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Characterization and activation of coconut shell activated carbon Research Paper

Pratibha R.Gawande[1], Dr.Jayant Kaware [2]
[1](*Chemical Engg.Depatment Datta Meghe College of Engineering Airoli, Navi Mumbai, India)*
[2](*Sant Gadgebaba Amravati university,Amravati,Amravati,India)*

Abstract: *In present research paper coconut shell carbon was activated by coconut shell carbon was converted into activated carbon by chemical activation using different activating agents like $Cacl_2$, H_2So_4, H_3Po_4, KoH, and $Zncl_2$ and thermally activated. Batch adsorption desulphurization operation was carried out at room temperature for adsorption for selection of final activation agent for continuous process.Characteristics of coconut shell activated carbon was studied such as P.H, Moisture Content, Ash content ,Volatile matter content, Ash content , Volatile matter content, Fixed carbon, Iodine Number , BET surfacearea, Scanning Electronic Microscope (SEM).*

Keywords *- activated carbon, chemical activation, activating agents.*

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I. Introduction

Activated carbons are mostly used in adsorbent process as it is inexpensive, easy to prepare in addition to the possibility of tailoring their structure, physical and chemical properties[Hisham S]. Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces. These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tunable surface-containing functional groups. Generally, the activation of a carbon can be performed through physical or chemical activation or a combination of both. The chemical activation is normally preferable over physical activation since it is a faster process with a lower activation temperature [pratibha Gawande] Activated carbons primary source is from organic material with high carbon content (coal, wood, peat, coconut shells, etc). Granular activated carbons produced by grinding, adding a suitable binder to give it the hardness, re-compacting and crushing to the correct size[Rhoda].Different chemicals are used like Zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), Sulphuric acid (H_2SO_4), potassium hydroxide (KOH),sodium hydroxide (NaOH), for activation of activated carbon. The adsorbents were characterized by using Scanning electronic microscope (SEM), X-Ray diffraction (XRD), Accelerated surface area.

II. Literature Survey

Preparation and Characterization of Activated Carbon derived from Fluted Pumpkin Stem Waste was studied by Ekpete and Horsfall. Investigators were used fluted pumpkin stem waste for the preparation of activated carbon. Characterization of pH, bulk density, pH, porosity and iodine number was conducted and compared to a commercial activated carbon. Authors were found that there is significant difference in the properties of moisture, pH, porosity, ash content, iodine number, carboxylic acid content, lactones, pH and basic sites content of activated carbons [1]. Characterization of activated carbon prepared by phosphoric acid activation of olive stones was studied by S.M.Yakout and G.Sharat. Authors were studied the effect of activating agent concentration on the pore structure and surface chemistry of activated carbon derived from olive stone with chemical activation method using phosphoric acid[2].

Kermit Wilson et.al.were studied select metal adsorption by activated carbon made from peanut shells. Investigators were carried out steam activation, followed by air oxidation of peanut shells for production of activated carbon.and they were compared to metal ion binding by three reference carbons, steam-activated, air-oxidized peanut shell carbons showed adsorption properties similar to the best commercial, Coal-based carbons[3].Pratibha R. Gawande and Dr. Jayant P. Kaware carried out review on Preparation and activation of activated carbon from waste materials. Authors were studied different value added waste for preparation of activated carbon is used as adsorbent and chemical activation using different activating agents like $Cacl_2$, H_2So_4, H_3Po_4, KoH, and $Zncl_2$[4]. Adsorption of dibenzothiophene on activated carbon from dates stones using

phosphoric acid was investigated by Hisham S.Bamufleh.Authors were prepared Granular Activated Carbon from dates' stones by chemical activation using phosphoric acid (H_3PO_4) as an activator [5].

Rhoda Habor et.al were studied Production of Activated Carbon and Characterization from Snail Shell Waste (Helix pomatia).Investigators were used Snail shell waste material for the preparation of activated carbon using $ZnCl_2$ and $CaCl_2$ with the temperature ranging from 500˚C to 800˚C. The activated carbon prepared was characterized, showing effect of temperature on ash content, pore volume and porosity [6]. Adsorbents from karanja seed oil cake and applications was studied by Ashish saksule and Pallavi kude.Karanja Seed Oil Cake is by-product after oil extraction, which otherwise goes waste or as fertilizers, is used as Precursor for Activated Carbon Preparations Investigators were used Karanja Seed Oil Cake which is for preparation of activated carbon which is by product after oil extraction. They were prepared Adsorbent from Karanja seed oil cake in laboratory by various Chemical and Physical Activation Processes [7].Dipa Das et.al.preparation of activated carbon from green coconut shell and its characterization. Authors were prepared activated carbon from green coconut shells by chemical activation method. Authors were studied different properties like pore size, surface area micro pore volume and thermal stability [8].

Roozbeh Hosein et.al. Preparation and characterization of activated carbon from apple waste by microwave assisted phosphoric acid activation. Authors were prepared activated carbon from apple pulp and apple peel by using phosphoric acid as an activating agent[9].Characterization of activated carbon prepared by phosphoric acid activation of olive stones was studied by S.M.Yakout and G.Sharat. Authors were studied the effect of activating agent concentration on the pore structure and surface chemistry of activated carbon derived from olive stone with chemical activation method using phosphoric acid[10].Mehdi Jahangiri et.al.preparation of activated carbon from walnut shell They were used chemical activation, using KOH to obtain high efficient adsorptive properties[11].Hassan M. et.al synthesis and characterization of activated carbon from saudi arabian datestree's fronds wastes .Investigators were used date's fronds waste as a raw material for producing activated carbon. Investigators were used phosphoric acid for activation and they were used various concentration of H_3PO_4 [12].Adegboyega Surajudeen Olawale et.al Preparation of phosphoric acid activated carbons from Canarium Schweinfurthii Nutshell. Activated carbons were prepared by phosphoric acid activation of Canarium Schweinfurthii spent nutshell[13].

Arenst Andreas Arie, Vincent and Aditya Putranto were studied Activated carbons from KOH activation of salacca peels as low cost potential adsorbents for dye removal. Salacca peel was used to prepare activated carbon (AC) by chemical activation with potassium hydroxide[14].Preparation and Characterization of Activated Carbon from Reedy Grass Leaves in a Two Step Activation Procedure was studied by Xu Jianzhong and Chen Lingzhi, FengXiaojie .Preparation of activated carbon from lignin obtained by straw pulping by KOH and K_2CO_3 Chemical Activation.Investigators were produced Activated carbons by chemical activation with potassium hydroxide [15].Preparaton of activated carbon from desiccated coconut residue by chemical activation with NaOH was studied by Mood Adib Yahya.Investigators were used agricultural waste for preparation of activated carbon.They were investigated the effect of temperature and impregnation ratio on the physicochemical properties of activated carbon prepared from desiccated coconut residue by chemical activation using sodium hydroxide[16].

Tang Shu Hui and Muhammad Abbas was investigated Potassium hydroxide activation of activated carbon. They were used Potassium hydroxide as an activating agent in activated carbon preparation. Authors were used activation temperature lower the boiling point of KOH 1327°C [17].Arunrat Cheenmatchaya and Sukjit Kungwankunakorn et.al preparation of activated carbon derived from rice husk by simple carbonization and chemical activation for using as gasoline adsorbent. Physical characterization of the activated carbon obtained was performed by scanning electron microscopy [18].Billy T H Guan et.al. Physical preparation of activated carbon from sugarcane bagasse and corn husk and its physical and chemical characteristics Sugarcane Bagasse and Corn Husk were used for preparation of activated carbon Authors were prepared activated carbon by physical and chemical activation method [19].

III. Material And Method

A.Material

Desulphurization of diesel fuel was carried out using coconut shell activated carbon. which were collected from local market. Coconut shell were collected and washed with fresh water and allowed to tray drying. Then dried coconut shell was burned at room temperature. Then grinded and sieved (particle size 6-52 mesh). The diesel fuel was used from local petrol station.

B.Activation of adsorbent

To increase the surface area of adsorbent for better adsorption, activation of the coconut shell activated carbon was conducted.

i)Activation by $CaCl_2$

100 grams of the coconut shell activated carbon was carbonized in 100 ml of 25% concentrated solution of $CaCl_2$ and covered with a lid for 24 hours. The soaked sample was transferred into a drain tray and washed repeatedly with distilled water to remove traces of chemical. The washed sample was transferred into an oven at 110˚C for 3 two hours, cooled and stored for use.

ii) Activation by $ZnCl_2$: The dried coconut shell was mixed with 10% boiling solution of $ZnCl_2$ for 24 hours. The excess solution was decanted off and air dried. Then washed with distilled water, dried in an oven at 110˚C for 2 hours. The sample was cooled to room temperature and stored in an air-tight container.

iii)Activation by KoH

The dried material was soaked in 10% KOH solution and kept for 24 hours. Then the dried material was carbonized at 400^{o}C for 30 min., and activated in muffle furnace for 10 min. Followed by activation, the carbon was washed with 4N HCl to remove the cations. Then washed with water to remove the acid, then sample was transferred into an oven at 110˚C for 3 two hours, cooled and stored for use.

iv) Activation by H_3PO_4

The dried sample was impregnated with 35% boiling solution of H_3PO_4 for 24 hours. The dried material was carbonized at 550oC for 1 ½ hrs in muffle furnace, powdered and activated at 800oC for 10 min. Followed by was washed with water to remove the acid, dried and powdered.

v) Activation by H_2SO_4

Activation of activated carbon was done by using activating agent H_2SO_4 For activated charcoal respective amount of coconut shell carbon was soaked in 5 N H_2SO_4 for 12-18 hours to become activated carbon. Then carbon was washed with distilled water and spread on tray at room temperature to be drained after draining dried in oven at temperature 110 ^{o}c for 3 hours. After cooling activated carbon was packed in airtight container.

vi) Activation by Thermally

Activation of activated carbon was done by thermally. Coconut shell carbon was thermally activated at 800^{o}C in muffle furnace for 3 hr.

C.Characterization of coconut shell Activated Carbon

i)Ph.

2.0 g of coconut shell activated carbon was weighed and transferred into conical flask with100 ml of distilled water and stirred for 1 hour. Samples were filtered using a filter paper.Then pH measured using an electronic pH/Conductivity meter. The same samples were further used for electrical conductivity of the coconut shell activated carbon.

ii) Moisture Content

2 gram of coconut shell activated carbon sample was measured and then taken in a silica crucible .It was then heated in an oven at a temperature of 110˚c for 1 hr. After heating silica crucible was removed and cooled in a desicator.After cooling the weight of dried sample was measured.

Moisture Content (M) =100(A-B)/ (A-C)

Where A =Weight of silica crucible + weight of activated charcoal sample (original) (g)

B= Weight of silica crucible + weight of activated charcoal sample (dried) (g)

C= Weight of silica crucible (g)

iii) Ash content

A silica crucible was preheated in an oven at 900 ^{o}C for 1 h. It was then allowed to cool in a dessicator and reweighed. The weight lost was recorded as the ash content of the AC sample.

Ash content (%) = (A-B) x100 (A-C)

Where A = Mass of silica crucible with sample (g)

B= Mass of silica crucible after weight loss (g)

C = Mass of silica crucible (g)

iv) Volatile matter content

A 2 gm sample was taken in closed silica crucible It was then heated to 900 oC for 10 min in a muffle furnace. Then the crucible was cooled in a desicator and weighted. The loss in weight gives the volatile matter.

Volatile matter on dry basis=(A-B) x100 (A-C)

Where A = Mass of silica crucible with sample (g)

B = Mass of silica crucible after weight loss (g)

C = Mass of silica crucible (g)

v) Iodine Number

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

number signifies higher micro-porosity of the sample.2 gm of dried activated carbon was mixed with10 ml of 5% by weight of hydrochloric acid in a conical flask.The conical flask was boiled for 30 sec not directly but by placing it on a hot plate. The contents of the flask were cooled to room temperature and then 100ml 0.1N iodine solution was added to it. The flask was shaken vigorously for30 sec.The contents were filtered through a filter paper. Initially 20-30 ml of the filtrate was discarded and the remaining filtrate was collected in a clean beaker.Then 50ml of this filtrate was titrated against 0.1N sodium thiosulphate solution until yellow color just disappeared. After that 1ml starch solution was added into it and titration was continued till blue color just disappeared. Concentration of the final solution was calculated.(ASTMD4607–94).

vi)BET surfacearea

Brunauer, Emmett and Teller-are the three men who proposed a theory to measure the surface area of porous powder type solid particles. The principle involved is the adsorption of gas molecules to the surface of the solid whose surface area is required. From the area of each molecule, the whole area of the solid can be calculated.

vii)

Density is nothing but weight per unit volume of material.First measure the mass of the measuring cylinder which was used for this experiment.Then the given sample of activated carbon was placed into this cylinder and reweighed it.After drying sample in oven at 100^0c for 1 hr weight of sample was measured.

$D_B=(m_2-m_1)/v$

M_1=Mass of measuring cylinder in grams

M_2=Mass of measuring cylinder and its contents

V=Volume of measuring cylinder in litre

vii)Scanning Electronic Microscope (SEM)

Scanning electron microscope is a type of microscope which is used for visualization of porous structure of a material. The activated carbon sample was analyzed in a SEM to visualize the porous structure.

viii) X ray powder diffraction (**X.R.D)**

X ray powder diffraction analysis of the adsorbent was carried out by Difractometer.

D.Batch adsorption of sulphur from diesel using activated carbon

50 ml of the diesel sample was taken in the Erlenmeyer flask and 2, 4, 6,8,10 gm of adsorbent having particle size (6 mesh) was added to it. And stirred with the help of a magnetic stirrer for about four hour at room temperature. After each time interval of 10 min the mixture was given a rest of 2 min and then filtered through Watt man filter paper. Repeated same procedure for different particle size. (12-52 mesh) for selection of particle size and adsorbent dose for further process.

E. Analysis of Sulphur

UV-visible 159 spectrophotometer was used for the finding out total sulphur concentrations in the standard samples and desulphurised diesel. The relevant equation for calculating sulphur content is shown as

Desulphurization Rate= {(SF – SP)/ SF} ×100

SF: Sulphur content in feed

SP: Sulphur content in the product

IV. Results And Discussion

I).Characterization of coconut shell Activated Carbon

Coconut shell activated carbon was prepared and characterized by above different process. Resulting properties are given in following table.

Sr No	Properties	Value
i	P.H	6.9
ii	%Moisture Content	0.5
iii	%Ash content	1.88
iv	Volatile matter content	18.86
v	Iodine number	942 mg/g
vi	BET surface area	435.1 m^2/gm
vii	Bulk Density	0.590 g/cc

Table 1: Characterization of activated carbon

viii) Scanning Electronic Microscope (SEM)

Scanning electron microscopy has been extensively used to study the surface morphology of activated carbons. The activated carbon sample was analyzed in a SEM to visualize the porous structure. The SEM images of the H_2SO_4 impregnated activated carbon are shown in following figure.

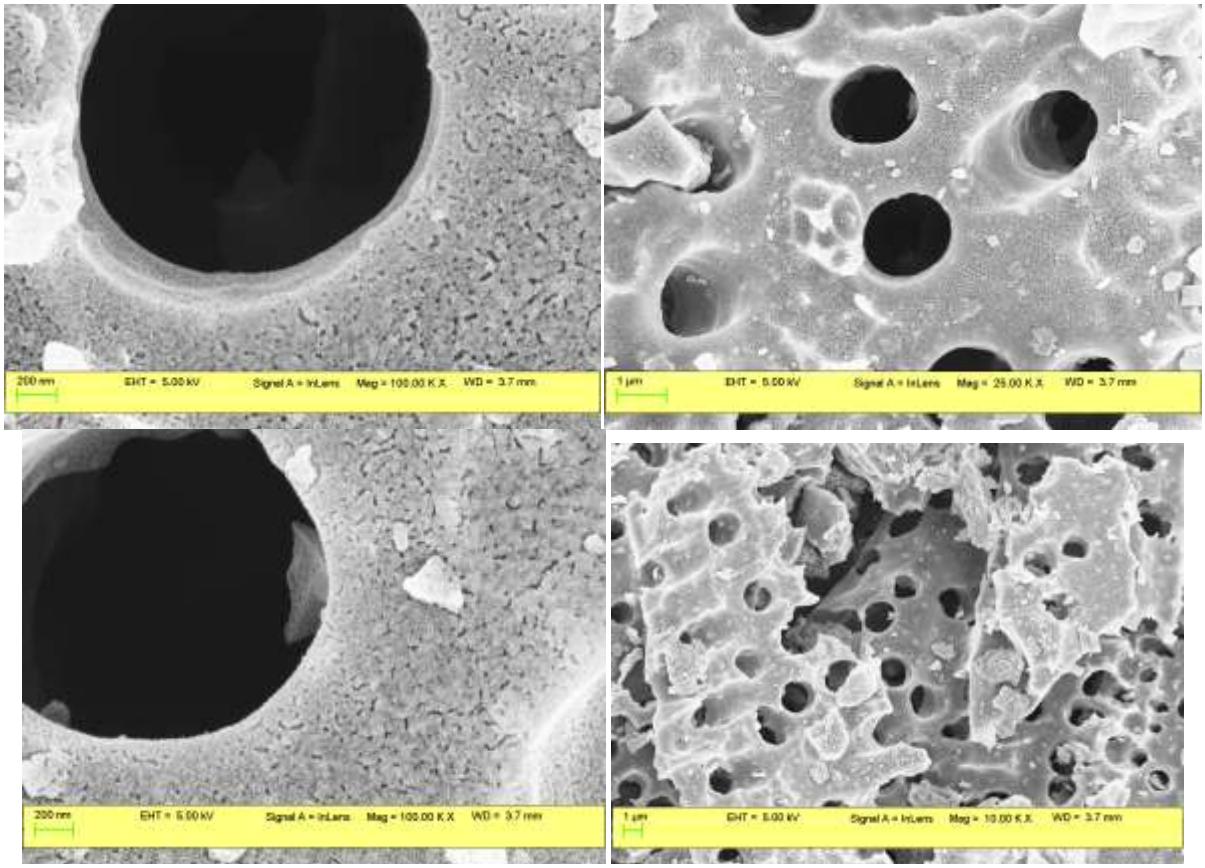

Figure1: SEM images of H_2SO_4 impregnated coconut shell activated carbon

i)X-Ray diffraction

Following figure no 2 illustrates the XRD pattern of the activated carbon prepared from coconut shell with an activated with H_2SO_4The activated carbon exhibited peaks at around $2\theta = 27^o$

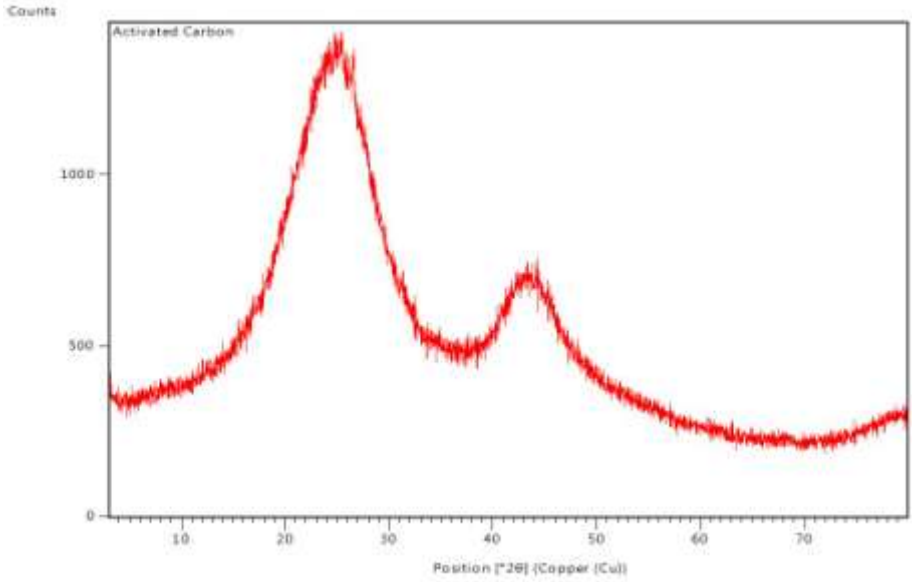

Figure 2: XRD for coconut shell activated carbon

II. Selection of activating agent

Batch experiments was carried out using coconut shell carbon activated by different activating agents like $Cacl_2$, $Zncl_2$, KoH, H_3PO_4, H_2SO_4, and thermally activated carbon .Following are the results which give an idea for selection of activating agent for further experiments.

Activating agent	concentration of adsorbent									
	2 gm		4gm		6gm		8gm		10gm	
	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal
$Cacl_2$	220	34.23	220	34.23	210	37.21	260	22.27	260	22.27
$Zncl_2$	268	19.88	260	22.27	250	25.26	250	25.26	210	37.21
koh	268	19.88	200	40.20	220	34.23	210	37.21	220	34.23
H_3PO_4	220	34.23	260	22.27	200	40.20	190	43.19	200	40.20
H_2SO_4	220	34.23	200	40.20	190	43.19	150	55.15	140	58.14
Thermally activated	268	19.88	268	22.27	260	22.27	250	25.26	230	31.24

Table 2 :Removal of Sulphur

As sulphur removal efficiency of activated carbon using activating agent H_2SO_4 was found to be maximum at adsorbent dose 10 gms.concentration of original sample was 334.5 ppm.

V. Conclusion

In the present work, coconut shells were used for production of activated carbons and chemical activation method using different activating agents. It was found that activated carbon which was activated by H_2SO_4 give better results. The yields of the activated carbons produced by chemical activation were found to be higher than untreated carbon. The prepared activated carbon was characterized by determining different parameters such as P.H, Moisture Content, Ash content ,Volatile matter content, Ash content , Volatile matter content, Fixed carbon, Iodine Number , BET surface area, Scanning Electronic Microscope (SEM) image.Activated carbon from coconut shell can be used for desulphurization of diesel .

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