PREPARATION, CHARACTERIZATION OF COCONUT SHELLS ACTIVATED CARBON USING $FeCl_3$ AS ACTIVATING AGENT AND REMOVAL OF TRANSITION METAL IONS

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A B S T R A C T

Waste materials pollute our environment or ecosystem and pose the problem of their disposal. Activated carbon can be produced through chemical activation process by using activating agent ferric chloride (FeCl3) in 20%, 40%, 60% concentration added in coconut shells (CS). The physico-chemical characteristics such as apparent density, Acid insoluble matter, water soluble matter, pH, conductivity, Iodine test studied. Adsorption process is a powerful technique that can be used for efficient removal of Ni2+ and Fe3+ transition metals. Similarly physical morphology was studied by analyzing Scanning Electron Microscopy, X-ray Diffraction and Fourier Transform, Infrared Spectroscopy.

Keywords: Activating agent, Transition metals ions, Adsorption, Coconut shells.

Introduction

Agriculture waste materials, which impact negatively on environment, have been commonly applied over other adsorbents due to low or zero cost materials. Also, these materials have excellent properties for removing pollutants like their high surface area, pore size distribution and functional groups on their surface. The carbonization process enriches the carbon content and introduces the porosity in the char while activation further develops the porosity and creates some ordering in the structure. Adsorption process has been used exclusively in water treatment and many studies have been carried out to find inexpensive and chemico-physically feasible adsorbent. [1,2] Governments have established environmental restrictions with regard to the quality of coloured effluents and have impossed upon dye industries to decolorize their effluents before discharging. For treatment of these coloured effluents, adsorption process is one of the effective techniques for removal of color. Commercially activated carbon is used as an adsorbing agent for removal of industrial colored effluents but is associated with greatest drawback of being expensive due to its high cost of manufacturing and regeneration [3,4].
There is batch experiment and column type laboratory experiment carried. This is due to properties of activated carbons which can provide high adsorption capacity and mechanical properties. A study of the effect of preparation conditions on the yield and quality of activated carbon (AC) produced from dates' stones was made using zinc chloride as an activator. Activated carbons are highly porous and adsorbent materials. They have wide applications in domestic, commercial and industries. The chemical activation technique has more advantages over the physical activation technique since the chemical reagents enhance the yield and increase the surface area of the resulted product.[5,6]. The activated carbons produced in powdered form as such in order to make them into tablet forms circular ring of a hydraulic press are used. The adsorbents are mixed with water (just enough to cause homogeneous mixing) and manually compacted into the ring[7].There are different technologies used for removal of heavy metals from waste water mainly precipitation, ion exchange, membrane processes, evaporation chemical oxidation or reduction, solvent extraction and biological materials[8]. Adsorption is a widely used as an effective physical method of separation in order to elimination lowering the concentration of wide range of dissolved pollutants (organics,

inorganic) in an effluent. In the recent years, it has been increasingly used for the prevention of environmental pollution and antipollution laws have increased the sales of activated carbon(AC) for control of air and water pollution. The heavy metals and minerals in wastewater is one of the most serious problems in India. Due to extensive anthrogenic activities and disposal of industrial waste materials. The concentration increases to dangerous levels in industrial effluent of nickel, chromium, lead, zinc, arsenic, cadmium, selenium, and uranium[9,10].

Objectives of Present Work

From literature survey we came to know that adsorption experiments are carried out to investigate the removal of heavy metal ions like Nickel(II),Lead(III) and Chromium(II) from coconut shells by using Phosphoric acid and Zinc chloride. In this research work we are going to perform adsorption experiment to investigate the removal of heavy metal but by using $FeCl_3$ which is yet not used for this purpose. The significant feature of activated carbon that makes it a unique and particularly economical adsorbent is that it can be produced from waste materials for environmental and ecological reasons. The innocuous disposal of these wastes has become immensely important. The main objective of the proposed work is the removal of heavy metal ions and elemental analysis.Similarly the scanning electron microscopy , Fourier transform infrared spectroscopy and X-ray diffraction analysis were carried out for the characterization of the prepared activated carbon was done by using various analyses such as apparent density, acid insoluble matter, water soluble matter, pH value ,conductivity, iodine test etc.. The prepared activated carbon compared with commercially available activated carbon for removal of transiton metals.

Experimental

Materials:

Coconut shells selected from the local farms, market. Activated carbon is prepared by taking 20%, 40%and 60% concentration of $FeCl_3$

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fig.1- Coconut Shells

Preparation of the activated carbon

The precursor used for the preparation of the activated carbon was the Coconut Shells. These are purchased from local market and sample were separated. The separated sample were then washed with distilled water to remove any dirt or impurity present and overnight. The selected batch of seeds was then dried to remove the water in an oven at 110 ^{0}C for 1 hour. The selected sample was put on a petri dish and inserted into a muffle furnance 300 ^{0}C for 1 hour.The carbonised samples are crushed in mortar and pestle to produce the uniform sized crushed particles . The crushed sample was then washed several times to remove other impurity, The washed samples particle is then dried overnight. On the other day the particles were further dried at 110 ^{0}C for 1 hour. The carbon produced is then put in the desiccators to cool them to the room temperature. The cooled particles are then stored in the air tight containers.The another batch then impregnated with the (w/w) of ferric chloride in 1:4 ratio by weight and mix well. The mixtured formed was then left for 24 hour. The mixture was then put in an oven at 160^{0}C for hour. The activated material was kept desicater and cooled to room temperature. The mixture was then washed several times with deionised water until the pH of the washing equilibrates to 6.5-7. The carbon was soaked in 1% sodium carbonate solution for 24 hour. Then it was washed with distilled water to remove excess sodium carbonate and dried at 110 ^{0}C for 1 hour. The dried carbon is then cooled in desicater and then stored in air tight container.

Characterization

1. Apparent density:

A specific gravity bottle of 25 ml capacity was filled with the adsorbent and packed well by tapping with a rubber stopper. The weight of the adsorbent was determined. The weight (g) divided by the volume (ml) gives the apparent density (g/ml) of the adsorbent.

2.Acid insoluble matter:

The adsorbent (0.5 g) is placed in an evaporating dish, mixed with distilled water to a thin slurry 5 - 10 ml of concentrated HClis added and digested by warming until sample is nearly dry. The digestion is repeated three times with 5 ml of the acid. Then it is diluted with 100 ml water, filtered using a previously weighed sintered crucible and the weight of the insoluble matter is calculated after drying for a constant weight at 103 °C.

3. Water-soluble matter:

0.5 gm of each adsorbent was added to 50 ml of distilled water and is shaken thoroughly for about 30 minutes and filtered. The residue i.e. adsorbent is dried, cooled and weighed.

4.pH :

Apparatus and material:

1. pH meter : ELICO LI120 is used for measurement of pH,where temperature is set on 20^{0}C .glass electrode and reference electrode Cl 51 B use.
2. Distilled water
3. Buffer solution at pH 9.00
4.Buffer solution at pH 4.00

Procedure:

0.5g of sample was weighed in 250ml beaker, add 50 ml of distilled,CO_2 free(boiled out)water, cover with watch glass and boil on the hot plate for 5minute.Insert the thermometer and set aside for a few movement to allow the bulk of the activated carbon particle to settle. Pour off supernanent as soon as possible and before its cool to 60 ^{0}C.Cool the decanted portion to the room temperature and measure the pH to one decimal place

5.Conductivity:

0.5g of the carbon was weighed and transferred into a 250 ml beaker and 50 ml distilled water was added and stirred for 1hour. Samples were allowed to stabilize. This solution is used to measure the electrical conductivity (EC)measurements of the ACs and results read .

6.Iodine Test.

Iodine Number is defined a the number of milligrams of iodine absorbed by one gram of activated carbon powder. Iodine Number is a measure of micro-pore content of activated carbon. A higher iodine number signifies higher micro-porosity.

1.1 Tables

2 charactrization of unactivated carbon of coconut shell

Unactivated carbon	Apparent density g/ml	Acid insoluble matter (gm)	Water insoluble matter (gm)	pH	Conductivity	IodineTest mg/g
Coconut Shell	8.63	0.10	0.40	2.93	0.37×10^3mhos	916.56

1.2Table

Charactrization of activated carbon of Coconut Shell

Activated carbon	Apparent density g/ml	Acid insoluble matter (gm)	Water insoluble matter (gm)	pH	Conductivity	IodineTest mg/g
20%	7.54	0.14	0.30	2.90	1.02×10^3mhos	892.76
40%	9.68	0.16	0.38	2.60	2.15×10^3mhos	847.82

60%	10.55	0.11	0.39	2.52	1.65×10^3mhos	824.02

batch experiments was carried out using coconut shells carbon activated by activating agent $FeCl_3$ following results which give an idea for percentage removal of Fe^{3+} and Ni^{2+}.

1.3 Table

2 **% Removal of Fe^{3+}**

ppm	20%	40%	60%	Unactivated
1000	13.44	12.16	13.76	10.56
800	12.88	11.84	13.44	10.45
600	12.64	10.56	10.32	9.11
400	7.52	8.00	8.8	7.9
200	7.36	6.72	8.16	7.10

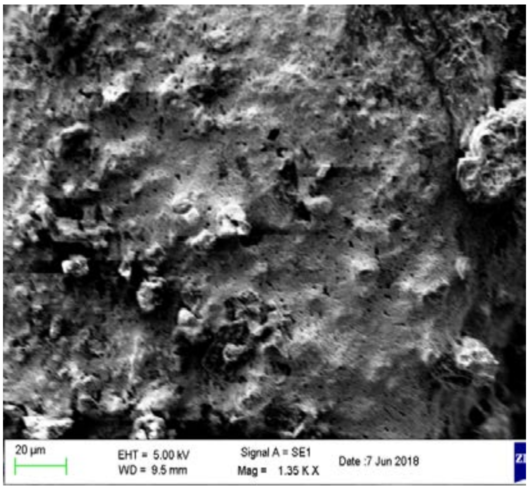
1.4 Table

% Removal of Ni^{2+}

ppm	20%	40%	60%	Unactivated
1000	14.88	15.28	13.12	10.59
800	8.48	15.12	14.48	8.40
600	8.16	14.00	13.76	8.12
400	7.84	9.44	12.64	7.00
200	6.48	8.72	12.08	6.29

Results

SEM analysis-The morphology of activated carbon analysed by using ZEISS machine.The activated carbon is known as a good adsorbent because of its high degree of porosity and an extensive surface area.



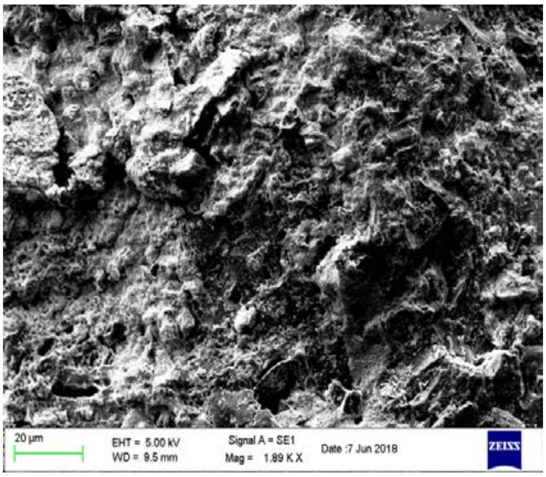


Fig. 1 - (a) SEM activated 60% coconut Shells (b) SEM ofunactivated coconut Shells

1.2 FT-IR analysis- The FT-IR spectra of chemical activated and unactivated carbon were given in fig.5-6. The absorption bands characteristic of c=c are observed at 1608,1693 cm^{-1}.At 451,756,414 cm-1 indicates existence of monosubstituted aromatic ring in both samples activated 60% and unactivated

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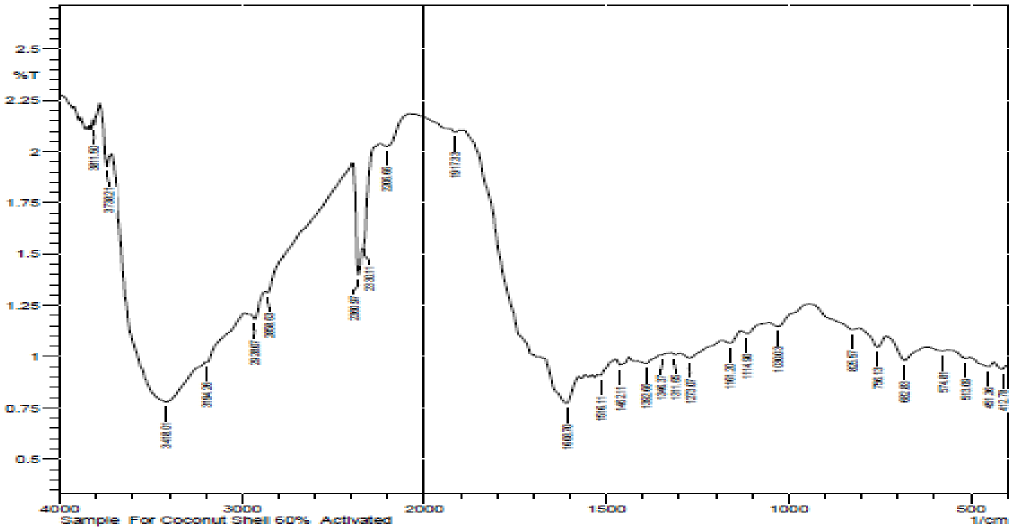
a)



b)

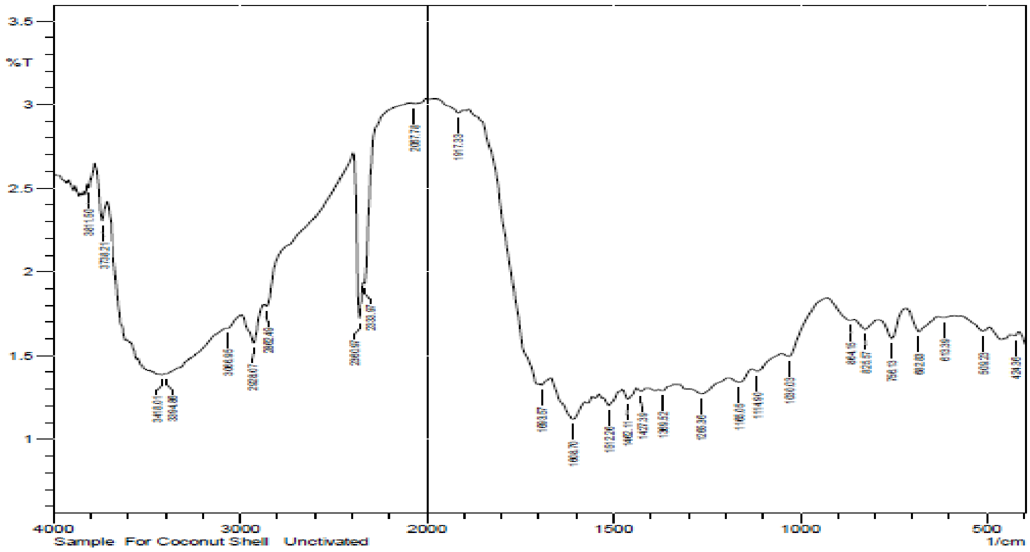
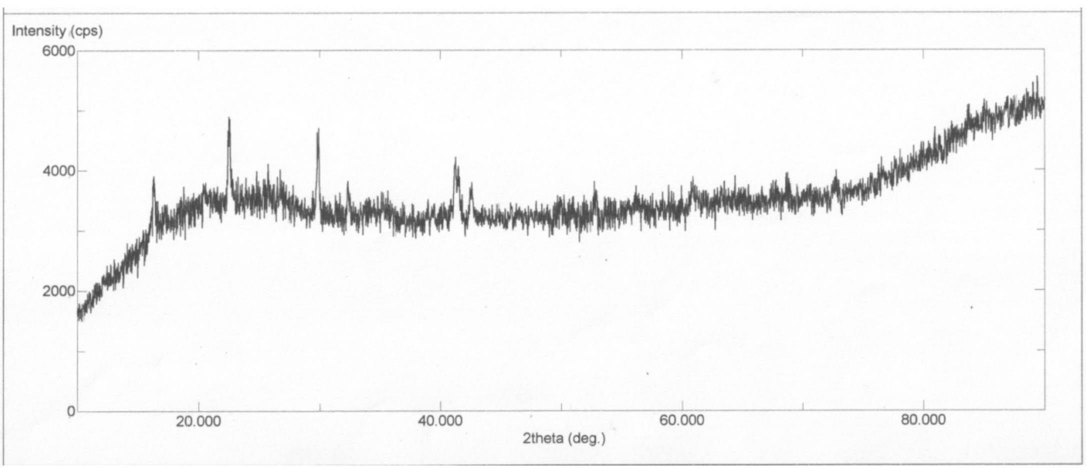


Fig. 1 - (a) FT-IR activated 60% coconut Shells (b) FT-IR of unactivated coconut Shells

1.3 XRD analysis-

Following figures 7-8: Illistrates the XRD pattern of activated carbon prepared from coconut shell by activating agens $FeCl_3$ The activated carbon of coconut Shells 60% exhibited peaks around 2 theta = 25^0,30^0

a)



b)

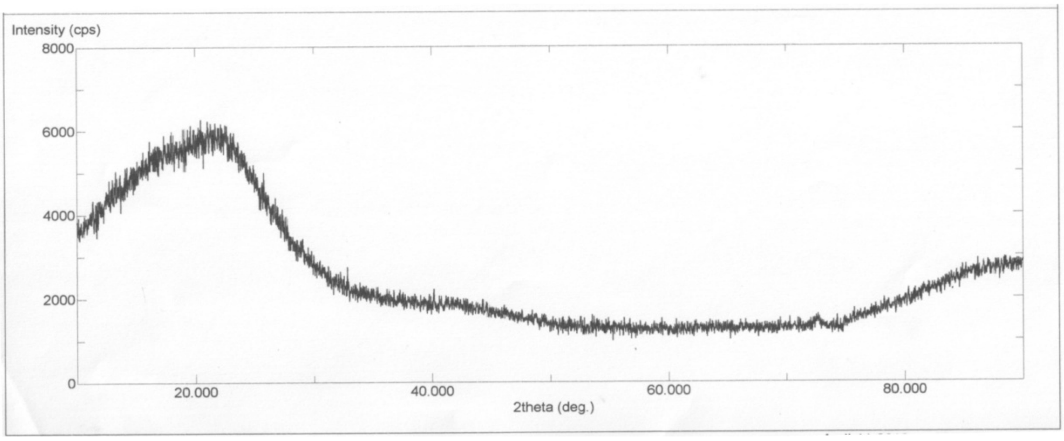


Fig7. (a) XRD of coconut Shell activated 60% (b) XRD of Unctivated coconut shell

Conclusion-

The experimental results show much lesser values of bulk density, nutrients and heavy metals than those reported in the literature.The coconut shell(CS) samples can be used in removing these toxic metal ions . The present study reveal that the variation of physical and chemical properties was found to be depending on the sample collection site.Based on the results, it is clear that the Iron and Nikel-removal values achieved activated carbon were higher than those unactivated . The main advantages of this removal procedure include (i) simplicity, (ii) cost effectiveness, (iii) rapidity, and (iv) a higher removal efficiency of toxic iron and nickel ions. In this study, activated carbon was successfully synthesized from a coconut shells, with the Ferric chloride activation process. This activated carbon was characterized by XRD, and FTIR. A comprehensive study was performed on its adsorption efficiency for the removal of transition element. XRD analysis proved that the sample had a perfect crystallite structure.

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