

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON PREPARED FROM *BALSAMODENDRON CAUDATUM* WOOD WASTE THROUGH VARIOUS ACTIVATION PROCESSES

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ABSTRACT

Activated carbon prepared from *Balsamodendron caudatum* wood waste by various chemical processes shows excellent improvement in the surface characteristics. Surface morphology also plays a significant role in the adsorption properties along with surface functional groups. Activated carbon contains micropores, mesopores and macropores. The volume of these pores varies depending upon the activating agents used during various processes used. The resulting samples were characterized by nitrogen adsorption measurements at 77 K to obtain surface area and pore size distributions. The morphology of the resulting sample was studied by scanning electron microscopy and the surface functional group was investigated by Fourier transformation infrared spectroscopy techniques. Physico-Chemical characteristics such as bulk density, moisture content, ash content, matter soluble in water, matter soluble in acid, pH, iodine number, conductivity, porosity, pH_{zpc}, yield percentage and surface area have been carried out to assess the suitability of the carbon as adsorbent. Results of this investigation indicate that the activated carbon prepared using *Balsamodendron caudatum* wood waste by H₂SO₄ impregnation process followed by activation at 800 °C under nitrogen atmosphere yield activated carbon with the highest surface area and more developed micro, meso and macroporosity.

Keywords: *Balsamodendron caudatum* wood waste; Activated carbon; Carbonization, Surface area.

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INTRODUCTION

Activated carbons having high specific porosity, high surface areas are extremely versatile adsorbents of major industrial significance. These are used in wide range of applications concerned principally with the removal of species by adsorption from the liquid or gas phase. Activated carbons can be produced from a number of precursor materials including wood, agricultural wastes, coal and synthetic resins. These precursors are normally exposed to a number of different activation method such as physical or chemical in an effort to achieve- carbon with the high adsorption capacity for a particular application. For the past few decades, attention has been shifted towards adsorption technique, which emerged as one of the widely accepted methods for the removal contaminants from wastewater. Activated carbon adsorption has been cited by the US Environmental Protection Agency (USEPA) as one of the best available environmental pollution control technologies¹. One of the major challenges associated with adsorption using activated carbon is its cost-effectiveness. Researchers in the recent past have mainly focused on the preparation of the activated from agricultural waste materials as an alternative for the commercial activated carbon. Consequently, numerous low cost alternatives have been proposed including sago waste², waste coir pith³, pine sawdust⁴, sugarcane dust⁵, rubber wood sawdust⁶, bottom ash and de-oiled soya⁷. In recent years, a number of adsorptive material, such as moss peat⁸, melon seed husk⁹, tea factory waste¹⁰, sheep manure waste¹¹, etc., which have been fruitfully used for the preparation of activated carbon.

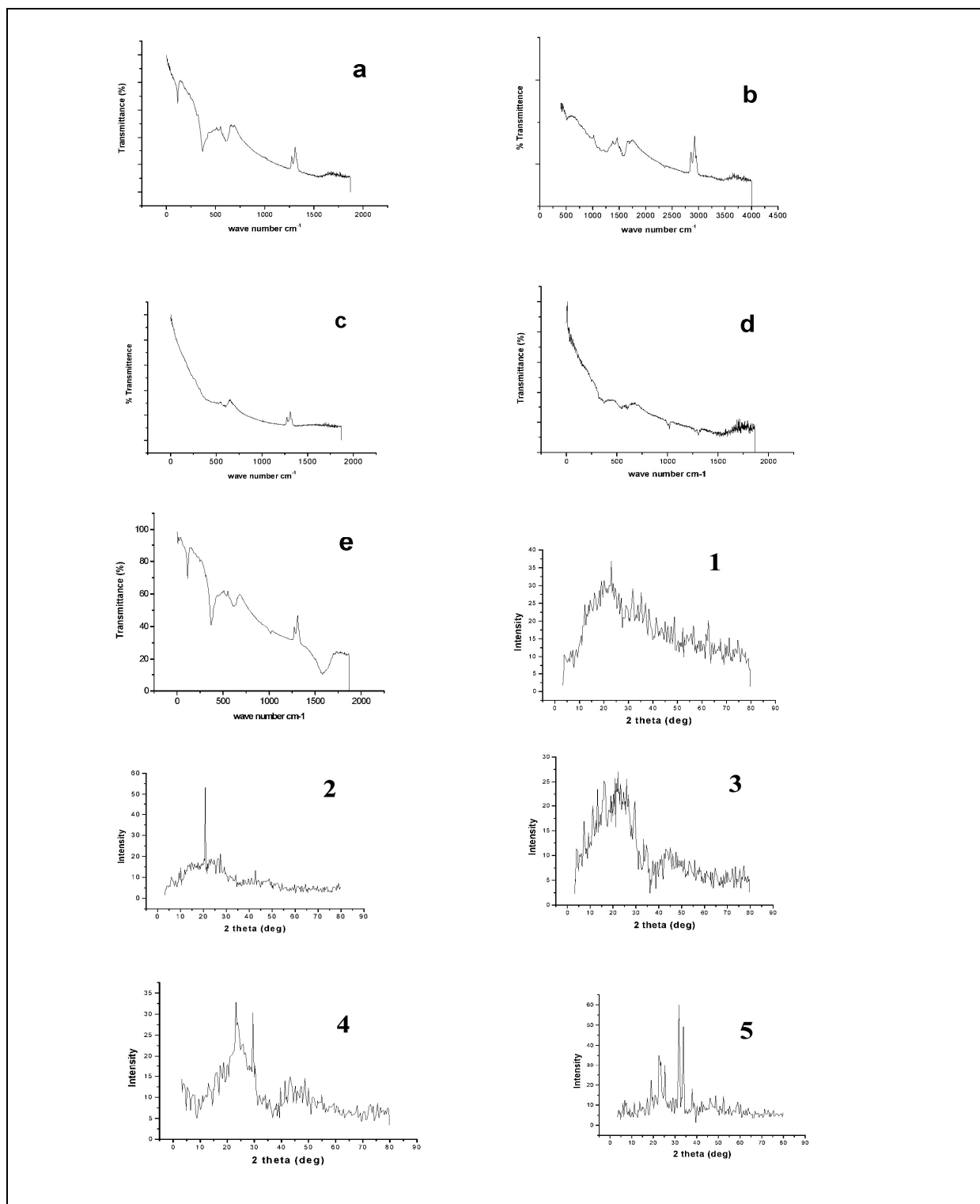


Fig.-1: FT-IR spectra of (a) BAC1, (b) BAC2, (c) BAC3, (d) BAC4, (e) BAC5 and XRD pattern of (1) BAC1, (2) BAC2, (3) BAC3, (4) BAC4, (5) BAC5.

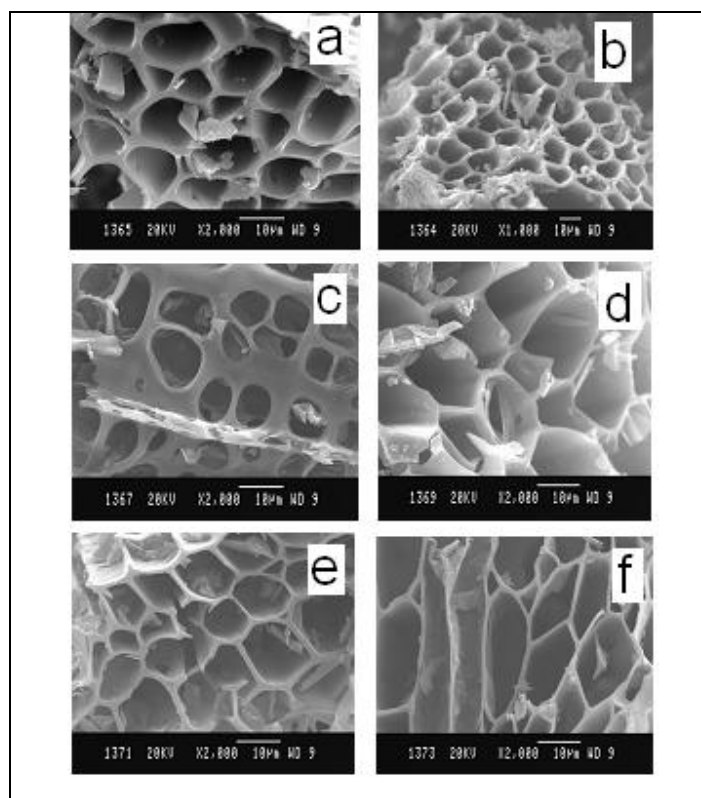


Fig. - 2: SEM Photograph of (a & b) BAC1, (c) BAC2, (d) BAC3, (e) BAC4 and (f) BAC5

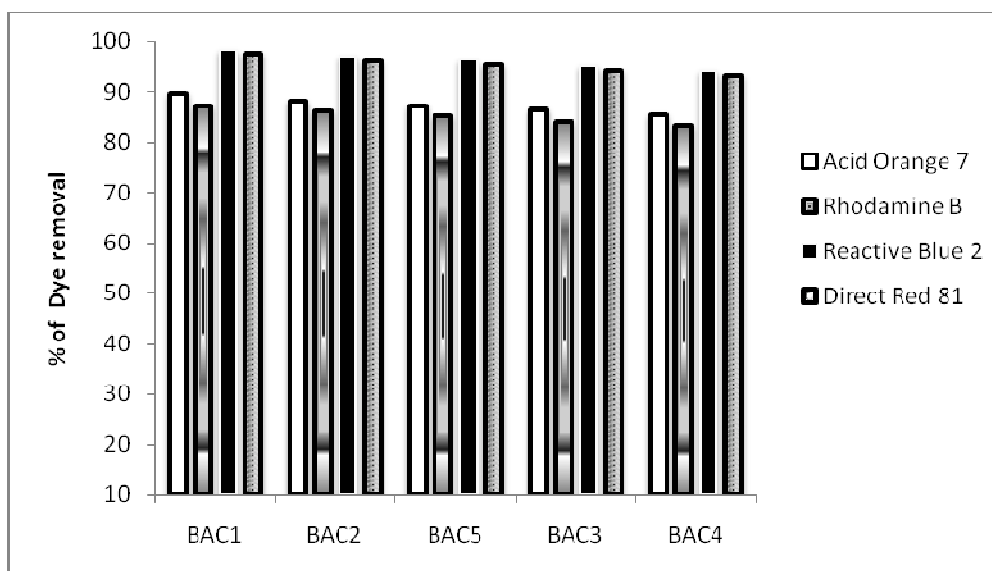


Fig.- 4: Percentage of dye removal by *Balsamodendron caudatum* wood waste activated Carbons.

The efforts do not go beyond some primary interpretations of the performance of the adsorbents in terms of their textural properties (porosity, surface area). More recently, some authors have started to interpret the surface chemistry of activated carbon with the adsorption performance¹². Therefore, carbons with

excellent surface properties and specific functionalities to be developed to create a high affinity for the adsorption of adsorbate in its solution. It will be beneficial to have an activated carbon with sufficient amount of super microporosity and mesoporosity for the enhanced solute adsorption. In this study, activated carbon derived from *Balsamodendron caudatum* wood waste by different processes is analyzed with various techniques such as Scanning Electron Microscopy (SEM) and Fourier Transformation Infrared Spectroscopy (FT-IR) in order to understand the properties. The objective of this paper is to study the physico-chemical characteristics of activated carbon prepared from *Balsamodendron caudatum* wood waste by various physical and chemical activation process with a view to use them in the treatment of wastewater.

EXPERIMENTAL

Carbonization and Activation

Acid Process

The dried material was soaked well with H_2SO_4 solution for a period of 24 hours. At the end of 24 hrs the excess of H_2SO_4 solution were decanted off and air-dried. Then the materials were placed in the muffle furnace carbonized at 120-130°C. The dried materials were powdered and activated in a muffle furnace kept at 800 °C for a period of 60 minutes. After activation, the carbon of obtained were washed sufficiently with large volume of water to remove free acid, Then the obtained material was washed with plenty of water to remove excess of acid, dried then to desired particle size. Another portion of the material was activated with activating agents H_3PO_4 as per the H_2SO_4 activation process described above, and they sieved to desired particle size. Final products obtained in each case were stored separately in a vacuum desiccator until used. The resulting carbons named as (BAC1 and BAC2).

Carbonization with Carbonate Salts

The *Balsamodendron caudatum* wood waste to be carbonized was soaked with 10% Sodium carbonate solution for a period of 24 hours. After impregnation, the liquid portion was decanted off and the material dried. The dried mass was subjected to carbonization process at 400 °C, then powdered well and finally activated at a temperature of 800 °C for a period of 20 minutes. The resulting carbon named as BAC3.

Dolomite Process

A sufficient quantity of dried *Balsamodendron caudatum* wood waste was taken over a calcium carbonate bed and the upper layer of the waste was also covered with a layer of Calcium Carbonate. The whole material was carbonized at 400 °C, powdered well and followed by the thermal activation at 800 °C. After the activation, the material was repeatedly washed with plenty of water to remove calcium carbonate and dried at 110°C. The resulting carbon named as BAC4.

Carbonization with Sulphate salts

In this method the *Balsamodendron caudatum* wood waste to be carbonized were soaked in 10% solutions of sodium sulphate for a period of 24 hours. After impregnation, the liquid portion was decanted off and then dried. The dried mass was subjected to carbonization process at 400 °C, powdered well and finally thermally activated at 800 °C for a period of 10 minutes. The resulting carbon named as BAC5.

Characterization of the Activated Carbon

pH and conductivity were analyzed using Elico make pH meter (model L1 -120) and conductivity meter (model M-180), respectively. Moisture content (%) by mass. Ash (on dry basis) % by mass, matter soluble in water, matter soluble in acid, pH, iodine number, conductivity, porosity, pH_{zpc}, Yield percentage and surface area were analyzed as per standard procedures¹³.

Surface area and Pore size distribution Analysis

The N_2 adsorption-desorption isotherms of activated carbon were measured at 77K using N_2 gas sorption analyzer (Nova 1000, Quanta Chrome Corporation) in order to determine the surface area and total pore volume. The surface area calculated using the BET equation. In addition, the t-plot method applied to

calculate the micropore volume and external surface area (Mesoporous Surface area). The total pore volume estimated using liquid volume of adsorbate (N_2) at a relative pressure of 0.99. All the surface area calculated from the nitrogen adsorption isotherms by assuming the area of a nitrogen molecule was 0.162 nm^2 .

FT-IR spectra and SEM

The electronic structure of carbon samples were examined using FT-IR 1725 x (perkin-Elmer) spectrometer. The measurements were carried out over the range $4000\text{-}400\text{cm}^{-1}$. Carbon samples (0.33 wt%) were stirred with dry KBr (Merk, spectroscopy grade) and then pressed to form appropriate tablets. The surface morphology of carbon samples observed with SEM (HITACHI S3000N).

Table-1: Activated carbon prepared from *Balsamodendron caudatum* wood waste by different activation Processes

Sample	Treatment Process
BAC1	H_2SO_4 Process + Thermal Activation under N_2 flow
BAC2	H_3PO_4 Process + Thermal Activation under N_2 flow
BAC3	Na_2CO_3 Process + Thermal Activation under N_2 flow
BAC4	$CaCO_3$ Process + Thermal Activation under N_2 flow
BAC5	Na_2SO_4 Process + Thermal Activation under N_2 flow

Table- 2 : *Balsamodendron caudatum* wood waste Activated Carbons Properties

S.No	Properties	Activated Carbons				
		BAC1	BAC2	BAC3	BAC4	BAC5
1	pH	6.4	6.5	6.12	9.52	8.21
2	Moisture content (%)	4.91	6.17	4.92	5.6	5.71
3	Ash Content (%)	2.39	2.09	2.89	3.01	2.45
4	Conductivity (ms /cm)	0.55	0.44	0.22	0.32	0.41
5	Specific Gravity	1.40	1.30	0.84	1.35	1.36
6	Bulk Density (g/mL)	0.47	0.45	0.72	0.46	0.44
7	Porosity (%)	49.19	43.64	26.18	44.60	43.61
8	Matter Soluble in water (%)	0.90	0.80	1.20	1.91	2.21
9	Matter Soluble in 0.25M HCl (%)	1.19	1.16	0.95	1.85	1.52
10	Surface Area (m^2/g)	505	458	300.71	240.42	339.64
11	Iodine Number (mg/g)	453	212	240	203	248
12	pH_{zpc}	4.9	4.2	4.0	4.1	3.91
13	Yield (%)	52	43	41	62	34

RESULTS AND DISCUSSION

From Table 2 the moisture content percentage of BAC1 found to be high, it shows that extensive porosity introduced by H_2SO_4 process in the carbon structure. When compare to all processes, the

moisture content of activated carbon prepared by sodium carbonate process was less. The ash content values from Table 2 indicate that the overall ash content for all carbonization processes have lesser values. This attributed to lower inorganic content and higher fixed carbon. The orders of surface area of carbons prepared by various processes are in order of BAC1 > BAC2 > BAC5 > BAC3 > BAC4. The higher surface area of carbon BAC1 prepared by H₂SO₄ processes (acid process) may due to the restricted pore shrinkage during activation. The H₂SO₄ process shows high iodine number value. This indicates that the carbon BAC1 have the maximum adsorption capacity. Carbons with high surface area considered the most superior for adsorption of organic substances¹⁴. All carbon samples show significant nitrogen uptake at low relative pressure. That can be ascribed to the strong interaction between nitrogen molecules and the wall with closely spaced pores. Nitrogen adsorption for the BAC3, BAC4 and BAC5 samples were low since the samples have such a low degree of activation and a low pore volume¹⁵. The highest surface area obtained for activated carbon prepared using *Balsamodendron caudatum* wood waste waste by sulphuric acid process followed by activation at 800 °C under a nitrogen atmosphere (505 m²g⁻¹).

The FT-IR spectrum of the *Balsamodendron caudatum* wood waste activated carbon prepared by various treatment processes shown in the Figurers 1(a-e) revealed that, but not all, of the carbons evaluated contain four classes of surface oxides: carboxyls, lactones, phenols and carbonyls. The concentration of the surface groups varied, depending on the various types of activation conditions. The assignment of the specific wave number to a given functional group was not possible because the adsorption bands of various functional groups overlap and shift depending on their molecular structure and environment. Shifts in absorption position may be caused by factors such as intramolecular and intermolecular hydrogen bonding, steric effect and degree of-conjugation. For instance, within its given range, the position of C=O stretching band (common to carbonyls, carboxylic acids and lactones) is determined by many factors, such as:

1. The physical state
2. Electronic and mass effects of neighbouring substituent's
3. Conjugation
4. Hydrogen bonding and
5. Ring strain¹⁶.

The FT-IR absorption bands of oxygen groups on the surface of activated carbon prepared using *Balsamodendron caudatum* wood waste by various processes were likely to be affected by some or all of the factors listed above. Most of the carbons exhibit similar IR spectroscopic features; those are very intense/sharp -OH stretching of carboxyl, phenol and alcohol vibration around 1100 cm⁻¹ and aliphatic C-H stretching absorption around 2800 cm⁻¹. Saturated aliphatic ethers show a strong band in the region 1108.69 cm⁻¹ was attributed to carbonyl groups, and broad band in the region 1500 to 1900 cm⁻¹ due to C=O stretching. The group of bands appeared in the region 2778.46 cm⁻¹ corresponding to -CH₂ groups. The broad band observed in the region of 1000 to 1250 cm⁻¹ was assigned due to a characteristic absorption of -OH group. These results are in good agreement with the findings of many investigators¹⁷. The morphological study by SEM of the above adsorbents shown in the Figurers 1(1-5) revealed that, it is highly porous in nature. From the SEM results, it was found that there are holes and cave type openings on the surface of the specimen that would definitely have increased the surface that are available for the adsorption¹⁸. Figurers 3(a-e) shows the wide angle XRD patterns for five carbon samples. The higher and sharper (002) peak for BAC1 indicates a higher degree of graphitization.

Assessment of Activated carbon samples for the removal of dyes

The acid, reactive and direct dyes have anionic character and basic dyes are cationic character. Figure 4 shows the percentage of anionic and cationic dye removal for five different activated carbons prepared from *Balsamodendron caudatum* wood waste. A secure connection between surface area and surface groups of the customized activated carbons and percentage of dye removal through adsorption can be observed. Five types of activated carbons prepared from *Balsamodendron caudatum* wood waste carbon, a similar behavior was observed for anionic dyes (*i.e* reactive, direct and acid dyes) following an increase

in the adsorption capacity with increase in the surface area. Different performances are obtained for the materials tested, varying from 98.4 % to 89.6 % (for sample BAC1) of anionic dye removal for the *Balsamodendron caudatum* wood waste activated carbon. Sample BAC1 is the most efficient material for the adsorption of all the anionic dyes. BAC1 which has higher surface area than BAC4 adsorbs larger amount anionic dyes.

CONCLUSION

Activated carbons with moderate surface area obtained from *Balsamodendron caudatum* wood waste. The difference in textural characteristics related to the activation processes. The highest surface area obtained for activated carbon prepared using *Balsamodendron caudatum* wood waste by H₂SO₄ process followed by activation at 800 °C under a nitrogen atmosphere (505 m²g⁻¹). The concentration of the surface groups varied, depending on the various types of activation conditions. Among the five carbons investigated, activated carbon prepared by H₂SO₄ process followed by N₂ gas activation method showed the highest concentration of surface groups. Carbon prepared from above process and materials conveniently used for both organic and inorganic effluent removal. Based on the surface area, the following activated carbons / processes are compared with commercially available activated carbons: Such as BAC1 Process, BAC2 Process, BAC3 Process, BAC4 Process and BAC5 Process. These carbons can be used for textile effluent removal because of capable of adsorbing organics from the solution. The adsorption of dyes with BAC1 Process is more effective than other processes.

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