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Preparation and Characterization of Granular Activated Carbon from Corn Cob by KOH Activation

*Hiremath M. N.¹, Shivayogimath C.B.², Shivalingappa S.N.³

¹B. E. C. Bagalkot and Assistant Professor, Department of Civil Engineering, Anjuman Institute of Technology and Management, Bhatkal-581320, Karnataka, INDIA ²Department of Civil Engineering, B. E. C.Bagalkot, Karnataka, INDIA ³STJIT, Ranebennur, Karnataka, INDIA

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Abstract : In the present work studies related to the production of granular activated carbon, by using agricultural waste corn cob granules (dried and sieved to 12x16 mesh size), as a precursor material and potassium hydroxide (KOH) as an activating agent are presented. Precursor material was analyzed for elemental analysis, and the effect of carbonization temperatures for 600, 700, 800 and 900°C is examined. Higher BET surface area of 940 m² /g was observed for temperature of 800°C. The results show that agricultural waste corn cob can be used as a promising precursor material for the production of low cost activated carbon.

Keywords: Activated Carbon, BET Surface Area, Carbonization, Corn Cob, KOH Activation

Introduction

In the present world, activated carbon plays very important role as adsorbent, due to its unique distinguished properties. Activated carbon, also called as 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 or chemical reactions. Adsorption is the adhesion of atoms, ions, bimolecular or molecules of gas, liquid, or dissolved solids to a surface. This process creates a film of the adsorbate on the surface of the adsorbent. Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements(be they ionic, covalent, or metallic) of the constituent atoms of the material filled by other atoms in the material. However, atoms on the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption or chemisorption (characteristic of covalent bonding). It may also occur due to electrostatic attraction. Therefore because of physisorption and chemisorption properties, activated carbons are therefore widely used in gas purification, gold purification, metal extraction, medicine, sewage treatment, air filters in gas masks and filter masks along with water and wastewater treatment and many other applications.

Activated carbons can be prepared from large number of precursor materials with a high carbon content and low level ash including coal, woods and bones ^[1]. In addition, some agricultural by-products residues from agriculture and agro industries can be also used as raw materials for preparing activated carbons. These includes olive stones^[2], almond shells^[3], apricot and peach stones^[4], maize cob^[5], linseed straw^[6], saw dust^[7], rice hulls^[8], cashew nuts^[9], etc. Besides these other sources are, coconut shells, eucalyptus bark, linseed cake, tamarind seeds, and tea waste ash suffocated coal, baggase, ground nut husk, activated bauxite, palm seed coat, de-oiled soya, cement kiln dust. Activated carbon can be prepared either by physical (or dry) activation or chemical activation. Physical activation involves two steps; in first step precursor material will undergo carbonization process (600-900 °C) in the absence of air, and second step involves activation/oxidation by gases like carbon dioxide, oxygen and steam, etc.

^[10-14] where as chemical activation is one stage process, carbonization and activation of a precursor occur synchronously in the presence of oxidative chemicals. From the literature it has been observed that, studies on utilization of corn cob as a precursor material for the preparation of activated carbon are minimal, only few researchers tried corn cob as precursor material for the production of activated carbon.^[15-18] In addition there was no considerable work done on preparation of granular activated carbon from corn cob, therefore the aim of this study is to describe the feasibility of the production of granular activated carbon by using agricultural waste corn cob granules (dried and sieved to 12x16 mesh size),as a precursor material and potassium hydroxide (KOH) as an activating agent, under varied temperatures of 600,700,800 and 900^{0} C.

Material and Methods

The chemicals, raw materials, and methodology to prepare activated carbon used in experimentation work are discussed below.

Chemicals and Reagents Used: All the chemicals and reagents used were of analytical grade and procured from Merc Specialties Private Limited, Mumbai. Following are the chemicals used in this study.

- I) Potassium Hydroxide (KOH Pellets)
- II) Hydrochloric acid of 3 N (HCL)

Materials: Dried whole corncobs which were crushed and separated from pith/chaff are sieved to mesh size 12x16 (1.2-0.7 mm) as precursor material that is corn-cob granules of 1.2-0.7 mm grain sizes were obtained from Venkateshwara Agro Products, Pvt Ltd, Gadag.

Characterization of Precursor Material: Precursor materials which were used in this study were analyzed for elemental analysis using Elemental Analyzer (CHNSO), Thermo Flash EA 1112 model.

Table 1 Elemental analysis corn cob

Composition	Value in %	
Carbon	44.0	
Hydrogen	7.0	
Oxygen	47.0	
Nitrogen	0.4	

Activation: 25g of KOH pellets were dissolved in 1 liter of distilled water, then oven dried; Cooled precursor material that is corn-cob granules of 100 grams were thoroughly mixed in the KOH solution in a beaker, and agitated on a magnetic stirrer at 80 degree Celsius for 1 hour; the beaker is taken out of the magnetic stirrer and material is strained out, cooled and washed 4 to5 times with distilled water till one get pH of washed water near to 7. Then the washed precursor material is kept in the oven for 24 hours at $110\pm$ 5 degree Celsius; after 24 hours the oven dried material were taken out of oven and cooled to room temperature, then the material is kept in the muffle furnace for carbonization process at 600° C for 2 hour. After two hours of carboneous process the materials were taken out, cooled and kept in a beaker which contains 3N HCl, for 30 min at

80 degree Celsius on a magnetic stirrer, after this once again the said material is washed with distilled water for 4 to 5 times or till one get pH of washed water near to7. Then the material is kept in a oven for 12 hours under 110 ± 5 degree Celsius for drying. After 12 hours of oven drying, material is taken out of the oven and used for further studies. The procedure is repeated for different temperatures of 700, 800 and 900°C separately to assess the effect of carbonization temperature on characteristic properties of prepared activated carbons.

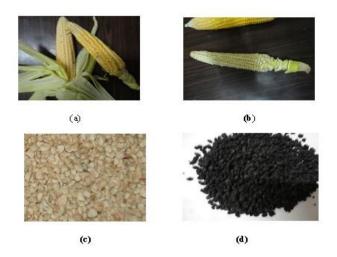


Figure 1: Images of (a) maize (b) corn cob (c) corn cob granules (d) prepared activated carbon

Characterization of Prepared Granular Activated Carbon: The porous properties of the prepared activated carbons were analyzed to observe developed SEM images as replica of their porous surfaces and surface area analysis were carried out using a scanning electron microscope JSM -5610LV and gas sorption analyzer NOVA 2200, Quatachroma respectively.. Other tests were carried out as per the requirement of IS 2752: 1995.

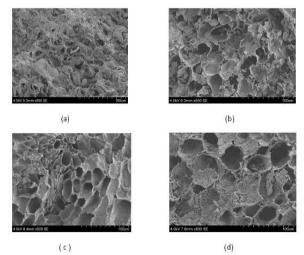


Figure 2: SEM Images of granular activated carbon carbonized at (a) 600 °C (a) 700 °C (a) 800 °C (a) 900 °C

Table 2					
Requirements for Granular Activated	Carbons as per IS 2752: 1995				

S. No.	Characteristics	Requirement For Type 1 (For use as a base for respirator carbons and solvent recovery)	Requirement for Type 2 (For use in water treatment)
1.	Adsorption capacity for carbon	55	
	tetrachloride, percent by mass, Min		
2.	Moisture, percent by mass, Max	5	5
3.	Ash, percent by mass, Max	5	6
4.	Hardness number, Min	90	90
5.	Retentivity index, percent by mass, Min	45	
6.	Adsorption capacity in terms of iodine number, <i>Min</i>	900	450
7.	Half dechlorination value, cm,	4	7
8.	Surface area, m2/g, <i>Min</i>	900	550

Table 3

Surface area, ash content and moisture content of prepared activated carbon

S. No.	Test Conducted	At 600° C	At 700° C	At 800° C	At 900° C
1.	Ash content %	6.8	6.2	3.9	6.4
2.	Moisture content %	4.3	3.6	2.8	3.4
3.	Surface area m ² /g	565	672	940	888

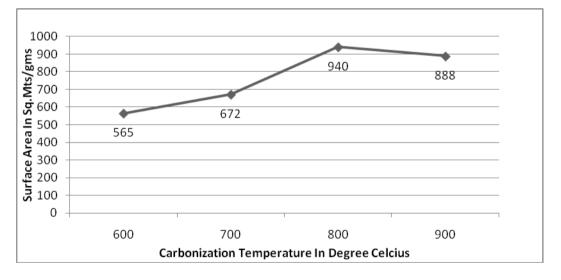


Figure 3: Effect of carbonization temperature on surface area

Results and Discussion

Results of various tests conducted to study the feasibility of preparation of granular activated carbon from corn cob by KOH activation under varied temperatures are tabulated in table 3.

The results shown in table 3 revealed that, higher surface area of 940 $m^{2/}$ /g was observed for carbonization temperature of 800°C, because of the generation of new meso and micro pores, which was comparatively higher

than 565, 672 and 888 $m^{2/}$ /g carbonized at 600°C, 700°C and 900°C respectively. Surface morphology examined by SEM clearly revealed the surface texture and porosity of the adsorbent with holes and small openings found on the surface indicating that it would increase the contact area and facilitate pore diffusion during adsorption, it was also observed that material carbonized at 800°C was fragmented and highly porous surface texture compared to other SEM images. The effect of activation temperature is presented in figure 3 clearly shows that as the carbonization temperature increases surface area also increases this is because, at lower temperature chemical residues left in the carbon block the pore structure, even washed with acid after carbonization process, there by reducing pore volume. It was also observed that at 900°C there is little reduction in the surface area this is because, higher temperature leads to excess carbonization, excess carbonization leads to the formation more ash there by produces larger diameter pores by pore widening and pore collapse, this also result in a decrease of surface area.

Conclusion

Present Study of describing feasibility of the production of granular activated carbon, by using agricultural waste corn cob granules (dried and sieved to 12x16 mesh size) as a precursor material and potassium hydroxide (KOH) as an activating agent, under varied temperatures of 600,700 ,800 and 900°C. The results and SEM images clearly reveal that, the prepared carbon at 900° C has higher surface area of 940 m²/g compared to other three carbons, carbonized at 600,700, and 800° C with surface area of 565, 672 and 888 m^2/g respectively. The results also indicates that the carbon carbonized at 900°C with higher surface area of 940 $m^{2/}$ /g satisfies specifications and requirements of IS 2752: 1995 of granular activated carbons, and can be used as low cost adsorbent in place of costly commercial carbons in water and wastewater treatment.

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