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Preparation and Characterization of Activated Carbon from *Caesalpinia pulcherrima* Pod

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Abstract

Activated carbon prepared from Caesalpinia pulcherrima pod (CPP) by chemical process shows an excellent improvement in the surface characteristics. Surface morphology plays a very important role in the adsorption properties along with surface functional groups. For the characterization of the activated carbons SEM, FTIR and XRD analyses were performed. The surface morphology porosity of activated carbon (activated carbon from Caesalpinia pulcherrima pod) was visualized via scanning electron microscopy (SEM) analysis. Surface functional groups were detected by Fourier transformation infrared (FTIR) spectroscopy techniques. The results of this investigation indicate that the activated carbon prepared by using Caesalpinia pulcherrima pod (CPP) is a low cost, ecofriendly adsorbent , is expected to be environmentally and economically feasible.

Keywords: *Activated carbon; Caesalpinia pulcherrima pod (CPP); FTIR; SEM.*

1. INTRODUCTION

Dyes and pigments are widely used, mostly in the textiles, paper, food, plastics, leather, and rubber, industry in order to colour their products (Robinson *et al.* 2002; Ramakrishana *et al*. 1997; Nigam *et al.* 2000). Considerable amounts of synthetic dyes are lost annually to waste streams during textile processing, which ultimately enters the environment. The presence of small amounts of dyes in water is highly visible and undesirable (Crini *et al.* 2006). Most of the commercial dyes are chemically stable and are difficult to be removed from wastewater. Various kinds of dyes and their break down products are toxic for the living organisms. The release of this coloured wastewater is hazardous for the aquatic life. At the receiving streams, the coloured wastes interfere with the transmission of sunlight in to streams and reduce the photosynthetic activity. In addition, some dyes or their metabolites are either toxic or mutagenic and carcinogenic (Chen *et al.* 2006; Heiss *et al.* 1992). Dyes are recalcitrant molecules, which are difficult to degrade biologically. The removal of color from wastewaters can be carried out by flotation, chemical coagulation, chemical oxidation or ozonation, membrane separation and activated carbon sorption. Adsorption technique is most widely used method because of its low cost, ease of operation and availability of a wide range of adsorbents and proved to be an effective and attractive process for removal of non-biodegradable pollutants (including dyes) from wastewater (Aksu *et al.* 2005).

The coal and agricultural byproducts of lignocelluloses materials are two main sources for the production of commercial activated carbons. Agricultural wastes have emerged as a better choice and can be used as adsorbents without further treatment, activation could enhance their adsorption capacity. The production of activated carbons from agricultural wastes convert unwanted, surplus agricultural waste of which billions of kilograms are produced annually to useful valuable adsorbents such as rice husk (Kumar *et al.* 2006), orange waste (Dhakal *et al.* 2005), coir pith (Kadirvelu *et al*. 2001; Shanthi *et al.* 2014), saw dust (Taty-Costodes *et al.* 2003b), peanut hulls (Brown *et al*. 2000), egg shell, maize bran, chitin. The aim of this study is to investigate the preparation and characterization of *Caesalpinia pulcherrima* pod (CPP) activated carbon, which is a low cost adsorbent for the removal of dyes.

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2. MATERIALS & METHODS

2.1 Preparation of the adsorbent

The *Caesalpinia pulcherrima* pod (CPP) were collected, and thoroughly washed with distilled water two or three times to remove dust and impurities. The pods were cut in to smaller pieces, dried and powdered.The powdered raw material was chemically activated by treating with concentrated sulphuric acid with constant stirring and kept for 24 hours. The carbonized material obtained was washed well with plenty of water several times to remove excess acid and dried at 105-110 °C in a hot air oven for 5 hours. The adsorbent thus obtained was ground well and kept in an airtight container for further use.

2.2 Characterization of activated carbon

2.2.1 Fourier transform infrared spectroscopy (FTIR)

FTIR is perhaps the most powerful tool for identifying the types of chemical bond (functional groups). The wavelength of light absorbed is characteristic of the chemical bond and can be seen in this annotated spectrum. Chemical bonds in a molecule can be determined by interpreting the infrared absorption spectrum. The spectra of the samples were recorded between 400 cm^{-1} and 4000 cm^{-1} using Thermo Nicolet, Avatar 370 spectrometer (Thermo Nicolet Inc., USA) to analyze the functional groups of the activated carbon. About 0.1 g of sample was mixed with 1 g of KBr, Spectroscopy grade (Merck, Germany), in a mortar. Part of this mix was introduced in a cell connected to a piston of a hydraulic pump giving a compression pressure of 15 kPa cm^{-2}. Then it was transferred to the FTIR analyser and a corresponding chromatogram was obtained showing the wavelengths of the different functional groups in the samples, which were identified by comparing these values with those in the library (Griffiths, 1986).

2.2.2 X-ray diffraction measurement (XRD)

X-ray scattering techniques are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting sample as a function of incident and scattered angle, polarization and wavelength or energy. The activated carbon were grinded to form fine powders and then pressed lightly on a stainless steel holder using a carbon conducting tape. XRD was performed at D8 Advanced XRD, Bruker and the spectra were analyzed using Origin 6.0 software. The crystallite domainsize was calculated from the width of the XRD peaks, assuming that they are free from non-uniformstrains, using Scherrer's formula.

$$D = 0.94\,\lambda / \beta \cos\theta$$

where D is the average crystallite domain size perpendicular to the reflecting planes, λ is the X-ray wavelength, β is the full width at half maximum (FWHM) and θ is the diffraction angle. To eliminate additional instrumental broadening the FWHM was corrected, using the FWHM from a large grained Si sample.

$$\beta\ \text{corrected} = (\text{FWHM}^2\ \text{sample} - \text{FWHM}^2\text{si})^{1/2}$$

The above modified formula is valid only when the crystallite size is smaller than 100 nm (Cullity, 1978).

2.2.3 Scanning electron microscopy (SEM)

Scanning Electron Microscopic (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. Morphology, size and structures of the particle were examined using scanning electron microscopy (SEM) (Model 6093, JEOL).

3. RESULT & DISCUSSION

3.1. FTIR analysis of activated carbon

Fig.1. shows FTIR results of *Caesalpinia pulcherrima* pod (CPP) activated carbon.

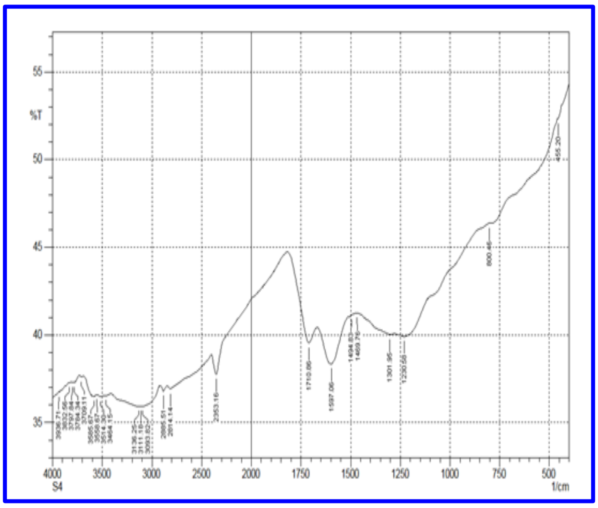

Fig. 1: FTIR analysis of *Caesalpinia pulcherrima* pod (CPP) activated carbon

The peaks at 3464.15 cm^{-1} are characteristic of -OH stretching vibrations of alcohol group, 1597.06 cm^{-1} characteristic of C=C stretching vibrations of aromatic group, 1710.86 cm^{-1} characteristic of C=O ketone group and 2814.14 cm^{-1} characteristic of C-H stretching vibrations of alkane group.

Yang *et al.* (2003) reported that the sample showed three major absorption bands at 2900-3500 cm^{-1}, 1300-1750 cm^{-1} and 1000-1250 cm^{-1}. A wide band with two maximum peaks can be noticed at 2930 and 3450 cm^{-1}. The band at 3450 cm^{-1} is due to the absorption of water molecules as result of O-H stretching mode of hydroxyl groups and adsorbed water, while the band at 2930 cm^{-1} is attributed to C-H interaction with the surface of the carbon. However, it must be indicated that the bands in the range of 3200-3650 cm^{-1} have been attributed to the hydrogen-bonded OH group of alcohols and phenols. Similar results were observed by Puziy *et al.* (2002).

3.2 XRD analysis of activated carbon

The X-ray diffraction study of activated carbon was carried out using X-ray diffractometer. The amorphous nature of the activated carbon was determined by using the intensity of the observed rays with respect to scattering angle (2θ). The XRD pattern of typical amorphous carbon showed broad asymmetric peaks corresponding to 2θ = 24.4°.

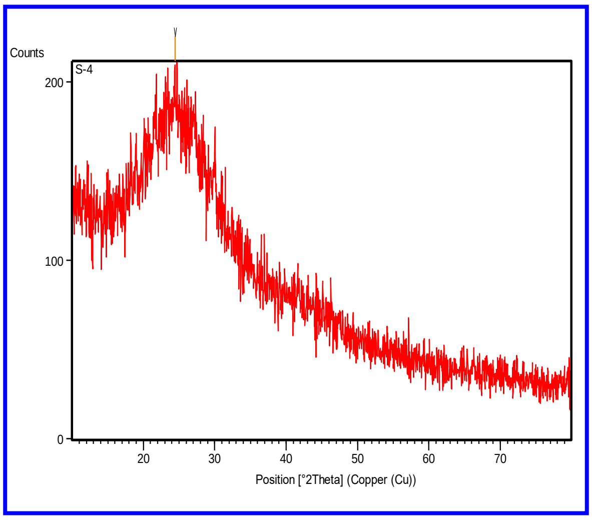

Fig. 2: XRD analysis of *Caesalpinia pulcherrima* pod (CPP) activated carbon

3.3 SEM analysis of activated carbon

The SEM image of the activated carbon is shown in Fig.3. It was noticed that the surface morphology of the AC is heterogenous pores and relatively smooth in nature. Similar results have been reported by Ghaedi *et al.* (2012). Tuan *et al.* (2011), suggested that, activated carbon samples consists of many particles with random form and sizes.

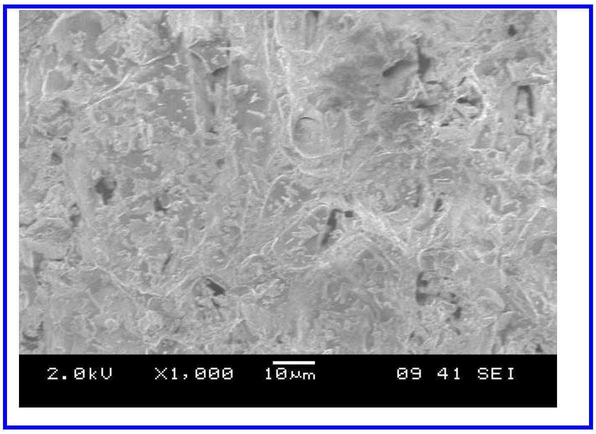

Fig. 3: SEM analysis of Caesalpinia pulcherrima pod (CPP) activated carbon

4. CONCLUSION

The present study shows that the waste material, *Caesalpinia pulcherrima* pod (CPP), was prepared ,characterized and can be effectively used as an adsorbent for the removal of the dyes from its aqueous solutions. Activated carbon is the most commonly used adsorbent for the removal of various pollutants from wastewaters.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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