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PREPARATION, CHARACTERIZATION USING *Vitex negundo* PLANT MATERIALS ASSORBENTS

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ABSTRACT

The powdered activated carbon was prepared from *Vitex negundo* stem agricultural waste, carbonization was done at 400°Cand the chemical activation was carried out using various dehydrating agent such as H_2SO_4, H_3PO_4, $ZnCl_2$, and KOH and activation process was carried out at 600^0C for five hours and allowed cool at room temperature for four hours. The materials were grained and sieved with four different sizes 53µ, 106µ, 300µ and 500µ.The aforesaid particle size 53µ was taken to further studies such as Physico-chemical parameters. The characteristic parameters were then compared with commercial activated carbon value. The instrumentation analysis such as Fourier transform infrared (FTIR), scanning electron microscope (SEM) were performed to determine the surface morphology of prepared adsorbent.

Keywords: Activated carbon, chemical activation, characterization, FTIR, SEM, BET

INTRODUCTION

The preparation of adsorbents from agricultural waste has potential economic and environmental impacts; it converts unwanted, low-value agricultural waste to useful, high-value adsorbents. Adsorbent are increasingly used in water to remove organic chemicals and metals present in environment **[1]**. Activated carbon from vegetable material was introduced industrially in the first part of the twentieth century and used in sugar refining **[2]**. In the US, activated carbon from black ash was found very effective in decolorizing liquids **[3]**. Agricultural by-products and waste materials used for the production of activated carbons include olive stones **[4]**. Sawdust **[5]**. Coconut shells husks **[6]**, Glycosmis mauritiana **[7]**, palm seed coat **[8]**, rice husks **[9]**. This study explores new activated carbon from natural plant materials through various processes. The general process to prepare activated carbon is based on carbonizing and activating the carbonaceous precursor material. Activation may be activated physically: a two stage process involving carbonization of the precursor material followed by controlled gasification (steam flow, temperature, heating rate etc) of the char or chemically, in which the precursor material is impregnated with a chemical agent and heated to temperature of 450-700^{0}C **[10]**. In chemical activation several type of chemicals are used as the activating agent including $ZnCl_2$, H_3PO_4 and KOH having different properties. Hayashi and coworkers **[11]** found that the greatest specific surface area of activated carbons was obtained at the carbonization temperature of 600°C with $ZnCl_2$ and H_3PO_4 activation. The industrials effluent treated by waste water treatment plants contain large amounts of organic matter and pollutants including metal such as Cu, Zn, Cd, and Pb etc., the uptake of metals in pollution control **[12]**.

Activated carbon is an efficient and versatile adsorbent for purification of water, air and may chemical and natural product **[13]**. To enhance the capacity of activated carbon to adsorb cations, many functional/surface modification **[14]** methods have been introduced. These include chemical or physical treatment **[15-17]**, surface modification **[18-23]** and porous activity **[24]**.

The aim of this paper is to produce activated carbon from *Vitex negundo*stem waste and characterize the carbon. The prepared carbon sample values are also compared with commercial activated carbon **[25]**.

2. MATERIALS AND METHODS

2.1. Chemicals

Sulphuric acid, Phosphoric acid, Zinc chloride, Potassium hydroxide and all reagents used in this study were procured from Merck, India. Double distilled water used for all the Physico-chemical characterization work.

2.2. Preparation of activated carbon

The *Vitex negundo* plant was collected from an agricultural waste in Ariyalur district, Eravangudi village, Tamil Nadu, India. The precursor washed with distilled water to remove impurities, cut into small pieces (2.5cm), dried in sunlight. Activation processes were carried out by physical and chemical activation.

I. In physical activation, the precursor sample was heated at high temperature at 600ºC for five hours and then cooled, grained, sieved and stored in air tight container, named as VNC.

II. Chemical activation is a two step process, in chemical activation the precursor samples were soaked in dehydrating chemicals such as H_2SO_4(50%), H_3PO_4(50%), $ZnCl_2$(50%)and KOH(50%) for 24 hours. So, that solution gets well adsorbed for a period of 24 hour at the end of 24 hours excess solution was decanted off and dried in hot air oven maintained at 110^0C for 4 hours. Then the chemically dipped precursor sample was placed in muffle furnace. The carbonization process was carried out at 400^0C and the carbonized samples were made to activate at 600^0C for 5 hours. All process was carried out in absence of air.

The activated carbon sample was washed with plenty of double distilled water to remove excess acids. During the washing process, 0.1M HCl or 0.1 M NaOH was used until the pH of the washing solution reached to 6-7 and dried at 110^0C. The dried material was grained and sieved with different sized meshes (0-53µ, 53-106µ, 106-300µ, and 300-500µ) and named as VNC-1(H_2SO_4), VNC-2(H_3PO_4), VNC-3($ZnCl_2$) and VNC-4(KOH). The different sized carbon sample was stored in air tight plastic container to further use.

2.3. Experimental techniques

The Physico-Chemical characterization was carried out for prepared carbon samples to identify the nature and properties for the prepared adsorbent. The samples were also subjected to FTIR to elaborate the functional groups present in the surface of the carbon samples. The various characteristic parameters such as moisture content, Bulk density, ash content, matter soluble in acid, matter soluble in water, loss on ignition, pH, iron content, iodine number and surface area

experimental procedure was followed by Hassler procedure from this characteristics work[26].We can conclude the nature of an adsorbent and results was listed in **Table 1**.

2.4. RESULT AND DISCUSSION

Bulk density of carbons obtained from all the materials show that carbon VNC-1 (0.41g/ml) and VNC-3(0.50g/ml). Higher bulk density due to its high fiber content, VNC-2(0.36g/ml) and VNC-4(0.36g/ml) has the lower bulk density and compared to a VNC (0.39g/ml) carbon has the lower bulk density and commercial activated carbon (CAC) has the higher bulk density. The moisture content is found to be VNC-1 is the 0.51% and is comparatively very less to other carbon (VNC, VNC-2, VNC-3, VNC-4 and CAC) and hence the experimental carbon is found to have more surface area for adsorption. Ash contents for all carbon were found to be very low thereby increasing the fixed carbon content for the carbon obtained from prepared samples. The characterization studies on iodine number VNC-1 possess high iodine number of 360 and it is compared with commercial activated carbon (CAC) has the lower iodine number of 200.36 indicated in **(Table 1)**. Iron content is almost low for all the carbons. This level of iron content will not affect the effluent water without the problem of iron leaching into treated water. The surface area was determined by BET method using nitrogen as the adsorbent at liquid nitrogen temperature. The BET surface area valueVNC-1 (H_2SO_4) is the 319 shown in **Figure 1**. Compared with commercial activated carbon 296 (CAC) has the high surface area value is theVNC-1.

This has been further confirmed by the surface morphology studies using SEM shown in **Figure 6** is the VNC1 (H_2SO_4) chemical activation from which it is evident thatVNC1 (H_2SO_4) has a significantly porous structure. However, it is seen from **Figure 7** that has nearly disappeared the after adsorption of Pb(II). The FT-IR spectrum shown in **Figure 2 to 6** reveals that the activated carbon contains a number of functional groups such as,-COOH, O-H,C-H, C-O, C=O,N-H, amides, monosubstituted aromatic structure *etc.* From the characteristics parameter it proves that the carbon prepared by H_2SO_4 (VNC-1) process found high in a vast margin when compared to other carbons because of high charring power of H_2SO_4, thus the H_2SO_4 activated carbon process showed better result.

Table 1: Comparison between the prepared activated carbon and commercial activated carbon

Parameters	Units	VNC	VNC-1	VNC-2	VNC-3	VNC-4	CAC
Ash content	%	2.79	5.32	11.0	9.96	29.80	17.1
Bulk density	g/ml	0.39	0.41	0.36	0.50	0.36	0.66
Iron content	%	0.6	-	-	0.16	-	-
Iodine number		279	360	359	343	291	200.36
Loss on ignition	%	89.42	91.61	92.39	89.29	93.96	97.09
Matter soluble in acid	%	0.27	0.89	2.89	3.09	4.03	4.58
Matter soluble in water	%	2.09	0.49	0.99	0.52	2.20	1.55
Moisture content	%	1.40	0.51	0.47	0.74	1.38	16.67
pH		5.7	4.2	1.9	7.0	12.2	7.0

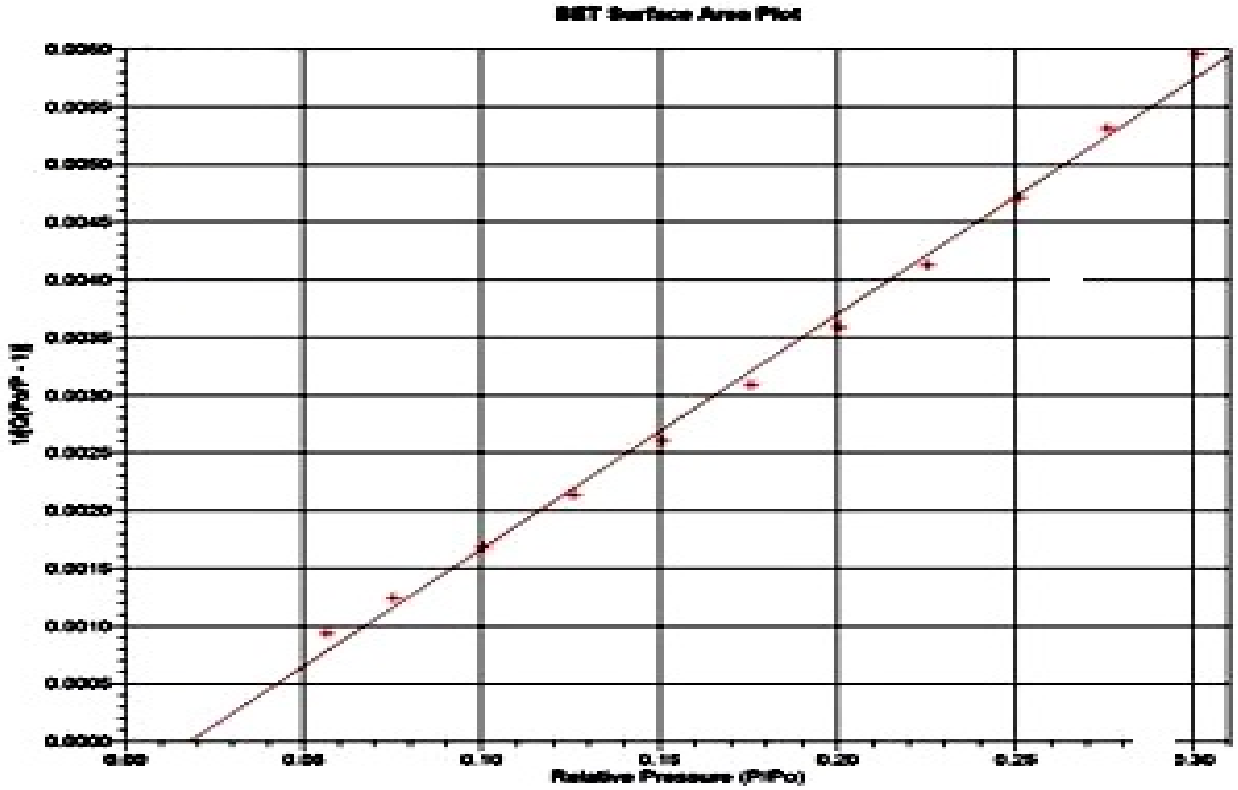

Figure 1: BET Surface area for VNC-1

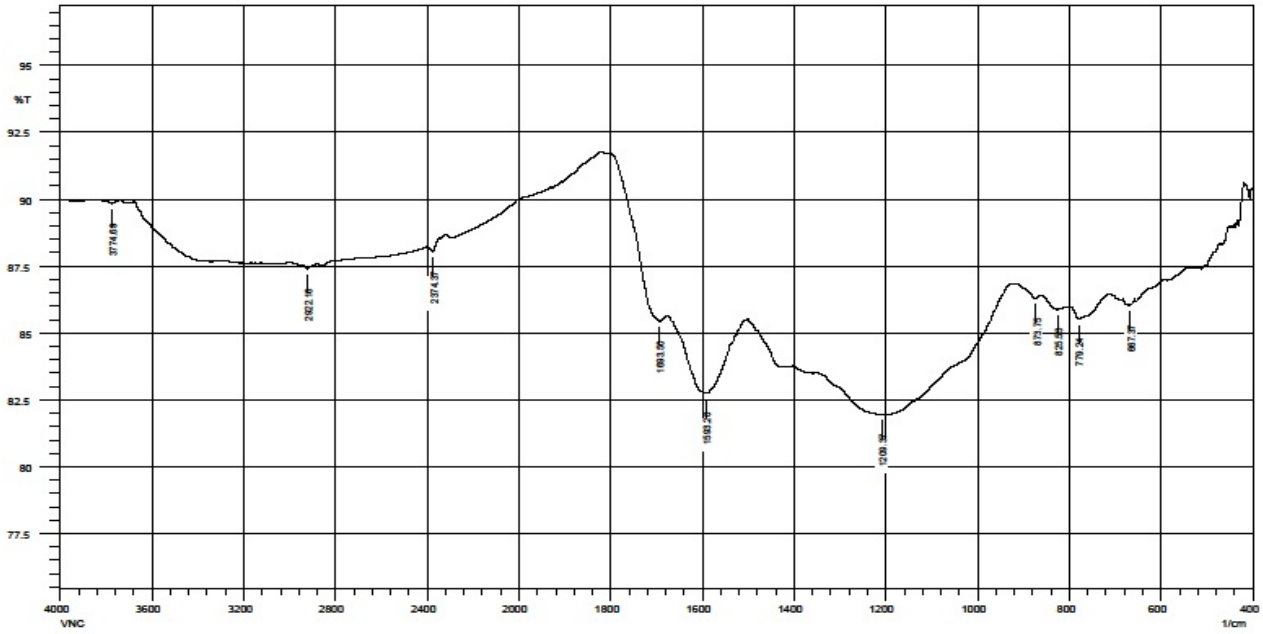

Figure 2: FTIR spectum of VNC Carbon

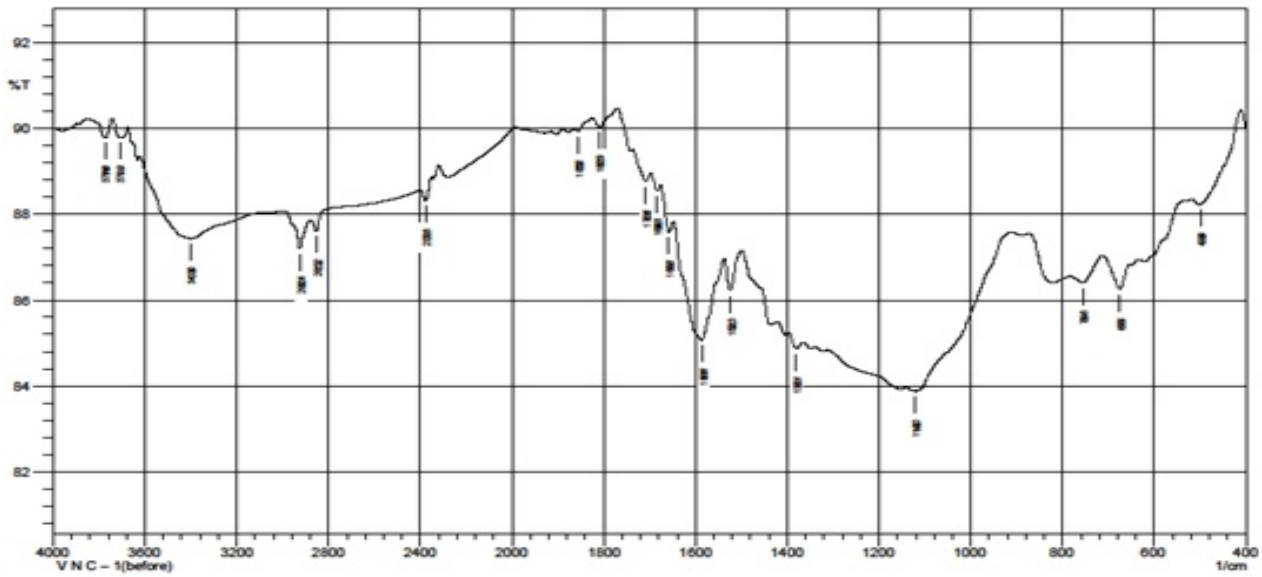

Figure 3: FTIR Spectrum of VNC-1 Carbon

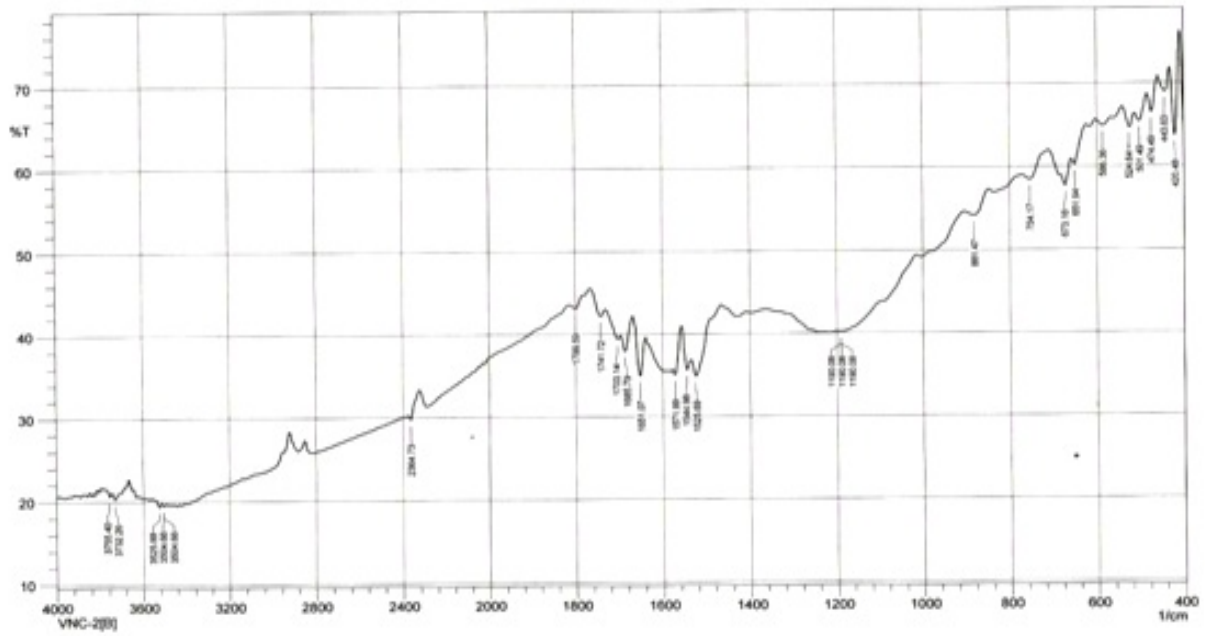

Figure 4: FTIR Spectrum of VNC-2 Carbon

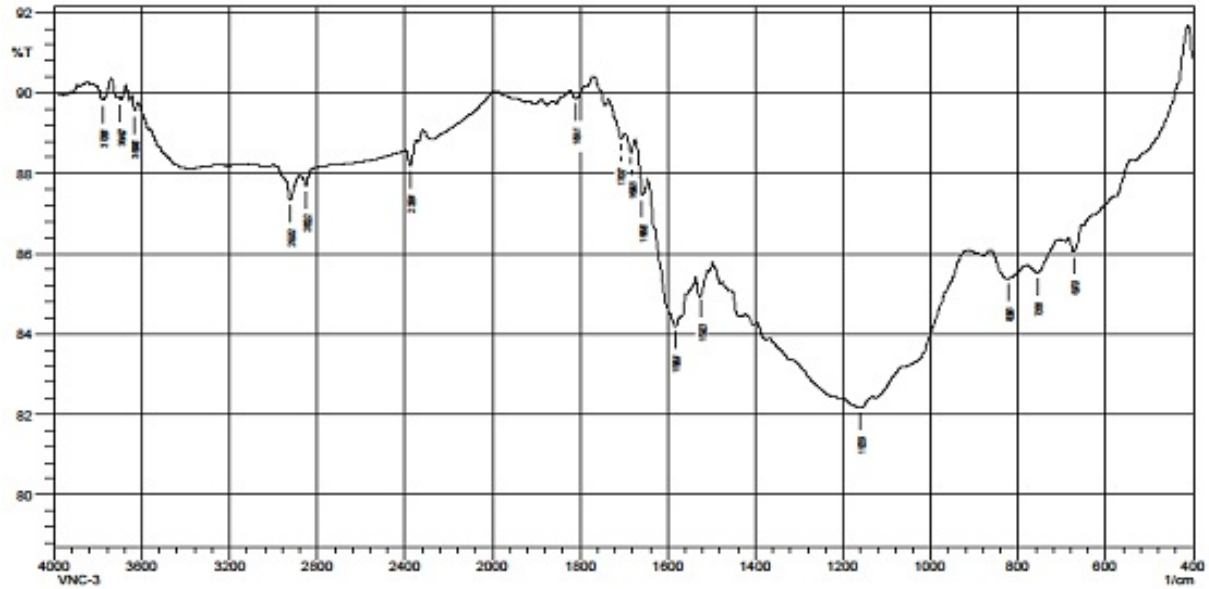

Figure 5: FTIR Spectrum of VNC-3 Carbon

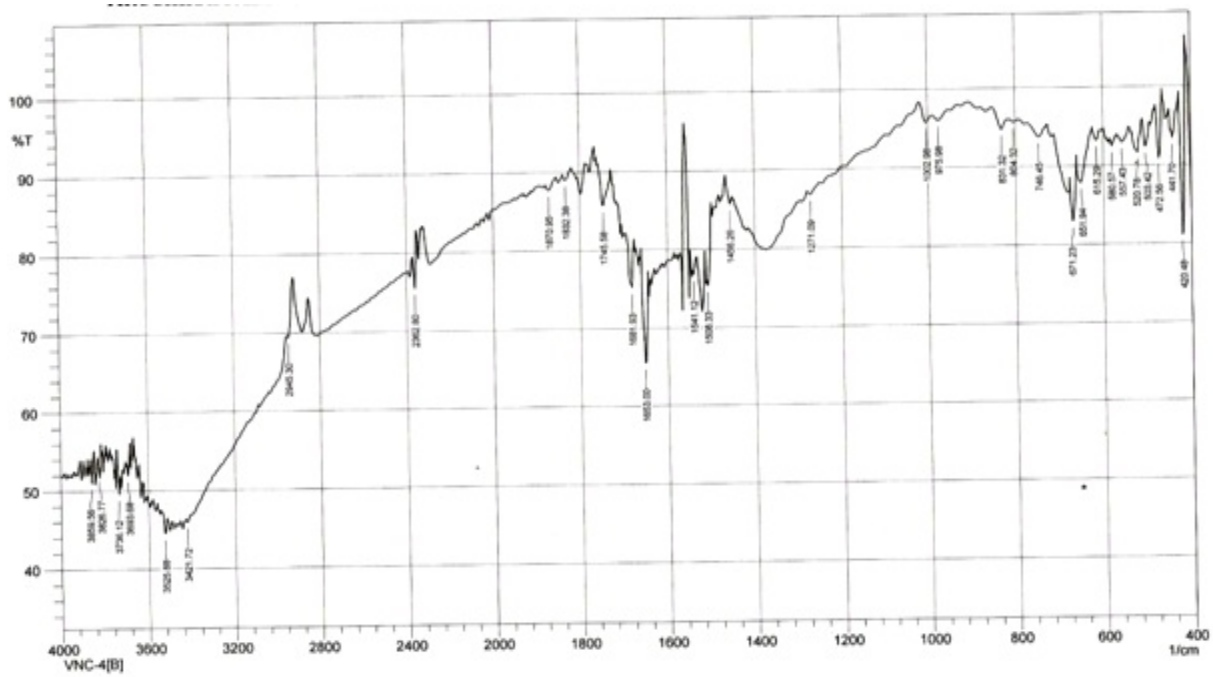

Figure 6: FTIR Spectrum of VNC-4 Carbon

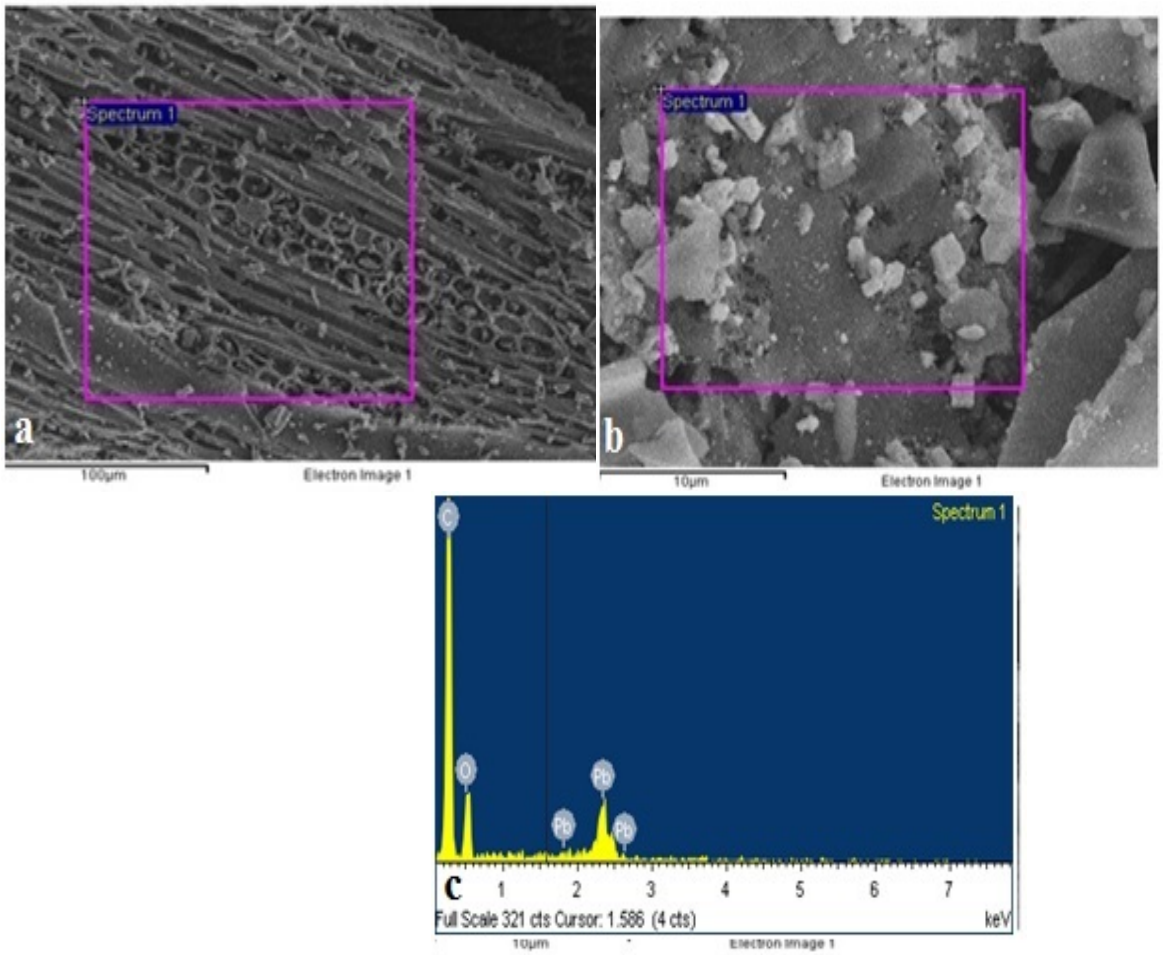

Figure 7: (a), (b) and (c) SEM micrograph of the VNC-1 before, after adsorption and EDAX of Pb(II)

CONCLUSION

The results obtained from the present study suggest the possible use of a widely available and environmental friendly material as an adsorbent for water treatment. *Vitex negundo*stem powdered chemically activated H_2SO_4 shows best for the removal of heavy metals.

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