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PRODUCTION OF ACTIVATED CARBON FROM RICE HUSK AND ITS PROXIMATE ANALYSIS

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ABSTRACT

With the increasing population, the pollution of the environmental elements is increasing day by day. Activated Carbon (AC) is solid, carbonaceous, non-hazardous, and highly porous complex compound and due to its adsorption property, this is widely used in the purification of various elements of the environment such as air, water, chemical, metal, etc. So, the enthusiasm in the production of activated carbon by the utilization of cheap agricultural and industrial wastes is growing rapidly. This study has been carried out to demonstrate the attempt of utilizing Rice Husk (RH) as it is abundantly available, environmentaly friendly, low cost and a kind of renewable precursor material for the production of activated carbon. The muffle furnace assisted alkaline activation with potassium hydroxide (KOH) at different ratios was performed. The effects carbonization temperature of 500°C and 700°C and impregnation ratios were evaluated on the basis of yield, volatile matter, carbon content, and activation burn-off. All in all, lower carbonization temperature 500°C gives better results with higher yield of 13.24% and 7.3% activation bunroff at 1:1 carbon to KOH ratio. The increasing ratio also decreased the volatile substances and comparatively better results obtained at less burning temperature.

Keywords: *Rice husk, activated carbon, impregnation, yield, proximate analysis.*

1. INTRODUCTION

Activated carbon (AC) is a form of carbon processed to have small, low volume pores that increase the surface area which is usually derived from charcoal. AC is a solid, tasteless, anamorphic black carbonaceous, non-hazardous, highly porous compound that has a complex structure made of carbon atoms with internal surface area ranging between 300-3500 m^2/gm. This surface area is related to its internal atomic structure that gives activated carbon the adsorbent property (Arnelli et al., 2019) and is composed of hierarchical pores named micro, meso and macropores with diameters <2nm, 2–50nm and >50 nm respectively. AC are characterized by their surface area, high micro-porosity and adsorption capacity, enabling them to be utilized as an effective adsorbent in the water treatment process (Poinern et al., 2015) and also for the removal of organic and inorganic pollutants (Pak et al., 2016; Gokce and Aktas, 2014; Korotta and Santhasivan, 2017). Application of ACs are most preferred not only due to their highly developed internal surface area, porosity and consequently a large adsorption capacity, but also due to their simplicity in design, and ease of operation (Bhatnagar and Sillanpaa, 2017).

The demand for using ACs is increasing day by day because of enhanced awareness about environmental protection. However, due to costly equipment and its maintenance, the production of commercial ACs are still very expensive for costly natural precursors such as wood, coal, etc. Therefore, much research has prompted finding lower production costs and eco-friendly alternative materials which can be transformed into low-cost AC. Previous researches illustrated that agricultural wastes can be suitable sources of raw material for the production of activated carbon including animal bones (Cechinel, et al., 2014), waste bamboo cums (Wang, 2012), mangosteen peel, sludge (Bjorklund and Li, 2017), waste tires (Betancur et al., 2009), durian shell, coconut shell (Iriarte-Velasco et al., 2008), rice husks (Chen et al., 2011; Foo and Hameed, 2011; Liou and Wu, 2009), nuts, sugar cane bagasse (Goncalves et al., 2016), rubberwood, molasses (Goncalves et al., 2016), and orange peel (Hashemian et al., 2014). Some factors such as cost, availability, ease of activation, renewability, inorganic content and carbon yield influence the choice of raw materials for the production of AC. Rice husk (RH) is an agricultural waste that has no commercial interest which usually ends up either being openly dumped or burnt instead of recycling. In Bangladesh, it is a major by-product of the rice milling industry. Moreover, globally the production of RH is over 120 million metric tons (Abbas and Santosh, 2010) and about 20% of the paddy weight is RH (Kumagai et al., 2007). Production of activated carbon from rice husk can increase the

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value of this agricultural waste and reduce the disposal cost at the same time can provide a cheap alternative to existing commercial activated carbon.

Rice husk has high percentages of carbon as contains lots of cellulose, hemicellulose and lignin. The surface of the carbon is tremendously increased by the removal of hydrocarbon through the physical change called activation. The synthesis of AC comprises two steps. Firstly, the dried RH is carbonized at lower temperatures (<800°C) to produce black char. Secondly, AC is prepared at higher temperatures either by physical activation (Alvarez et al., 2014) or by chemical activation (Isoda et al., 2014). The physical process requires the presence of steam or carbon dioxide (CO_2) and the chemical process requires activating agents e.g. potassium hydroxide (KOH), sodium hydroxide (NaOH), zinc chloride ($ZnCl_2$) and phosphoric acid (H_3PO_4) followed by activation in an inert atmosphere (Kalderis et al., 2008). Apparently, for recommended lower temperature for Chemical, the development of porous structure outcomes better as the porous framework can destroy at higher temperatures.

The characteristic of AC depends on its activation method and its Physico-chemical properties (Lua and Guo, 2000). Physical properties such as moisture content, volatile matters, fixed carbon, ash residues, iodine value, methylene blue value, pH value, conductivity (Sm-1), bulk density, specific gravity, decolorizing power and percentage of carbon (C), hydrogen (H_2), oxygen (O_2) value. Many studies have been carried out with potential and locally available rice husk as a raw material for low-cost adsorbent material and it is proved that activated carbon derived from rice husk shows better performance than industrial-grade activated carbon for decolorization of textile water (Rahman et al., 2013). However, a carbonization temperature-oriented study along with activating chemical ratio has not been much explored. This concern served as a motivation for our research. In this study, rice husk as a precursor material for the production of AC is proposed. However, an approach is done in this research of considering varying carbonization temperature along with impregnation ratio during AC production and carrying out a comparative study between products through proximate analysis.

2. METHODOLOGY

In this study, rice husk was used as the main raw material for the production of activated carbon where potassium hydroxide (KOH) was used as an activation agent. The Carbonization was performed at 500^{o}C and 700oC temperatures to produce rice husk carbon (RHC). The each rice husk carbon (RHC) produced at temperature of 500^{o}C and 700^{o}C, respectively were impregnated with KOH at four ratios of 1:0.25, 1:0.50, 1:1 and 1:2 (w/w). All weights were taken in digital balance in the characterization study. The whole process is shown with a flow diagram in Figure 1.

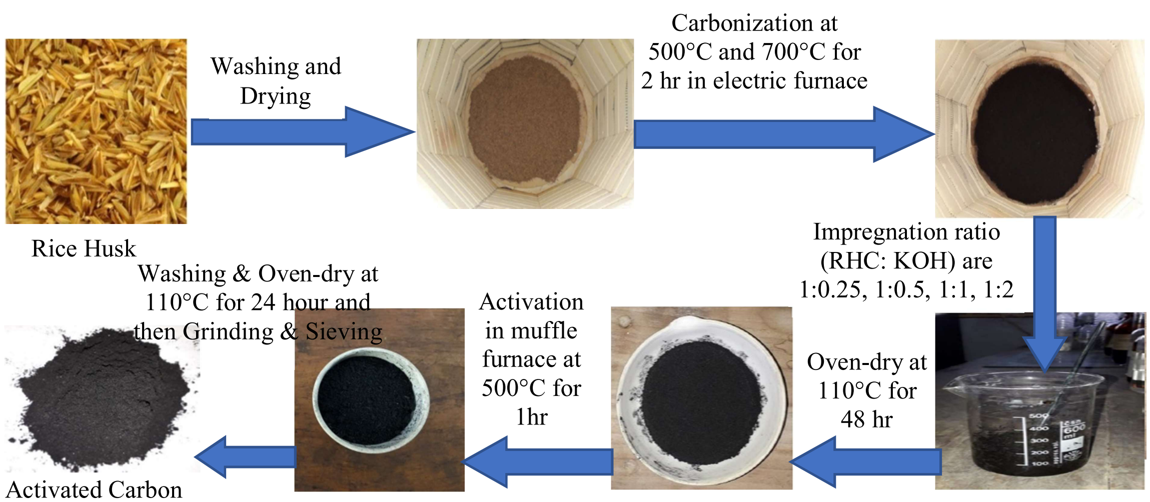

Figure 1: Flow diagram of ACs production procedure.

2.1 Preparation of Carbon from Rice Husk

Locally available rice husk (RH) was collected from Nabil Auto Rice Mill (unit-2), Rajshahi. Raw rice husk was washed with distilled water several times so that dirt and other substances could be removed initially from RH. Then it was placed in the oven at 60°C until the rice husk got completely dried. This washed and oven-dried rice husk was used for the preparation of rice husk carbon. That rice husk was then placed into the electric furnace (Model: SHTL-233). The temperature was fixed at 500°C and 700°C for burning and kept for about 2 hours.

After cooling the electric furnace, the rice husk carbon was obtained. The prepared carbon is shown in the following Figure 2.

Figure 2: Rice husk & Rice husk carbon.

2.2 Activation of Rice Husk Carbon

In this experiment, potassium hydroxide (KOH) solution was used as activating agent. For each temperature (500°C and 700°C) four ratios of rice husk carbon and potassium hydroxide solid were taken. The ratio of rice husk carbon and potassium hydroxide were maintained at 1:0.25, 1:0.50, 1:1 and 1:2 by weight. Two samples of 20 gm rice hust carbon produced at 700°C carbonizations temperature were mixed with 20 gm and 40 gm of KOH to obtain the ratio (carbon:KOH) of 1:1 and 1:2 respectively. Other two samples of 15 gm rice hust carbon produced at 700°C carbonizations were mixed with 7.5 gm and 3.75 gm of KOH for preparing the ratio of C: KOH as 1:0.5 and 1:0.25 respectively due to the shortage of sample. Distilled water of 100 ml was added in each carbon and KOH mixed sample and stirred thorougly. However, four samples of 20 gm of rice hust carbon produced at 500°C carbonization temperature were mixed with 5gm,10 gm,20 gm and 40 gm of KOH to maintain the ratio of 1:0.25, 1:0.5,1:1,1:2 (w/w), respectively. The samples were stirred for about 1hour and then placed in the oven at 120°C for 48 hours (Muniandy et al., 2014). The oven-dried impregnated carbon samples were put into the muffle furnace (Barnstead Thermolyne 47900 Furnace) for further activation at 500°C for 1 hour.

Figure 3: Final Activated Carbon.

After activation in the muffle furnace, the activated carbon was washed with distilled water several times till the activating agent removed completely and the sample got a neutral pH value. Then these all eight samples were oven-dried at 110°C for 24 hours. After which all samples were grounded by using pestle mortar and passed through 0.075mm sieve and collected the produced activated carbon (Muniandy et al., 2014). All the samples were stored in air-tight boxes for further analysis. The sample of produced AC can be shown below in Figure 3.

2.3 Proximate Analysis

Proximate analysis was done to determine the carbon content, volatile matters, activation burn-off,and product yield, for separate cases. The methods of these analyses are briefly described in the following sections.

Carbon content

Carbon content could be determined from the difference of weightsof samplesbefore and after burning in the electric furnace as ash-free. The following equation is used to calculate the carbon content where w_1 is the weight of dried rice husk and w_2 is the weight of produced ash-free carbon after burning in the furnace.

$$Carbon\ content\ (\%) = \frac{w_2}{w_1} \times 100 \quad (1)$$

Ash Content

Ash content of rice husk was determined as a percentage of rice husk from the weight of ash produced after burning at 550°C for 1 hr in the muffle furnace. Ash content was determined in this experiment following the standard ASTM E1534-93 method where the ash content prevails the remaining percent after muffle activation of the initial raw material of dried rice husk. The weight of produced ash is w_3 and the following equation is used to calculate.

$$Ash\ content\ (\%) = \frac{w_3}{w_1} \times 100 \quad (2)$$

Volatile matter

The mass losses are due to the release of volatile matters in a continual carbonization process with an increase in temperature. Therefore, the content of volatile matter can easily be determined from the differencein weight of carbon and ash from the weight ofrice husk taken for carbonization. The calculation is expressed as equation 3.

$$Volatile\ matter\ (\%) = \frac{w_1 - w_2 - w_3}{w_1} \times 100 \quad (3)$$

Activation burn-off

The loss of mass during the activation process varied with the variation of temperature (Saka, 2012). Burn-off refers to the loss of weight during the activation process. It can be determined with the difference between the weight of char with activation reagent and weight of product after activation. It can be expressed as equation 4 (Rhaman, et al., 2015).

$$Activationburn - off (\%) = \frac{(w_4 + w_5) - w_6}{w_4} \times 100 \quad (4)$$

Where, w_4, w_5 and w_6 are the weight of carbon before activation, the weight of activation reagent and the weight of carbon after activation, respectively.

Yield of AC

Yield is the percentage of the final product with respect to the original raw material. It could be determined from the mass of AC produced finally and the mass of rice husk used for the production.The yield of activated carbon was estimated with equation 5. The weight of the final product of activated carbon after washing for complete removal of residual alkali and drying is denoted as $w_7$and the weight of rice husk used is w_1.

$$Yield\ of\ activated\ carbon\ (\%) = \frac{w_7}{w_1} \times 100 \quad (5)$$

3. RESULTS AND DISCUTIONS

The produced activated carbon from rice husk was characterized with proximate analysis and determined the yield, carbon content, volatile matter, and activation burn-off. The obtained results are presented in Table 1.

Table 1: Activated Carbon Characterization.

Carbonization Temperature	Carbon Content (%)	Volatile Matter (%)	Ash Content (%)
500°C	14.96	63.22	21.82
700°C	14.28	63.81	21.91

The results demonstrate that there is no significant effect of temperature on the production of carbon from rice husk and it is around 15% of the raw material. Similarly, while comparing between samples, it can be noticed that the percentages of volatile matter of rice husk obtained at 500°C and 700°C are the same (63.22% and

63.81%) which means that volatile matter has been completely removed at or below the temperature of 500°C. As volatile matter works as an obstruction for the activating agent, the complete removal of the volatile matter before activation (during the production of carbon) from the raw material indicates an excellent property of precursor material for the production of activated carbon. Furthermore, the ash content obtained was 21.82% and 21.91% at 500°C and 700°C, respectively. Rhaman, et al. (2015) obtained fixed carbon 14.40% and Sharath, et al. (2017) obtained 20.99% from rice husk. Therefore, a comparable quantity of carbon was produced in this study.

The mass decreased during activation was called activation burn-off and this varied with varying activation temperature (Rhaman, et al., 2015). The extent of activation of the char depends on the extent of burn-off. The yield of activated carbon and activation burn-off for rice husk carbon produced at 500°C and 700°C with different ratios of the activating agent are presented in Figure 4. It is observed that the activation burn-off is increasing with the increase of activation agent and temperature. On the other hand, the yield of activated carbon decreased with the increase of activation agent and temperature.The yield of activated carbon decreased with the increase of temperature due to the increase of activation burn-off with an increase of temperature. Furthermore, the yield strongly depends on the amount of alkali used (Rhaman, et al., 2015).

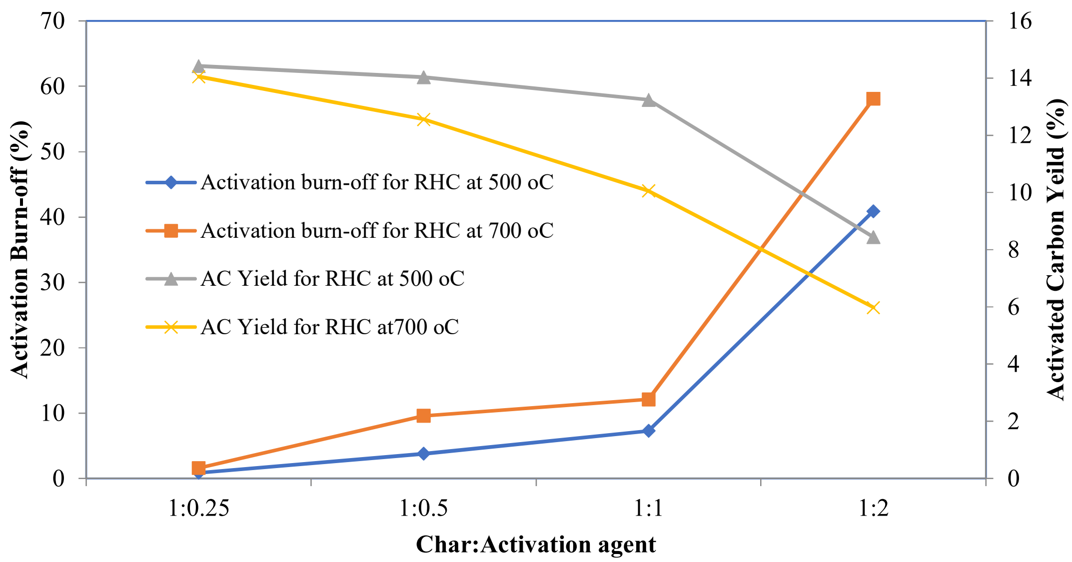

Figure 4: The relations of activation and yield of activated carbon with the ratio of activation agent.

Chang, et al. (2000) showed that the higher temperature char has a more developed pore structure. Pore development in the carbon would enhance the surface area and pore volume of the activated carbon by promoting the diffusion of activating agent molecules into the pores and thereby increasing the agent-carbon reaction via acid hydrolysis processes which would then create more pores (Hong et al., 2012.). Here below can be seen typical SEM image of rice husk, rice husk char and activated carbon in (a),(b) and (c) section of Figure 5 respectively presented by (Hanum et al., 2017).

Also, high temperatures can reduce the activation time of the same burn-off. Thus, a higher temperature can overcome the drawbacks of a longer period of activation time required to attain a larger surface area and offer a higher potential to produce activated carbon of greater adsorption capacity. The higher burn-off develops microporosity and widens the micropore by removing the obstructing agent from the char (Carrott et al. 2003). It is due to the more reduction of volatile substances at higher temperatures and with higher content of activation agent. However, a higher amount of alkali used for the activation to obtain a high surface area with increased porosity might not be economical sometimes considering the production and material costs.

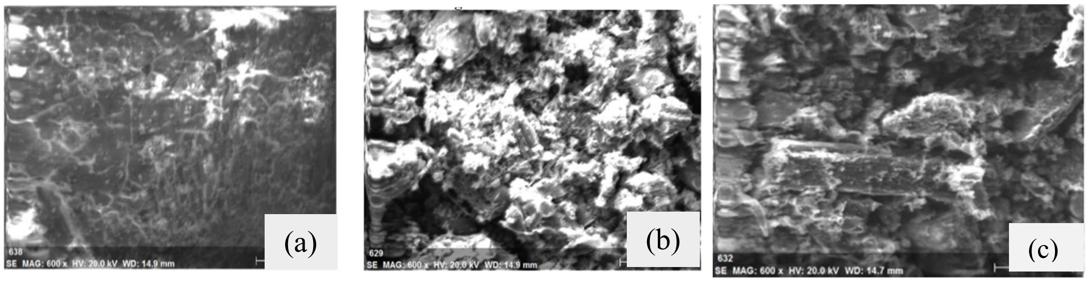

Figure 5: (a) SEM of rice husk; (b) SEM of rice husk char; c) SEM of activated carbon (Hanum et al., 2017).

Hidayu, et al. (2013) produced activated carbon from oil palm empty fruit bunches and activated with superheated steam at 765 ^oC for 77 min. Martínez-Mendoza, et al. (2020) produced activated carbon from Bituminous and subbituminous coal in two stages. Carbonization was carried out using an atmosphere of N2 with a flow of 0,65 L/min and a temperature of 850 ^oC for 2 hours. In the second stage steam (700 ^oC and 850 ^oC) was used as the activating agent with a flow of 0,275 L/min for 1.5 hours and 2.5 hours. Duan, et al, (2012) conducted experiment for the production of activated carbon from coconut shells. Carbonization was performed in a horizontal tube furnace by electric heating at a temperature of 600 ^oC for 2 hours under N_2 gas flow (100 cm^3/min). The steam activation was carried out in a horizontal electric tube furnace and in a self-made microwave tube furnace at 900 ℃ by conventional heating and microwave heating. However, muffle furnace assisted activation with KOH was incorporated after carbonization at 500 ^oC and 700 ^oC following two steps process in the present study.

4. CONCLUSIONS

In this study, rice husk-based activated carbon is produced with different alkali ratios (1:0.25, 1:0.5, 1:1 and 1:2 (w/w)) in two different carbonization temperatures and an approach of proximate characterization is done. The study showed the percentage of volatile matter is nearly the same for both temperatures. Although activation burn off increases with the temperature as well as the yield of activated carbon decreases. Although higher carbonization temperature 700^{o}C might provide better surface area, but the yield of activated carbon is lower. Again, higher alkali content can increase the porosity in activated carbon. However, it might be uneconomical production due to higher consumption of alkali. Therefore, activated carbon produced at 500^{o}C with 1:1 of carbon to activation agent would be profitable considering the activation burn-off, yield of activated carbon, carbon content and volatile matter.

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