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Synthesis and Characterization of Activated Carbon from Corn Cobs using KOH Activation

Jinesh Verma[a], Rita Dahiya[a*], Vinay Kumar[a], Raman Devi[a] & Sachin Kumari[b]

[a]Department of Physics, COBS&H, CCS Haryana Agricultural University, Hisar, Haryana 125 004, India

[b]Department of Chemistry, COBS&H, CCS Haryana Agricultural University, Hisar, Haryana 125 004, India

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The enormous production of agricultural waste has created major problems for polluting the environment. Current efforts are focused on developing cost-effective and eco-friendly alternatives for disposal of waste materials. Agricultural wastes might be utilized as biochar and activated carbon (AC) precursors due to their higher carbon contents. In the present study, the activated carbon derived from corn cob biomass has been prepared successfully by the pyrolysis method at different temperatures *i.e.* 600 & 800 °C using potassium hydroxide activation. The synthesized materials (AC) were characterized using different techniques like X-ray diffraction, UV-Visible Spectroscopy, FTIR and N_2 adsorption/desorption isotherm. The FTIR study identified the functional groups and XRD analysis revealed the structure of the prepared material. The FE-SEM images showed that activation of biochar resulted into formation of porous activated carbon with various shapes and sizes of pores. The high surface area 575 m^2/g and pore volume 0.291 cm^3/g of AC at 800 °C temperature were observed with BET analysis. The optical band gap determined using UV-Vis absorption spectroscopy indicates that the absorption edge lies in the ultra-violet region of the optical spectra. The findings of the present study highlight the potential of utilizing agro-wastes as effective precursors for producing activated carbon with minimal expenses. This carbon variant shows promise in various applications such as water purification, metal recovery, energy devices, etc.

Keywords: Corn cob; Activated carbon; FE-SEM; FTIR; Pyrolysis

1 Introduction

To ensure food security to feed the rapidly growing population of the country, intensive agriculture has been widely adopted. The intensive agriculture resulted in generation of large amounts of biomass. India is amongst largest producer of agricultural waste countries and produces over 130 million tons of agricultural biomass, about half of which is used as fodder and remaining half is either piled up as wastes materials or burned in the field[1]. The agricultural waste management based bio-economic strategies have potential to solve the problem of burning of crop residues to ensure food and health security, waste valorization to generate value-added products, farmers' livelihood and eco-friendly agriculture[2]. The agricultural biomass is non-toxic, cost effective, renewable and composed of lignin, cellulose and lignocelluloses which might be beneficially utilized as raw material for preparation of value-added products such as bio-fuels, biochar and activated carbon. Such processes of converting waste into value added products offer alternatives to solve environmental problems[3]. Biochar is a carbon-rich black coloured porous solid which is produced from biomass via pyrolysis or hydrothermal process in the absence of oxygen. The activated carbon is usually produced from biochar with the help of activation process. There are two types of activation processes namely physical and chemical. The characteristics of AC depend on the methods of its preparations as well as on precursor materials. In India, annual crop residue production of major crops is 198 MT of rice, 173 MT of wheat, 62 MT of maize, etc. Similar to rice and wheat straw, 82.6% of the corn cobs are also burned contributing to environmental pollution[4]. The conversion of corn cobs into activated carbon offers an opportunity to convert wastes into value added products being an effective adsorbent for absorbing the different contaminants from the environment[5]. Keeping in view the above facts, the study was carried to synthesize activated carbon from corn cob biomass and its characterization.

2 Materials and methods

The corn cob derived AC was synthesized using pyrolysis process. The corn cobs were collected from

*Corresponding author: (E-mail: ritajbd@yahoo.com)

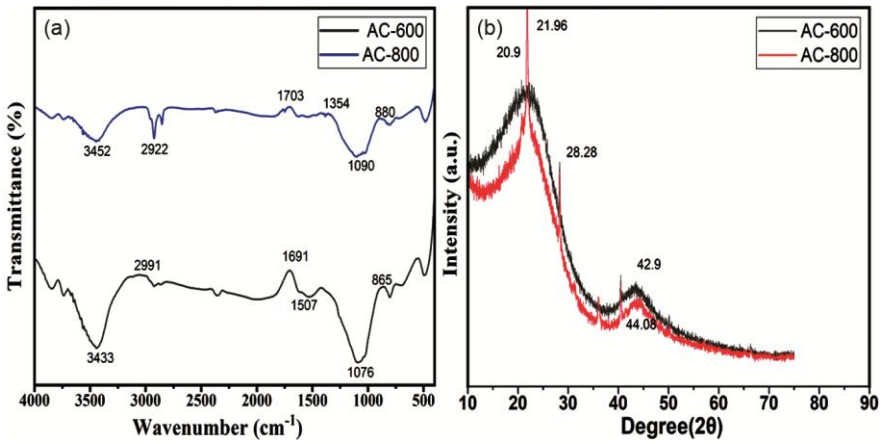

Fig. 1 — (a) FTIR and (b) XRD patterns of corn cobs activated carbon at 600 °C (AC–600) & 800 °C (AC–800).

field and cleaned to remove the dirt. After cleaning, the corn cobs were air dried and ground into the fine powder using grinder. The 30g powder was put in stainless-steel (SS) boat of tube furnace reactor. The biochar was prepared in furnace at 450 °C for 2 hours in absence of air at a fixed heating rate of 10 °C per minute. The biochar was collected from the SS boat at room temperature. The 3.5g of biochar was mixed with the 80ml solution of 5M KOH activating agent and the prepared mixture was put in the oven at temperature 90 °C for 3 hours. After 3 hours, the mixture was filtered and placed in the oven for 24 hours at 100 °C for removing the moisture. After being dried, the sample was pulverized with the help of mortar and pestle into a fine powder. The fine powder was put into a SS boat of a tube furnace reactor and carbonized with an argon gas flow (100 cm^3/min) at different temperatures viz. 600 and 800 °C with the heating rate of 5 °C/min and a residence time 2 hours. The activated product was collected from the SS boat at room temperature. The resulting activated carbon had been washed with a 1M HCl solution to remove surface ash particles, followed by distilled water to remove chemical compounds until the slurry attained a pH of 6-8. The prepared activated carbon was then dried at 110 °C for 12 hours in the oven. The characterization of the activated carbon was performed using different techniques such as Fourier Transform Infrared Spectroscopy **(**FTIR), X-ray Diffraction (XRD), UV-Visible spectroscopy (UV-Vis), Brunauer-Emmett-Teller (BET) and Field Emission Scanning Electron Microscopy (FESEM).

3 Results

The Fig. 1(a) represents the FTIR pattern of the activated carbon in the range of 4000-400 cm^{-1} which revealed the presence of various functional groups

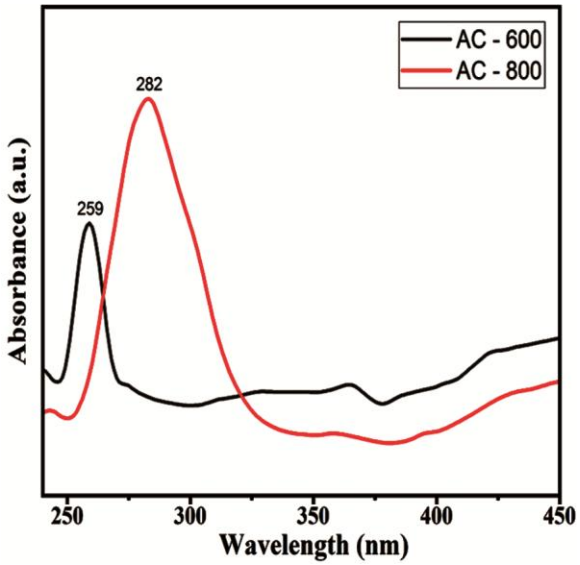

Fig. 2 — UV-Vis spectra of corn cobs activated carbon at 600 °C (AC–600) & 800 °C (AC–800).

such as phenol, alcohol and carboxylic acid present in the prepared AC. The XRD investigation was performed from 10° to 75° (2θ) values to determine crystal structure of corn cob activated carbon produced at temperatures 600 °C (AC-600) & 800 °C (AC-800). The XRD pattern [Fig. 1(b)] indicated the amorphous and semi-crystalline structure of the AC.

The UV-Vis spectra in Fig. 2 represented the dependency of the absorption peaks with the wavelength as well as on the carbon materials.

The specific surface area and pore volume of AC-600 and AC-800 were determined using BET technique and the surface area was confirmed with the profiles of isotherms shown in Fig. 3 (a,b).

The AC samples were further morphologically characterized by FESEM analysis as shown in Fig. 4 (a,b).

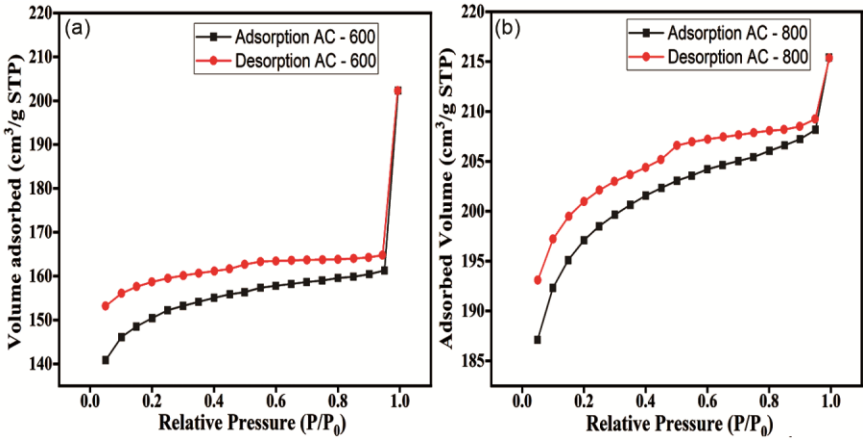

Fig. 3 — BET isotherm plots of corn cobs activated carbon at (a) 600 °C (AC–600) & (b) 800 °C (AC–800).

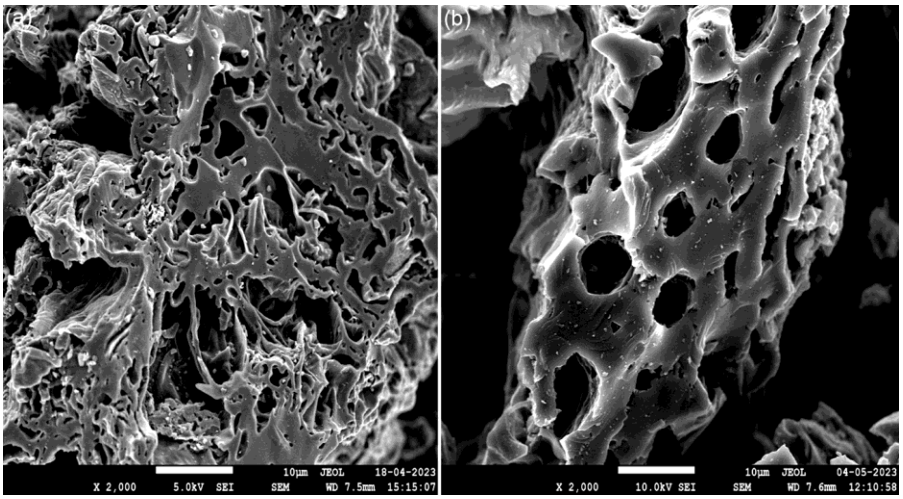

Fig. 4 — FESEM image of corn cobs activated carbon at (a) 600 °C (AC–600) & (b) 800 °C (AC–800).

4 Discussions

The FTIR peaks [Fig. 1(a)] observed in AC at 3500-3400 cm^{-1} indicated –OH stretching due to presence of phenol, alcohol and carboxylic containing hydroxyl groups. The prominent peaks at 2992 cm^{-1} ascribed to the presence of an aliphatic – CH stretching at 800 °C indicates the formation of aliphatic groups on decomposition of the AC at high temperature. The peaks at 1700-1500 cm^{-1} and 1150-1000 cm^{-1} in AC-600 and AC-800 indicate the formation of aromatic C=C rings, and C-O stretching of carboxylic acids and alcohols due to carbonization of the material[6]. The functional groups (*e.g.*, deprotonated carboxylic acids and phenols) favour sorption via cation exchange[7].

The XRD spectra of synthesized AC at 600 and 800 °C exhibit hump at 19.44° to 22.0° and 40.70° to 45.06° in Fig. 1(b). These results reveal that activated carbons have amorphous structure, but the peaks at 21.92°, 28.28°, 36°& 40.53° (2θ) correspond to semi-crystalline nature which increased with increase in temperature[8].

The UV-Vis spectra of synthesized AC-600 and AC-800 in Fig. 2 indicated that maximum adsorption wavelength increased with increasing temperature showing peaks in the region of 259-282 nm. The electrical transition between the bonding and antibonding π orbitals is associated with the UV absorption peak in materials containing carbon. The increase in absorption wavelength with increasing pyrolysis temperature from 600 °C to 800 °C might be due to increasing surface area of the activated carbon which is in concurrence with the BET surface area[8]. The surface area of AC has increased from 166 m^2/g to 575 m^2/g and the pore volume has increased from 0.099 to 0.291 cm^3/g as the temperature has increased from 600 to 800 °C, respectively. It was also confirmed from higher values

of adsorbed volume of N_2 gas for AC-800 as compared to AC-600 in plots of isotherm shown in Fig. 3 (a,b)[9]. A similar method was used by Medhat *et al*. (2021) to synthesize activated carbon from maize cobs of specific surface area 492 m^2/g. The FE-SEM images show that as the temperature increased from 600 to 800 °C, the pore size in AC also increased [Fig. 4(a & b)] at 2000x magnification with 10µm scale[10].

5 Conclusion

Conversion of agricultural waste biomass into value added products such as biochar and activated carbon offer environment friendly strategy to solve the problem of burning of crop residues. In the present study, corn cobs based activated carbon was prepared using pyrolysis process. The XRD analysis showed amorphous and semi-crystalline structure of activated carbon having large BET surface area of 575 m^2/g and pore volume of 0.291cm^3/g at 800 °C temperature. The FE-SEM images confirmed porous structure of the prepared activated carbon. The results of the present study suggest that corn cobs activated carbon due to its high surface area and presence of functional groups may be used for treatment of waste water, energy storage, cosmetics, pharmaceuticals, pollutant removal, etc.

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