halenda (BJH) method for calculating pore size distributions is one of a family of methods based on a model of the adsorbent as a collection of cylindrical pores. It uses the modified Kelvin equation to relate the amount of adsorbate removed from the pores of the material, as the relative pressure (P/P_0) is decreased from a high to low value, to the size of the pores. Pore size distributions of the ACFs prepared at optimized conditions by BJH method are shown in Figure 4. The results showed the improvement of porous structure, fabric shape, surface area ($690 \text{ m}^2/\text{g}$), total pore volume ($0.3216 \text{ cm}^3/\text{g}$), and well-preserved fibers integrity. The plot represents microporosity, in which the majority of the pore sizes are in the range between 1.2 and 3 nm with an average pore diameter of 1.8621 nm, which indicates that the ACFs produced at optimized conditions has well-developed porous structure made of micropores.

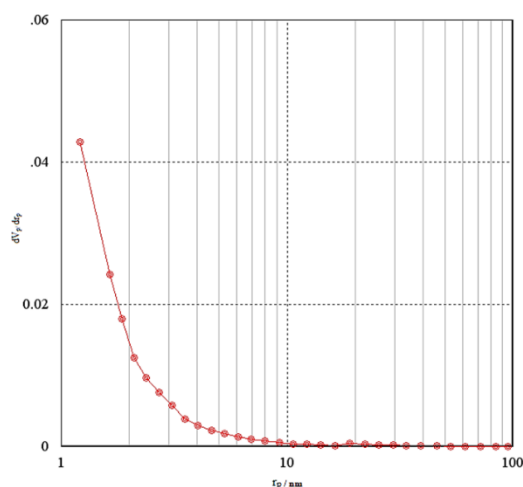


Figure 4. Pore size distributions of the ACFs prepared at optimized conditions by BJH method (dv_p/dr_p = the derivation of volume in pores of Radius to or larger than r (cc/gr), r_p = Pore Radius (nm))

Physical and chemical properties of activated carbon prepared at the optimum condition summarized in **Error! Reference source not found. 1**.

Table 1. Characteristics of ACF produced at optimized conditions.

Iodine number (Iodine number (mg I ₂ /g C)	595.21
BET specific surface area (m ² /g)	690.79
Total pore volume (cm ³ /g)	0.3216
Mean pore diameter (nm)	1.8621
Dimension Yield (%)	38.45
Weight Yield (%)	26.53

4. Conclusion

Based on the obtained results, Optimized conditions were: impregnation ratio of 2, the rate of temperature rising of 7.5 °C min⁻¹, the activation temperature of 500 °C and the activation time of 30 min. The results showed the improvement of porous structure, fabric shape, surface area (690 m²/g), total pore volume (0.3216 cm³/g), and well-preserved fibers integrity.

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