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Research Paper

PREPARATION AND CHARACTERIZATION OF PLANT BASED LOW COST ADSORBENTS

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Abstract

The objectives of present work based to prepare the plant based adsorbents from pine needle (PN) and lantana shoot (LS). The adsorbent prepared without any physical or chemical treatment. The mesh size 75-150 μm of different sieve was selected for both adsorbents. The physico-chemical parameters of adsorbents were pH, moisture content, ash content, bulk density and elemental analysis. The surface morphology of adsorbent surface was analyzed by Scanning Electron Microscopy (SEM). The chemical composition of adsorbent was determined by Energy Dispersive X-ray Spectroscopy (EDX). The surface chemical nature of adsorbents was studied by Fourier Transform Infrared Spectroscopy (FTIR), which shows functional groups on adsorbents surface. The present research work indicated that both PS and LS adsorbents could be employed as low cost adsorbents in removal of toxic pollutants from water and wastewater.

Key words: Characterization, low cost adsorbents, SEM-EDX, FTIR, plant biomass.

INTRODUCTION

Adsorption technique played a major role in treatment of various types of water and wastewater. Many wastewater treatment techniques are used worldwide for pollutant removal from aqueous solution such as reverse osmosis, coagulation, membrane filtration, ion exchange and adsorption. Among all techniques, adsorption is considered as most suitable process because of its simplicity and cost effective [1].

Adsorbents are widely applied in removal of organic pollutants from water and wastewater [2]. The most common adsorbent is activated carbons which contain properties like high porosity, large surface area, and high carbon content. Activated carbons are most used as water purified adsorbents in industrial and domestic sectors but they are not low cost hence, there is need to develop the low cost adsorbents derived from plant and agricultural biomass. The cost of

naturally occurring biosorbent is quite low as compared to the cost of commercially available adsorbents in waste water purification.

Among many adsorbents, naturally available plant biomass commonly used as low cost adsorbents. Most of the adsorbents are used without any treatment such as bamboo husk [3] blue pine [4], walnut [4], fruit cortex [5], banana peel [6], coffee husk [7]. The plant based adsorbents are lignocellulosic materials and applied in the removal of heavy metals, dyes, phenol and other organic pollutants. Generally, these materials are considered as low cost due to cheap in nature, wider and locally availability, and most abundant in nature. The pine (*Pinus roxburghii*) needle considered waste in pine forest in hilly areas. These plant waste cause forest fire and make soil acidic. The forest waste disposal is mandatory for the point of environmental conservation. Adsorbents prepared from pine needle biomass reduce the impact of waste disposal. Lantana (*Lantana camara*), a noxious weed, grows in many reason of the world it poses major threats to environment due to allelopathy action [8].

This paper aims to study of plant based adsorbents derived from pine needle and lantana shoots. Physico-chemical characterization was also analyzed, for a potential production of low cost adsorbents. The characterization was studied by parameters such as ash content, moisture content, bulk density, pH, elemental analysis, SEM-EDX, and FTIR.

MATERIAL AND METHODS

Collection and preparation of material

Pine needles were collected from Garhi Cantt, Dehradun, India and lantana shoots were collected from the road side areas of Jagjeetpur, Haridwar, India. The materials were left in open sun drying for 3-4 days. Then, the materials were washed with double distilled water several times to remove impurities. After that materials were again dried in oven at $105~^{\circ}\text{C}$ upto 24 hours. Both raw material were crushed into small pieces and prepared fine grind powder by use of mixing grinder. After grinding the material, they were sieved (75-150 μ m) and used for characterization without any prior chemical treatment.

Sieving

For sieving purpose, the materials were passed through the 150-300 mesh sieve plates.

pН

 $1\ gm$ of sample was added in $100\ mL$ double distilled water. The mixture was stirred upto constant pH occurred. The pH was determined by digital pH meter.

Moisture content

For the determination of moisture content, 1 gm sample was weighted and dried in an oven for 24 hour at 105 °C, until the constant weight gained. The moisture content was determined by following formula:

Moisture content = W_i - W_f / $W_i \times 100$

Where,

W_i= initial weight of adsorbent (g)

W_f= final weight of adsorbent after drying (g)

Ash content

Ash content was determined with the help of muffle furnace, 1 gm of each sample placed into a porcelain crucible and heated in muffle furnace set a temperature of 500 ° C for upto 4 hours. The material was allowed to cool in desiccators for several minutes. Then, the ash content (%) was calculated from following formula:

Ash content (%) = W_2 - W_0/W_1 - $W_0 \times 100$

Where,

 W_0 = weight of empty crucible (g)

W₁= crucible + adsorbent weight (g)

W₂= crucible + ashed sample weight (g)

Bulk density

Bulk density generally represents the flow consistency and packaging quantity of solid sample. This was determined by the measuring dried cylinder and the sample was packed inside the measuring cylinder, and weighed. The weight of the sample packed in measuring cylinder was determined from the difference in weight of the empty and adsorbent filled measuring cylinder. Then, the material was placed into the aluminium plate and then dried in oven $(105\,^{\circ}\text{C})$. The bulk density was measured by following formula:

Bulk density= M₂-M₁/V

Where.

M₂= weight of measuring cylinder (gm)

 M_1 = weight of measuring cylinder + material (gm)

V= volume of measuring cylinder (L)

Elemental analysis

The elemental analyses of both adsorbents were performed by using a CHNS/O elemental analyzer. The chemical characterization of adsorbent is determined by elemental analysis technique.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX)

For the surface morphology determination SEM, SEM-EDX performed. The fine sample was dried in oven at 105° C for 24 hour. Then, the sample was analyzed by SEM instrument (JEOL Japan-6490). EDS was also analyzed by EDS (EDS-133) instrument.

FTIR (Fourier Transform Infrared Spectroscopy)

The FTIR studies were performed to determine the various functional groups on the surface of the material. The instrument used to record the FTIR spectra by FTIR instrument with KBr as reference. The spectrum was recorded by using Fourier Transform Infrared (Shimadzu, Japan) spectrophotometer in a spectral range of 400-4000cm⁻¹.

RESULTS AND DISCUSSION

The physico-chemical characteristics of pine needles and lantana shoots are mentioned in Table 1. As shown in table 1, pH analysis of PN adsorbent reported slightly acidic (6.3) in nature, where as LS shows alkaline natural pH (8.0).

The moisture content tended to be low for both adsorbents (< 1%) showing that these adsorbents were properly prepared and handheld. It was reported 0.46 percent for PS and 0.18 percent for LS. The ash content was reported higher in lantana (11%) as compared to pine (4.81). Ash content lower values favour the good adsorbent characteristics. The sieving of adsorbents was also analyzed by sieving plates.

The bulk density is determines the mass of adsorbent in a specific volume. The bulk density of PN and LW were calculated 0.34 g/cm³ and 0.22 g/cm³, respectively.

Table 1 Physico-chemical characteristics of adsorbents

Parameter	Pine needle (PN)	Lantana shoot (LS)
pН	6.35	8.0
Moisture content (%)	0.46	0.18
Ash content (%)	4.81	11.0
Mesh size (µm)	75-150	75-150
Bulk density (gm/cm ³)	0.34	0.22
N(%)	0.879	0.834
C(%)	48.16	44.32
S(%)	-	-
H(%)	8.848	7.515

SEM analysis

Scanning Electron Microscopy (SEM) reveals the structure and behaviour of the PN and LS adsorbents. Fig. 1 shows the SEM micrograph of a magnification of 3000 respectively. The surface of PN adsorbent showed several irregular structures with deep cavities whereas LS adsorbent surface area is also irregular with flat cavities. The cavities provide binding area of between the material surfaces with adsorbate.

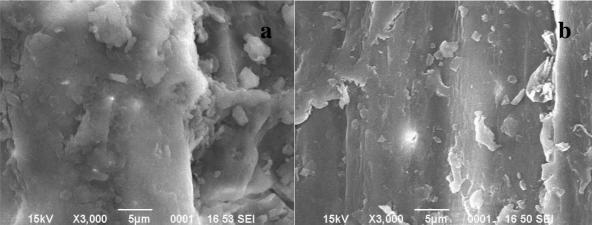


Fig. 1 SEM micrograph (3000X): (a) Pine needle (b) Lantana shoots

SEM-EDX analysis

The chemical compositions of adsorbents are determined by SEM-EDX. Fig. 2 shows the elemental percentage composition of N, C, S and H in the PN and LS adsorbents. The nitrogen content was recorded in PN (0.879%) and LS (0.834%). H content was found to be 8.848% in PN and 7.515% in LS. The carbon content was found high in PN (48.16%) adsorbent than LS (44.32) adsorbent. The CHNS/O concentrations are much higher in PN adsorbent as compared to LS adsorbent.

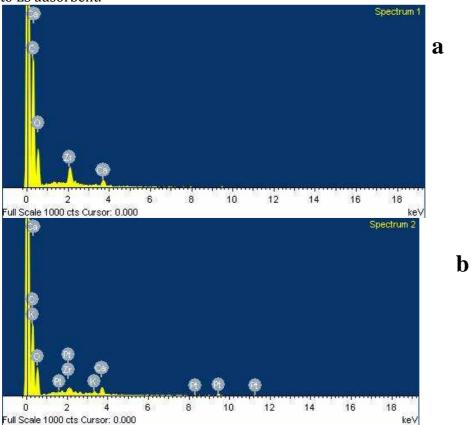


Fig. 2 SEM EDX micrograph: (a) Pine needle (b) Lantana shoots

FTIR analysis

Fig. 3 shows IR spectra for PN adsorbent, the peaks at around 3140 to 2947 cm⁻¹ corresponding to the stretching vibration of CH. The peak at 2710 cm⁻¹ was typical of OH, while, the peak at 2586 cm⁻¹ was indicative of S-H stretching vibrations. Bands around 2353 and 2326 cm⁻¹ correspond to the C≡C. The band at 1308 and 1261 cm⁻¹ were associated with C-O. Peaks at 1220 cm⁻¹ was indicating the C-OH stretching vibration. Some weak bands were also observed in PN for a series band in the range of 1161 to 951 cm⁻¹, which indicates CH stretching.

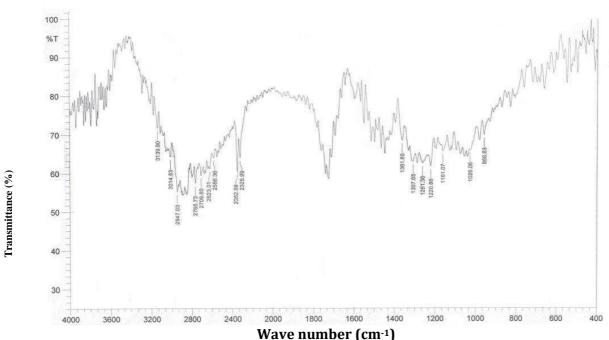


Fig. 3: FTIR spectra of PN adsorbent

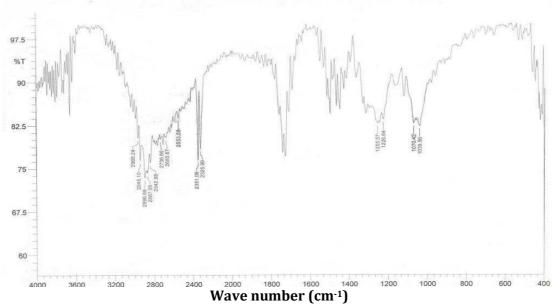


Fig. 4: FTIR spectra of LS adsorbent

Fig. 4 shows IR spectra for LS adsorbent, the medium peak ranged from 2968 to 2737 cm⁻¹ was assigned to the CH bonds. The absorption bands ranged from 2681 to 2553 cm⁻¹ were due to contribution from SH stretching. The bands observed at 2351 indicated $C \equiv N$ stretching, while, 2325 cm⁻¹ was assigned to $C \equiv C$ bonds. The medium bands located at 1256 cm⁻¹ was

Transmittance (%)

corresponded with C-O stretching. The band located at 1226 cm⁻¹ was assigned to the stretching vibration of C-N group. The band occurred at 1070 cm⁻¹, which corresponds to the C-O-C groups. The appearance of bands at 1039 cm⁻¹ can be attributed to the C-H groups. The FTIR spectra indicate the presence of oxygen containing surface functional groups such as alkyl groups, alkynes groups, thiol and ether groups in the selected adsorbents. The presence of various functional groups on the carbon surface contributes to preferential uptake for different molecule species by carbon [9].

CONCLUSION

The low cost adsorbents were prepared from pine needles (PN) and lantana shoot (LS) plant based materials. The prepared adsorbents were characterized by determining different parameter such as pH, moisture content, ash content, mesh size, bulk density, elemental analysis, SEM-EDX and FTIR spectroscopy. Pine needle (PN) and lantana shoot (LS) are potential precursor for low cost adsorbents due to having high carbon content, low moisture and ash content. The irregular cavities are present on the surface of adsorbent, which indicate the feasibility of binding sites. The materials also possess properties like high carbon content, availability of nitrogen and hydrogen content. The chemical properties were also indicated the functional groups present on the surface of adsorbent by FTIR analysis. It was found that functional groups such as CH (alkanes), OH (carboxylic acids), $C \equiv C$ (alkynes), S-H (thiols) and C-O-C (ether) groups are present. The plant based material were used in present investigation are available free, abundant form and locally available. The preparations of these adsorbents are cheap and economically suitable for removal of toxic pollutant from water and wastewater.

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