

Preparation and Characterization of Activated Carbon from Jute Stick by Chemical Activation: Comparison of Different Activating Agents

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Abstract

Activated carbons were prepared from jute stick by chemical activation using ZnCl_2 , H_3PO_4 , and H_2SO_4 . The influence of activating agents and carbonization temperatures ranging from 300°C ~ 350°C were studied. The properties of the carbons were characterized by iodine adsorption and the Fourier-transform infrared spectroscopy (FT-IR) method. The results of iodine tests showed resemblance close to the values found in literature 500~1200 (mg/g). FTIR results revealed the existence of O-H, C=O, C=C, C-O and C-H bonds.

Keywords: Jute Stick, Charcoal, Activated Carbon, Iodine Adsorption.

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INTRODUCTION

Activated carbon (AC) obtained from agricultural by-products has some advantages like efficiency and low cost as well as environment-friendly procedure (Martin *et al.*, 2003). The abundance and availability of agricultural by-products make them good sources of raw materials for activated carbon production (Malik *et al.*, 2007). Activated carbon production from agriculture waste materials has attracted researchers in recent years (Kadirvelu *et al.*, 2003, Prahas *et al.*, 2008; Ioannidou *et al.*, 2007). Finding cheap raw materials for producing activated carbon is a difficult task. That's why different agricultural waste materials like corn cob (Tsai *et al.*, 1997), coconut shell (Hu *et al.*, 1999), palm shell (Daud *et al.*, 2000), apple pulp (Sua *et al.*, 2002), chickpea husks (Hayashi *et al.*, 2000), grain sorghum (Diao *et al.*, 2002), pistachio nutshell (Lua *et al.*, 2004), jute fiber (Senthilkumar *et al.*, 2004), olive stones and walnut shell (Martinez *et al.*, 2006), cherry stones (Olivares *et al.*, 2003), coir pith (Ash *et al.*, 2006), wild rose seeds (Gurses *et al.*, 2006), rice bran (Suzuki *et al.*, 2007), gopher plant (Ozgul *et al.*, 2007), oil palm shell (Tan *et al.*, 2007) rubber tree seed coat (Hameed *et al.*, 2008), cotton stalk (Deng *et al.*, 2009) examined to produce AC.

Jute is a potential natural fiber, besides, emphatic textile usage, it can be used for making pulps and various products. As jute grows abundant in the subcontinent, researchers are always focusing on jute and its bi-products jute stick. Jute stick is a good source of carbon (Banerjee *et al.*, 1985). The production of AC from jute is one of the promising ways now a day for fulfilling the crisis of raw material. But ACs is highly adsorbents due to their high surface area, controllable pore size distribution, and amendable surface activity. AC production is involved in two stages at first carbonization and then activation. Carbonization removes the non-carbon elements (Bansal *et al.*, 1988). The porosity of the charcoal that find after activation is not then completely accessible. The purpose of the activation process is to increase the pore structure of the charcoal. Activation can be done in two ways. Physical or thermal activation completes by steam, carbon dioxide, air, or a mixture of these. Chemical activation involves activating the charcoal by chemical agents like H_3PO_4 , ZnCl_2 , H_2SO_4 , KOH, FeCl_3 , etc. By chemical activation from the carbonized product (charcoal), AC with well-developed porosity (after appropriate washing) may be obtained in a single operation. Here, Charcoal was prepared from jute stick. Activating agents H_3PO_4 , ZnCl_2 , H_2SO_4 were used. The present work was therefore, undertaken to prepare the activated

carbon from jute stick charcoal to diverse use in various industrial purposes.

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MATERIALS AND METHODS

Materials

Jute sticks used in this study were provided by Bangladesh Jute Research Institute (BJRI). The whole experimental works had been carried out in the Industrial Chemistry Laboratory, Chemistry Division, Bangladesh Jute Research Institute (BJRI), Dhaka and

Chemicals

Activated carbon preparation chemicals such as H_2SO_4 , H_3PO_4 , $ZnCl_2$, Iodine, Sodium Thiosulfate, Starch Solution, Potassium Iodate, Hydrochloric Acid, (Merck, Germany) were procured from local market in Bangladesh. All reagents were laboratory grade and were used without further purification. Table 1 shows the preparation of activated carbon from jute stick charcoal.

Table 1: Preparation of activated carbon from jute stick charcoal

Sample No.	Activated carbon with chemicals
A	Activated carbon preparation with 10% H_2SO_4
B	Activated carbon preparation with 10% H_3PO_4
C	Activated carbon preparation with 10% $ZnCl_2$

METHODS

Charcoal Preparation

Jute stick used in this study were provided by Bangladesh Jute Research Institute (BJRI). The jute stick as received was first washed with distilled and deionized water and dried in an oven at $85^\circ C$ for one day. Then the washed jute stick was grounded in a coffee mill, and the fraction passing through 50 mesh US Standard sieve was used in the preparation. After

that, it was kept in a muffle furnace at $300^\circ C \sim 350^\circ C$ for 3 hrs. After that, prepared charcoal powder as stored in a desiccator. When charcoal was at room temperature, it was taken from the desiccator, and weight is calculated. Then, Analysis of fixed carbon (SNI 13-3479-1994), ash content (SNI 3478: 2010), volatile matter: (SNI 13-3999-1995), moisture in the charcoal (SNI 13-3477-1994) was calculated and shown in Table 2.



Fig 1: Preparation of activated carbon from jute stick charcoal

Table 2: Composition of charcoal powder from jute stick

SI No	Name of the content	Result
1	Fixed Carbon	71.86%
2	Moisture	5.31%
3	Ash Content	4.73%
4	Volatile Matter	18.10%

Preparation of Activated Carbon from jute stick charcoal with chemicals

At first, 10% sulfuric acid has prepared. Then by maintaining Charcoal: Acid = 1:10, charcoal powder soaked with 10% sulfuric acid solution. The charcoal soaked with the sulfuric acid sample was kept at room temperature and it was kept for 5 ~ 6 hrs. Soaked

samples were washed with distilled water to avoid an excessive amount of acid solution. Then pH was checked to ensure neutrality. After that sample was filtered by a vacuum dryer and Then the sample was dried in an oven at a temperature of $70^\circ C$ to $80^\circ C$. The dried sample was collected and weighed in the electric balance. In the same way, Activated Carbon was prepared from 10% phosphoric acid and 10% Zinc Chloride.



Fig 2: Activated carbon from charcoal with H_2SO_4



Fig 3: Activated carbon from charcoal with H_3PO_4

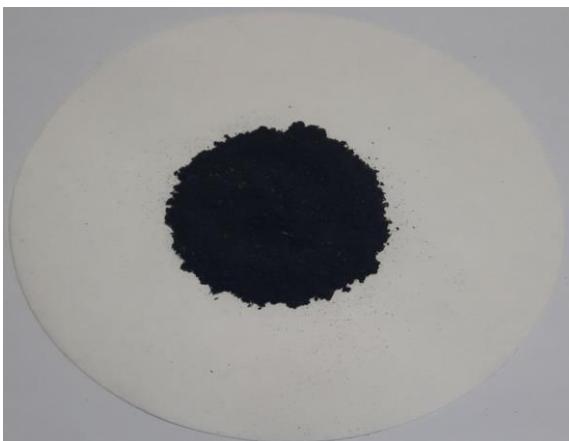


Fig 4: Activated carbon from charcoal with $ZnCl_2$

Determination of Iodine Number of Activated Carbon

At first (5% by weight) Hydrochloric Acid Solution was prepared. Then, Sodium Thiosulfate (0.100 N) was prepared, and Allow the solution to stand at least 4 days before standardizing. The solution should be stored in an amber bottle. After that, Standard Iodine Solution (0.100 ± 0.001 N) was prepared and stored in an amber bottle. Potassium Iodate Solution (0.1000 N) was prepared and then mixed thoroughly and store in a glass-stopper bottle. Starch Solution was prepared and this solution should be made fresh daily. Then Standardized the Solutions of sodium thiosulfate and iodine. The normality of sodium thiosulfate and iodine solutions was also calculated. At first, carbon grinded into powder form until 60 wt% passed through a 100-mesh screen. Cool the dry carbon to room temperature in desiccators. Then it transfers to a flask and 5% HCl was added dropwise until the carbon was completely wetted. It was then boiled and cooled to room temperature. Standardized the iodine solution just before usage. Then titration has done and calculated the iodine number according to ASTM4607.

RESULTS AND DISCUSSION

In activated carbon preparation, the yield is usually defined as the final weight of activated carbon produced after activation, washing, and drying, divided by the initial weight of raw material, both on a dry basis (Diao *et al.*, 2002). The activated carbon yield, R(%) was calculated using the following formula: $R(\%) = m/m_o \times 100$. Where, m and m_o are the dry weight of final activated carbon and dry weight of precursor, respectively. Preparation of activated carbon from jute stick charcoal by using various chemicals in different procedure were shown in Table-3. But in every procedure concentration was 10%. In this study, cellulosic fiber 10% concentration acid best result provides (Kim *et al.*, 2001). Different things were observed from the yield percentage of activated carbon stable. The yield percentage value was better if AC was prepared by activation with H_3PO_4 than the other two samples. Again other notable things observed Zinc Chloride activation shown good results than sulfuric acid.

Table 3: Yield percentage of activated carbons from jute stick charcoal

Sample No.	Time(hr)	Temperature($^{\circ}C$)	Activation Burn off (wt %)	Yield (wt %)
A	3	350	15	85
B	3	350	6	94
C	3	350	14	86

Iodine adsorption

Determination of iodine numbers is one of the methods to determine the adsorption capacity of activated carbons. It is a measure of the micropore (0–

20 \AA) content of the activated carbon by adsorption of iodine from the solution. The typical range is 500–1200 mg/g, which is equivalent to the surface area of carbon between 900 and 1100 mg/g (Mopoung *et al.*, 2015). As

we see the typical range is between 500 to 1200 mg/g and all prepared AC samples were in that range. Activation by phosphoric acid also showed good results

compared to other samples. It was also found that AC by Activation with ZnCl_2 has a lower Iodine adsorption number shown in Figure 5.

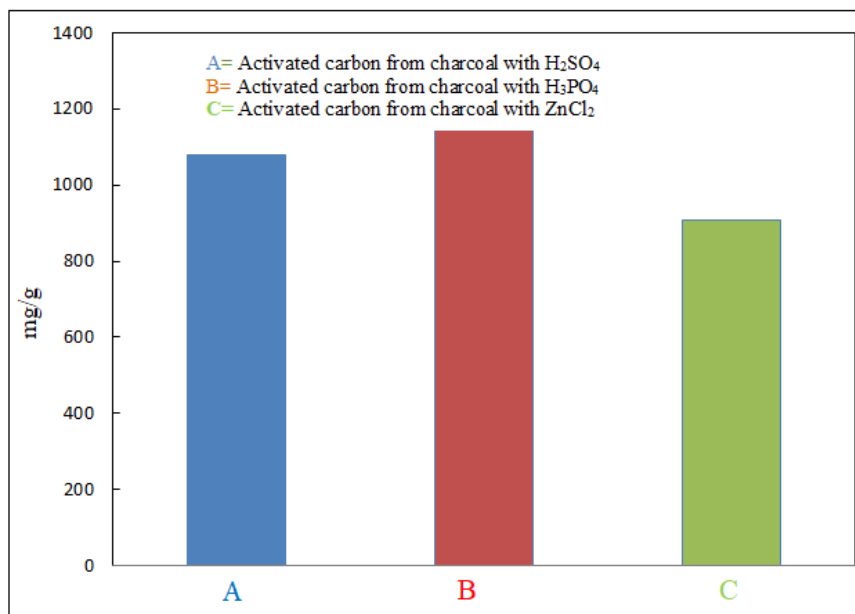


Fig 5: Iodine adsorption of activated carbon

FTIR analysis

FTIR is perhaps the most powerful tool for identifying types of chemical bonds (functional groups). The jute stick, impregnated sample with chemical agents and activated carbons (ACs) were analyzed by FTIR to clarify the structural changes after the activation and carbonization processes. The assignments of the absorption bands are listed in Table 4. The FTIR spectrum of A, B, and C activated carbons are shown in Fig 6. The broad and intense absorption peaks in the $3700\text{--}3100\text{ cm}^{-1}$ correspond to the O-H stretching vibrations of cellulose, pectin, absorbed water, hemicellulose, and lignin (Gundogdu *et al.*,

2013). The presence of the peak at $1740\text{--}1700\text{ cm}^{-1}$ in the spectrum indicates the carbonyl (C=O) stretching vibration of the carboxyl groups of pectin, hemicellulose, and lignin in A, B and C, of activated carbon (Hameed *et al.*, 2008). The peaks around $1620\text{ to }1580\text{ cm}^{-1}$ are due to the C=C stretching that can be ascribed to the presence of aromatic in lignin of jute stick. The bands in the extent of $1300\text{--}1000\text{ cm}^{-1}$ can be attributed to the C-O stretching vibration of carboxylic acids and alcohols (Angin *et al.*, 2014). The band in between $700\text{ and }900\text{ cm}^{-1}$ contains different peaks assigned to aromatic out of plane C-H bending with varying degrees of substitution (Mastalerz *et al.*, 1995).

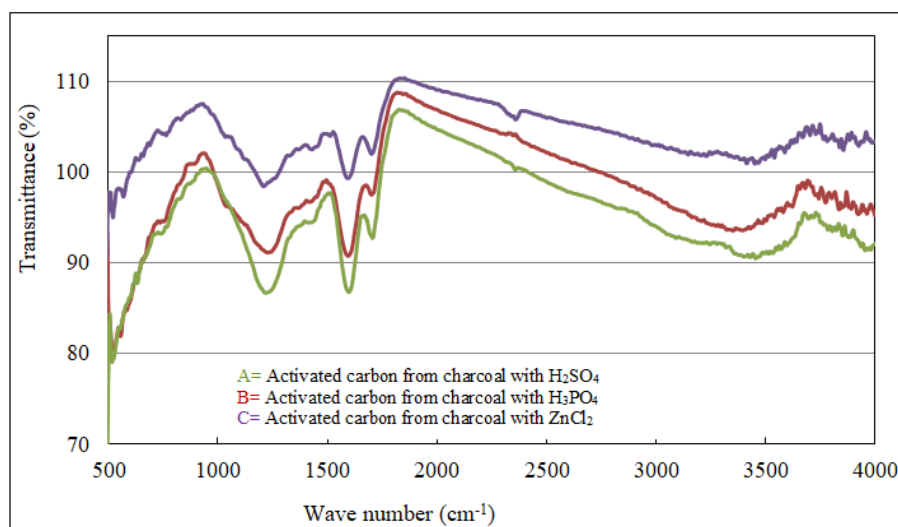


Fig 6: FT-IR spectra of activated carbon from jute stick charcoal

Table 4: Peak assignments of functional group of charcoal and AC from different chemical activation

Wave number (cm ⁻¹)	A	B	C
3600-3400 O-H stretching	3354	3456	3452
1740-1700 C=O stretching	1701	1706	1701
1620-1580 Aromatic C=C stretching	1594	1597	1593
1300-1000 C-O stretching	1225	1217	1208
900-700 stretching of C-H Group in alkane, alkene and aromatic group	744	630	757

CONCLUSION

There is the difficulty in analyzing the chemical properties of the surface derived from the special nature of activated carbon itself, which is a black body and has complex components on the surface, so the different characterization methods are proposed and used. As iodine adsorption number is a good indicator to identify the better and yield percentage is also important parameter both for research and economic purposes. From the Iodine adsorption result, we found that activation with the phosphoric acid result has given better AC compare to other two samples. The result of the work also demonstrated that yield percentage is higher in phosphoric treated sample as well as its Activation burn off (wt %) is lower. The jute stick charcoal is one of the promising raw materials for production of activated carbon for use in various industrial purposes.

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