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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM RICE HUSK

Hariprasad.p[1*] Rajeshwari sivaraj[2] Aniz cu[3]

[1*]*Research Scholar*
Environmental Engineering and Technology Laboratory,
Department of Environmental Sciences, Bharathiar University,
Coimbatore - 641 046, Tamilnadu, India.

[*2] *Assistant professor,Department of chemistry Government arts college*
udumalpet,,Tirupur district-642126,
**3 R&D Division Sud-Chemie IndiaPvtLtd*
Binanipuram P,O cochin-683502. Kerala

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Abstract

Activated carbon was, prepared from rice husk and its characteristics were investigated. The materials selected were initially physically activated at temperature ranging from 300 ^0C to 700^0C in muffle furnace with a holding time of 1Hrs and the carbonized material thus obtained was soaked in 1N KOH, in 1:1 ratio for overnight and was followed physical activation at 300^0C for 2hrs in muffle furnace. The activated carbon thus obtained is used for characteristic studies. The BET surface area of the carbon thus obtained were found to be ranging from 11.9985 m^2 /g to 279.7752 m^2 /g and the pore size were found to be 13.3909 Å to 15.4260 Å. It was found to that the pore diameters of rice husk activated carbon were increasing (13.3909 Å to 15.4260 Å) when the activation temperature was increased. Surface analysis of activated carbon was done by scanning electron microscopy (SEM), FTIR analyses in order to identify the appearance and disappearance of functional groups during different activation temperatures. From the data obtained rice husk activated carbon obtained at 600^0C could be used as a low-cost adsorbent with favourable surface properties.

Key words: Rice husk, Activated carbon, Physical and Chemical activation, FTIR, 1N KOH

1. INTRODUCTION

Activated carbon, also called activated charcoal or activated coal is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption of chemicals[1-2], heavy metals[3-4], toxic chemicals,separation of gases, recovery of solvents, removal of organic pollutants, petrochemicals etc. Activated carbon is well known for its porosity and adsorption capacity thus it is used in different industries for a vast verities of application. In short we can tell that it can be made from almost all types of carbonizable materials such as bituminous coal[10], pistachio shell[11], coconut shell coir pith[12], cassava peel[13],firewood[14-15], oil-palmshell[16-17], sugarcane bagasse[18-19],babassu[20], corncob[21-22],agricultural[23-26] ,waste[27-28] ,chicken waste [29-30]etc, and also can be used in almost all industries. Although activated carbon was the first recognized absorbent and is still widely used in industry, the development of appropriate methods to make them and the understanding of their porous structure still continue. The specific surface area of activated carbon ranges usually from 500 to 3000m2 g-1. The complex network of pores of activated carbon has been classified into macropores (diameters<2nm),mesopores(diameters2–50nm),and macropores(diameters>50 nm).Different types of activated carbons can be produced with specific characteristics depending on the raw material and activation technique used in their production, a noticeable point of activated carbon is that depending up on the need and necessity its pore size, pore volume and surface area can be altered or made by changing its physical activation temperatures or chemical activation Conditions, or the chemical used to activate or materials used to impregnate on to it. The general process to produce activated carbon is based on carbonizing and activating the original carbonaceous material. Activation may be achieved either physically or chemically. In carbonization of physical activation, the starting material is pyrolyzed at temperatures below 800^0C in an inert atmosphere so that volatile matter is removed, leaving behind rudimentary structures in the carbon material, this is followed by impregnation of strong oxidizing agents like HCL,KOH.NaOH etc.Different processes have been used for the treatment of wastewaters, including microbial degradation, chemical oxidation, chemical precipitation, ion exchange, membrane filtration; chemical reduction electrodepositing, reverse osmoses and, these technologies are frequently ineffective or too expensive. Activated carbon has been widely used in the sorption of chemical species from aqueous solutions as a versatile adsorbent with optimal sorption properties. However, production and regeneration of commercial activated carbons is still expensive, and cost effective alternative adsorbents have been the target of recent research for environmental protection. Rice husk is a cheap and abundant agricultural by-product found in India. More over as far as now, much studies has been not carried out in rice husk especially a temperature oriented study has been not much explored, all these factors together served as a motivation for material selection.

2. EXPERIMENTAL

2.1 PREPARATION OF ACTIVATED CARBON

100g of 5 rice husk samples were weighed and then subjected to physical activation in a muffle furnace, temperature ranging from 300^0C,400^0C,500^0C,600^0C,to700^0C for 1 hr ,and the samples were weighed after physical activation .Samples thus obtained were soaked in 1M KOH in 1:1 ratio for 24hrs followed by weighing the sample in order to know the impregnation of 1M KOH to the samples and is followed by activation in muffle furnace at temperature 300^0C for 2hr. The carbonized material was washed with distilled water to remove the free alkalis and dried at 100 ± 5^0C 2hrs and weighed to calculate the yield.[30-32]

2.2 CHARACTERIZATION OF ACTIVATED CARBON

The physico- chemical characterization of carbon samples prepared has been done; the yield of activated carbon is the % amount of activated carbon produced at the end of the activation process. This value indicates the activation process efficiency. The surface of activated carbon prepared has been analyzed using SEM (Scanning Electron Microscope), in order to identify the functional group responsible for adsorption Fourier transform infrared spectroscopy (FTIR) analysis was carried out.BET surface of both samples were identified using nitrogen adsorption–desorption isotherms, along with this pore size, pore volume created in samples were also identified.

3. RESULT AND DISCUSSIONS

The preparation of activated carbon has been carried out from rice husk at 300^0C to 700^0C and chemical impregnation has been done with 1 M KOH in 1:1 ratio, the yield of KOH activated carbon has been decreased gradually when activation temperature has increased from 300^0C to 700^0C after physical activation, but after chemical activation the yield has been increased up to 600^0C temperature range due to KOH impregnation. The porosity has been not much changed when temperature increased, but pore size has been found increased when temperature increased, moisture content, decolourising power, ion exchange capacity was also increased slightly when activation temperature increased, phenol adsorption capacity has been found increasing highly when activation temperature increased. Matter soluble, pH, bulk density and conductivity have not much varied according to rise in temperature.

The surface area of rice husk KOH activated carbon has found to be increased up to the temperature 600^0C (313.5100 M^2/g Langmuir Surface area), but got decreased at 700^0C .This is due to increase in pore diameter and microspore volume. In rice husk KOH activated carbon the least surface area found to be 13.9821 M^2/g at 300^0C (Langmuir Surface area).

Table- 1 Yield of rice husk carbon under different activation methods

SL NO	Activation temperature	Initial weight in gm	Yield in gm	Yield after chemical activation in g
1	300^0C	100	41.42	50.60
2	400^0 C	100	39.40	46.60
3	500^0 C	100	38.55	50.62
4	600^0C	100	37.39	56.66
5	700^0C	100	36.79	47.03

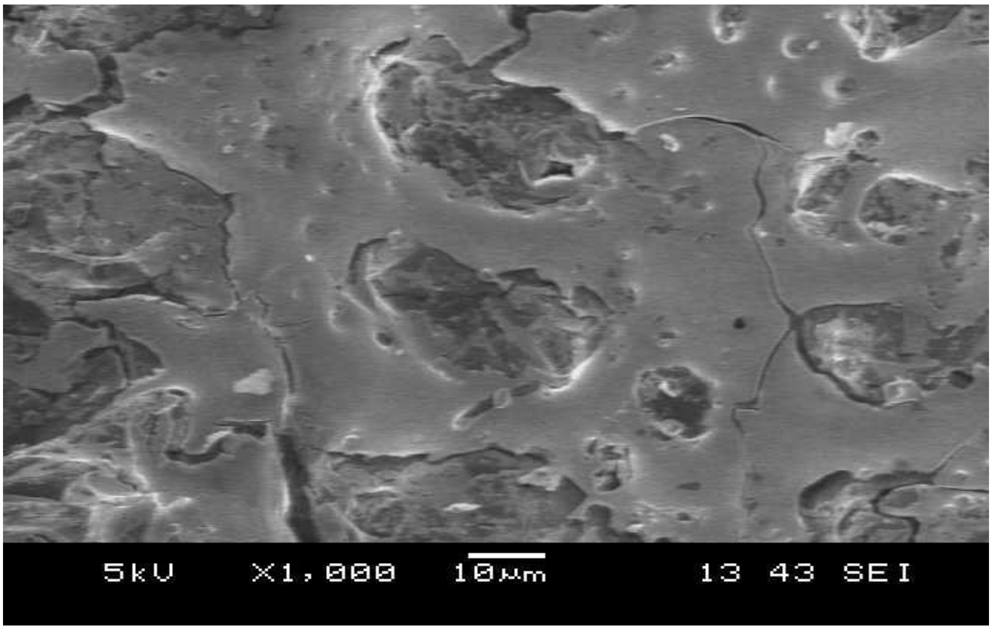

Figure-1 SEM analysis of KOH Activated carbon sampl

Table- 2 Characteristics of Rice husk activated carbon prepared

SL NO	Parameters	300^0C	400^0C	500^0C	600^0C	700^0C
1	pH	8.04	8.14	8.14	8.22	8.06
2	Conductivity ($S{\cdot}m^{-1}$)	0.14	0.13	0.11	0.10	0.09
3	Bulk density	1.1	1.0	0.90	1.1	0.83
4	Porosity Å	>17.140	>17.193	>17.135	>17.144	>17.142
5	Specific gravity	1.74	1.76	1.71	1.73	1.735
6	Ion exchange capacity	0.112	0.113	0.125	0.099	0.125
7	Decolorizing power	3.0	3.75	5.25	5.8	3.75
8	Pore size/value Å	13.3909	14.3912	15.1097	15.3031	15.4260
9	BET Surface area in m^2/g	11.9985	21.1059	138.8019	279.7752	264.0239
10	Langmuir Surface area in m^2/g	13.9821	24.0988	156.0854	313.5100	295.5394
11	Moisture content in3.0g	0.11	0.20	0.35	0.80	0.24
12	Phenol adsorption capacity in %	25.6	66.66	87.4	94.6	95.8
13	Matter soluble in %	0.026	0.031	0.029	0.035	0.015

Table -3 FTIR analysis

SL NO	Temperature	Functional groups identified in FTIR analysis
1	300^0C	CH3,C=O,C=C conjugated,intra molecular H bond,C=O non conjugated.
2	400^0C	CH3, CH=CH-cis, C=C conj,C=O non-conj intra molecular H bond, free OH
3	$500\ ^0C$	CH3,CH=CH-cis,,C=C conj,C=O non-conj intra molecular H bond, free OH.
4	600^0C	CH_2,C=C conj=O non-conj intra molecular H bond, free OH.
5	700^0C	CH3, CH=CH-cis, C=C conj=O non-conj intra molecular H bond, free OH.

The FTIR analysis shows the presence of functional groups present in the surface of activated carbon. It shows that C=O, functional group has been removed when temperature has raced in to 600^0C and 700^0C and conj=O bond has been appeared in the surface of activated carbon.

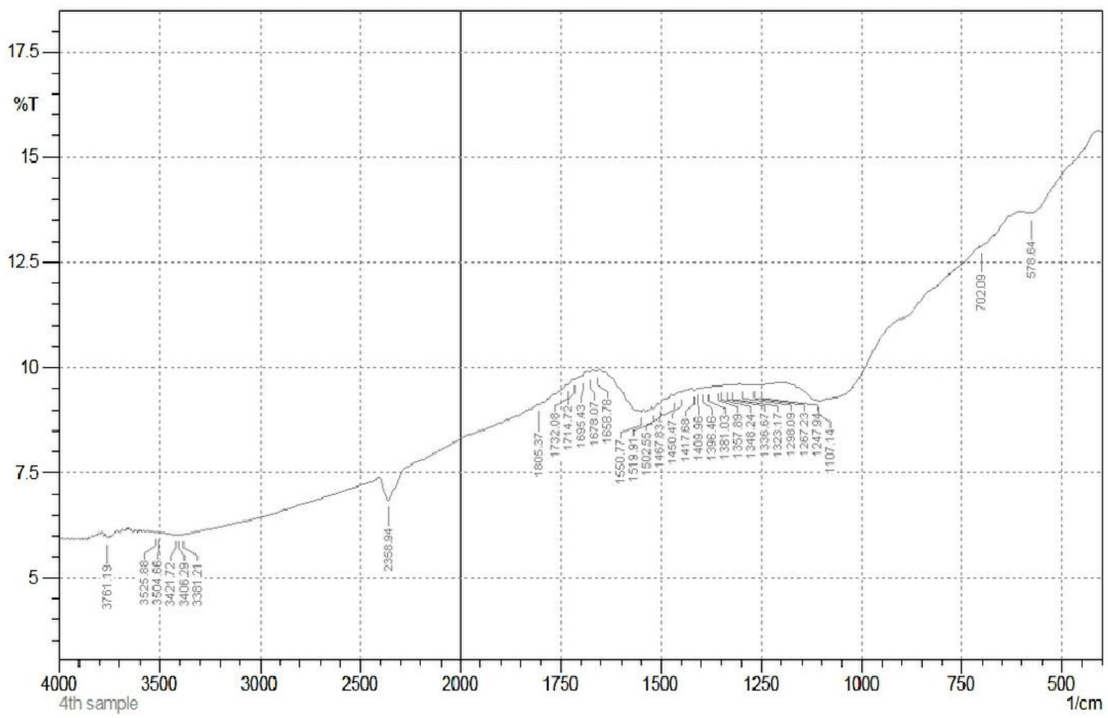

Figure-2 FTIR analysis

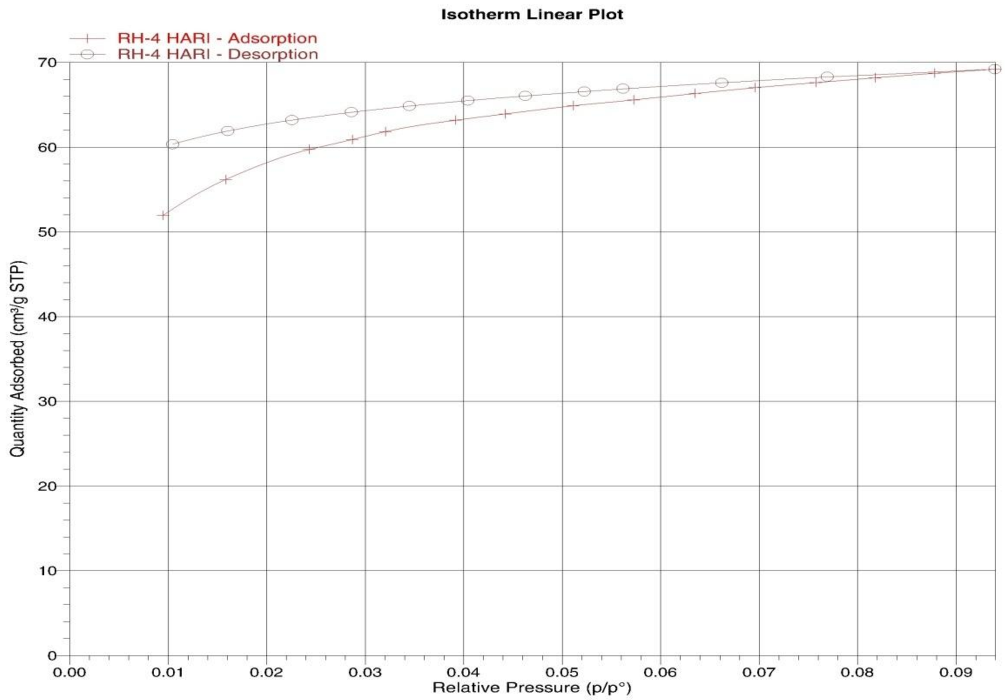

Figure -4 Isotherm plot of rice husk activated carbon

4. CONCLUSION

Due to the presence of high surface area, porosity, decolorizing power the activated carbon prepared from the agricultural waste rice husk, can be used for a variety of environmental application, dye removal, wastewater treatment and adsorption process too.

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