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Research Article

PRODUCTION OF ACTIVATED CARBON FROM SOLID WASTE RICE PEEL (HUSK) USING CHEMICAL ACTIVATION

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ABSTRACT

Activated Carbon is an essential substance for many industrial activities. For instance, bleaching agent (in sugar factory) and for water filtration. Most of the Activated Carbon for industrial activities is being imported from other countries. However, there is no sufficient amount of production to satisfy the need in our country and the demand for Activated Carbon in the market is high. So, to satisfy the demand the Activated Carbon is being produced using solid waste Rice Husk. The purpose of this project is the preparation of Activated Carbon using a suitable rice husk. The Activated Carbon produced from Pyrolysis of rice husk was chemically activated with activating agent sodium hydroxide (NaOH). The chemically activated carbons were characterized by measuring yield percentage and bulk densities. The activated carbon produced from rice husk at different activating temperature of 650ºC, 700ºC and 800ºC exhibit a yield percentage of 48.2%, 47.65% and 45.95% respectively and corresponding bulk densities were 0.2 g/ml, 0.16 g/ml and 0.117 g/ml respectively. Proximate analysis also performed for precursor selection to choose the appropriate precursor. The quality of activated carbon is highly proportional to the dehydration rate of the sample and also on the process of removal of the volatile substances present in the precursor. According to proximate analysis, rice husk has a volatile matter of 68.06%, ash content 0.952%, fixed carbon content 20.988% and moisture content of 10%. This contributes to a total volatile content (easily escapable components) of about 68.06%. The proximate analysis of rice husk also reveals that the selected rice husk has good carbon content which is 20.988%. Therefore, proximate analysis served as an evidence for choosing rice husk as the precursor. Finally, a preliminary material and energy balance on pyrolysis or carbonization was performed.

INTRODUCTION

Recently, activated carbon has known as its large porous surface area and controllable pore structure. Activated carbons are carbonaceous materials and the materials starting from cheap natural precursors that can be produced by physical and chemical activation. The significant things are the high surface area and complex pore structure resulting from physical or chemical activation processes. Normally, rice husk/peel is treated as waste and disposed power plant sites, and leads to a serious environmental problem. Therefore, it is important to make full use of the husk/peel. Recently, rice husk/ peel is used as precursors to produce Activated Carbon (AC) (Liou, *et al.*, 2009), Zeo-lite (Panpa and Jinawath, 2009; Jang, *et al.*, 2009), Silica (Liou, 2004; An, *et al.*, 2011), Concrete (Gorhan, *et al.*, 2013), etc. With porous structure, high surface area and low cost, AC has attracted considerable attention and has been widely used as catalyst carriers (catalytic support), adsorbent to adsorb metal ions and organic

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molecules or as electrode materials for batteries and capacitors (Chen, *et al.*, 2011), etc. However, rice husk/peel is one of the most popular materials with low cost. In some reports, Activated Carbons were prepared from rice husks by chemical activation with KOH, NaOH, Na_2CO_3, $ZnCl_2$, and so on, at different temperatures for various times (Chen, *et al.*, 2011; Foo and Hameed, 2011). Activated carbon, by its wonderful structure, has been applied in various industries for several years: it is used for the separation of gases, recovery of solvent and removal of organic pollution (Bagheri and Abedi, 2009). The applications of activated carbons are also expanded, such as super-capacitors, electrodes and gas storage, (Yuan and Zhang, 2006; Oda and Nakagawa, 2003; Biloé, *et al.*, 2002) which require the activated carbons containing a higher surface area. The activated carbon structure properties have been demonstrated to be effective for removal of a wide variety of inorganic (Gupta and Rastogi, 2008; Gupta, *et al.*, 2008; Gupta, *et al.*, 1997) and organic, (Gupta, *et al.*, 2005; Gupta, *et al.*, 2002) pollutants dissolved in aqueous media, or from gaseous environment (Foo and Hameed, 2010).

Activated carbons have been prepared by many synthesis methods such as carbonization process, chemical agent activation, pyrolysis and microwave heating. The use of alkali hydroxides such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) as activating agents for the production of micro porous activated carbon had attracted great interest due to the valuable properties of the materials produced by this process. For preparation of activated carbons, conventional heating method is usually adopted. The previous researches have demonstrated the ability of rice husk-based activated carbon for many metal ions and organic molecules from aqueous phase adsorption. The aim of this work is to prepare a low-cost, high specific surface area activated carbon with micro-porous and meso-porous ranges using rice husk as the raw material. The precursor selection by using proximate analysis was performed. However, after proximate analysis results, it proves that the selected rice husk has good carbon content which is 20.988%. Therefore, proximate analysis served as an evidence for choosing rice husk as the precursor. The chemically activated carbons were characterized by measuring yield percentage at different temperature. The Bulk densities of AC samples were also studied at different temperatures and a preliminary energy balance on the pyrolysis or carbonization is also performed.

METHODOLOGY

Lab equipment and chemical required

This project was carried out in the laboratory, Wolkite University. A lot of chemical has been used throughout the project. Chemicals and lab wares used for the project were hydrochloric acid, sodium hydroxide, potassium chloride, distilled water, sieve, crasher, marker, plastic bag, PH meter, shaker, beakers, oven, spoon, stirrer, furnace, water bath, mass balance and nitrogen gas (Fig. 1 and 2).

Precursor selection

Precursor is the procedure that comes before the production of activated carbon and it specifies whether the process is continued or not.

Ash content

- The 10 g of rice husk was taken.
- The measured sample was burn for 1:30 hr by using furnace.
- The burnt sample was measured again.
- The ash content was calculated.

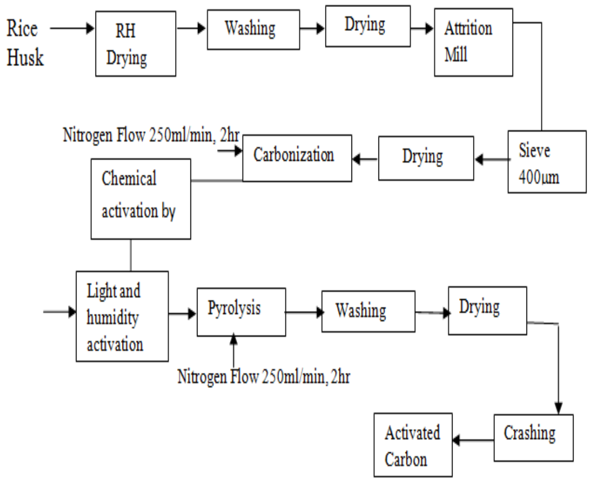


Fig. 1 Process block diagram for the production of AC.

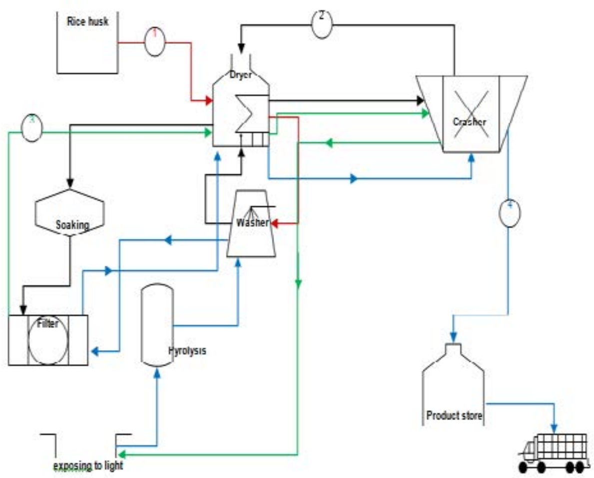


Fig. 2 Process flow diagram for the production of AC.

$$Ac = \frac{(\mathrm{Mc+Ma}) - \mathrm{Mc}}{(\mathrm{Mc+Ms}) - \mathrm{Mc}} \times 100$$

$$= \frac{219.5942 - 219.5}{229.5 - 219.5} \times 100$$

Ac = 0.952%

Where: - Mc = mass of crucible

Ma = mass of ash

Ms = mass of sample

Moisture content

$$Mc = \frac{(\mathrm{ww-wd})}{ww} \times 100\%$$

$$= \frac{50 - 45}{50} \times 100$$

Mc = 10%

Where: ww = amount of sample before drying

wd = amount of sample after drying

Volatility matter

$$Vd = \frac{100(B-C) - Mc(B-A)}{(\mathrm{B-A})(100 - \mathrm{Mc})} \times 100$$

$$= \frac{100(256.3\text{-}248.5)\text{-}0.1(256.3\text{-}246.3}{(256.3\text{-}246.3)(100\text{-}0.1)} \times 100$$

V_d = 68.06%

Where: A= weight of crucible

B= weight of crucible plus sample

C= weight of crucible plus sample after heating

Mc= moisture content

Fixed carbon content

% Fc = 100 - Mc -Vd -Ac

= 100 – 10 – 0.952 – 68.06

% Fc = 20.988%

Where: Mc= moisture content

Vd= volatility matter

Ac= ash content

EXPERIMENTAL PROCEDURE

To The first step to produce activated carbon was raw material collection. Raw material was rice husk and it collected from Gonder. The sample (rice husk) was washed to remove some impurities and dusts. And it was dried manually. The 50 g of washed raw material (rice husk) was measured. The measured rice husk was dried by using oven to get well dried sample and also to calculate the moisture content for 24 hours.

The sample was measured after drying and got 45 g rice husk. The dried rice husk was crashed manually. The last preparation was sieving by 600 µm size sieve. 30 gram of pretreated rice husk (600 µm size) was soaked into the 300 ml aqueous solution containing 1M of NaOH solution for 5 hrs. After chemical activation, samples were dried at 110°C for about 10hrsAfter drying; the rice husk was crushed again into a fine powder. And activated sample were exposed to light and humidity (L&H) for about 22 h to enhance the development of the pore structure during pyrolysis. The dried, chemically activated, and light and humidity treated sludge was placed into a furnace. The pyrolysis was carried out under a flow of nitrogen gas (70 ml/min) at 600-800°C for 1 to 2 hr. After the pyrolysis, the sample was cooled and removed from the furnace and crushed. After crushing the activated carbon was rinsed using of 1M HCl, and distilled water to remove excess activating agent and residual inorganic matter. Then the activated carbon was dried and stored.

RESULTS AND DISCUSSION

Proximate analysis

The Proximate Analysis result of rice husk determines the distribution of its contents. It may be noted that the volatile matter present in rice husk, contributes maximum to its contents. The moisture content present in the sample can also be considered as water vapor when it is heated to high temperatures. Hence, about 78.06% of the contents tend to leave the sample when heated, of which 68.06% is volatile matter and 10% is moisture content. The values from the Table 1 indicates that the ash content of the sample is 0.952% and also fixed carbon content of the sample is 20.988%. This gives an overview about the properties and components of rice husk Ash is a non-carbon or a mineral additive that does not combine chemically with the carbon surface. It consists of various undesired mineral substances, which become more concentrate on activation and comprises of 1-20% and primarily depends on the type of raw material.

High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity (Fig. 3).

Table 1. Proximate analysis

Rice husk content	Composition
Ash content	0.952%
Moisture content	10%
Volatility matter	68.06%
Fixed carbon content	20.988%

Yield percentage

The yield percentage result of chemical activation using sodium hydroxide is denoted in Table 2. From Table 2 it can be clearly noted that the yield of activated carbon is 48.2%, 47.65%, and 45.95 at 650°C, 700°C and 800°C respectively.

From Table 2 one can conclude that the yield of AC decreases as temperature increases. The yield at temperature of 650°C, 700°C and 800°C is 48.2%, 47.65% and 45.95% respectively. The activated carbon yield decreased with an increase in the carbonization temperature. The yield significantly decreased from 650°C to 700°C and then gradually decreased at temperatures higher than 800°C. This result indicates that the lower yield obtained at a higher temperature is caused by a much larger release of volatile matters (Fig. 4).

Bulk density

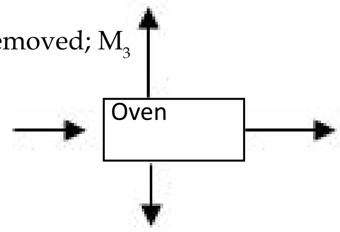
The bulk density is defined as the mass of a unit volume of the sample in air, including both the pore system and the voids between the particles. It is expressed as kg/m^3 on dry basis. The bulk density of activated carbon depends on the shape, size and density of the individual particles (Table 3) (Fig. 5).

MATERIAL AND ENERGY BALANCE

Material balance

Drying:

Moisture content removed; M_3



Rice husk

Table 2. Percentage of yield

Sample	Temperature °C	Yield %
1	670°C	48.2
2	700°C	47.65
3	800°C	45.95

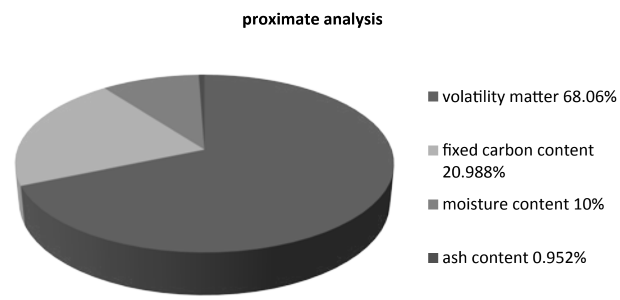


Fig. 3 Proximate analysis of rice husk.

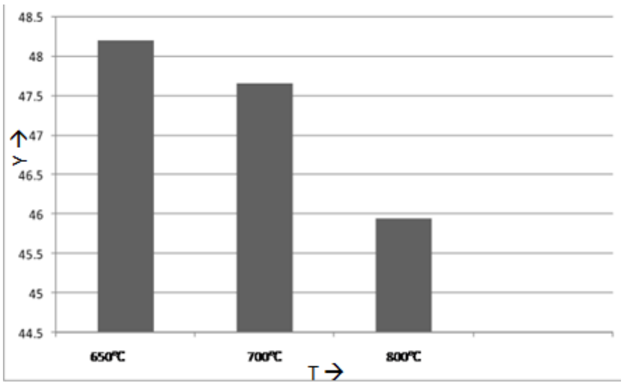


Fig. 4 Yield % vs. different temperatures of activated carbon using chemical activation.

Table 3. Bulk density of activated carbon at different activation temperatures

No.	Samples	Bulk density g/ml
1	AC at 650°C	0.2
2	AC at 700°C	0.16
3	AC at 800°C	0.117

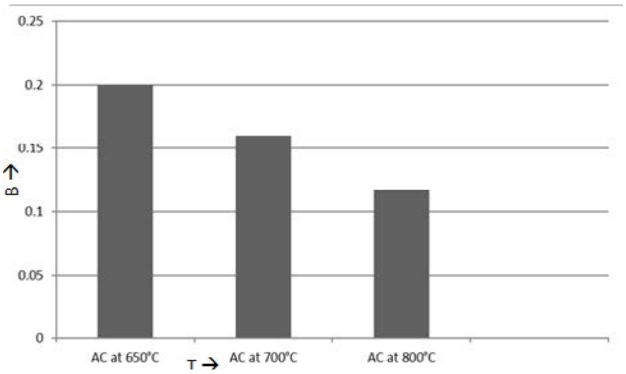


Fig. 5 Bulk density of activated carbon at different activation temperatures.

M_1=1000 kg/day

Dried rice husk, M_2

Lab scale

In=out

$M_1 = M_2$ (dried rice husk) + M_3 (moisture content)

50 g= 45 + M_3

M_3 = 5 g, then;

For small scale plant

M_1=1000 kg/day

When 50 g = 45 g

1000 kg =?

$$M_2 = \frac{45000}{500} = 900 \text{ kg/day}$$

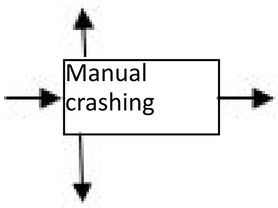
M_3 = M1 – M2

= 1000 kg/day – 900 kg/day

M_3=100 kg/day

Crashing:

Dried sample



Desired size M_2 = 900 kg/day

Rice Husk;

M_4=600 kg/day Granular sample, M_5

Lab scale

$M_2=M_3 + M_4$

45 g= 30 g+M_4

M_4= 15 g

Small scale plant

45 g=30 g

900 g=?

M_2=600 kg/day

$M_4= M_2-M_3$

=900 kg/day-600 kg/day

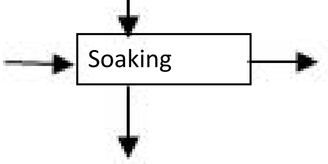
M_4=300 kg/day

Note: for small scale plant can't use manual crashing, we use miller.

Soaking:

M_6 (NaOH + H2O)

Desired size



Rice husk M_4=600 kg/day M_7

Lab scale

For 30 g sample, use 20 g NaOH

30 g=20 g

600 kg=?

Amount of NaOH for 600 kg/day sample $= \frac{12000}{30} = 400 kg/day$

20 g=400 g of H_2O

400 kg/day= Xg of $H2O = \frac{160000}{20} = 8000 kg/day$

Ratio of lab scale and small scale plant

Lab scale $= \frac{20}{40} = \frac{1}{20}$

$= \frac{400}{8000} = \frac{1}{20}$

Small scale plant $= \frac{400}{8000} = \frac{1}{20}$

The impregnation ratio is 2:3, this implies,

mNaOH = 400 kg/day

M_7= 600 kg/day + 400 kg/day NaOH + 8000 kg/day H_2O

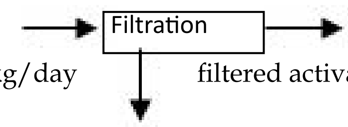
M_7=9000 kg/day

Filtration:

No loss of activated rice husk during filtration

95% of water and 90% NaOH will be filtered

In lab scale, 30 g of rice husk will be soaked by diluted NaOH and after drying the activated rice husk will be 33 g. This implies, $\frac{33-30}{30} \times 100 = 10\%$ of NaOH will remain in the rice husk. Thus,



M_7=9000 kg/day filtered activated

Rice husk=M_8

Filtrate=M_9

M_8 =5% of mass of H_2O + 12% NaOH + 600 kg/day = ((0.05*8000) + (0.10*400)) kg/day+ 60 kg/day =1040 kg/day

$M_9= M_7- M_8$

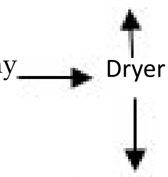
= 9000 kg/day to 1040 kg/day

=7960 kg/day

Drying:

Moisture removed (M_{10})

M_8=1040 kg/day



M_{11} (dried activated rice husk)

Assume

100% moisture removed

M_{10}=5%(400 g)

=0.05(8000) = 400 g

M_{11}= M8-M10

M_{11}=1040 kg/day-400 kg/day

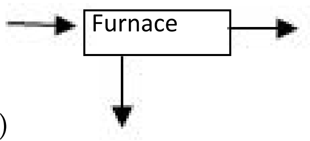
=640 kg/day

The dried activated rice husk was exposed to light and humidity so catch some amount of moisture.

=640 kg/day become 660 kg/day

Furnace

M_{11}=660 kg/day



M_{12} (gases

H_2O, NOX)

Or Volatility matter

M_{13} (activated carbon)

In lab 660 kg/day of activated rice husk will be pyrolyzed and will it of activated rice husk will it 318.12 kg/day of activated carbon.

For lab scale

M12=M11-M13

=33 g to 15.906 g

M12 = 17.094 g

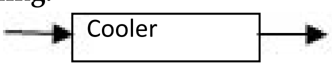
For small scale plant

660 activated rice husk

$= \frac{15.096}{33} \times 100\%, 48.2\%$ activated carbon was produced

Cooling:

M13

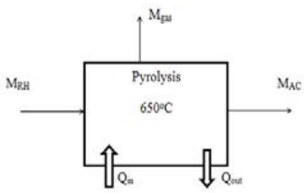


M4

M13 = M14=318.12

Preliminary energy balance on pyrolysis

The main energy consuming process on production of activated carbon is Pyrolysis or carbonization. While doing the energy balance of the system consider the rice husk and Pyrolysis product. Rice husk requires heating, and then undergoes a reaction resulting in phase change. The pyrolysis product requires cooling and condensation.



Where M_{RH} = mass flow rate of rice husk

M_{gas} = mass flow rate of gas

M_{AC} = mass flow rate of activated carbon

$E\ out = Ein + Egenerated - Econsumed - Eaccumulated + Elosses$

At steady state the accumulation of energy is zero. A well-insulated reactor will only have minor heat losses to the environment. Pyrolysis heat requirements between 200 J/g and 400 J/g were reported for various biomasses by Van de Velden *et al.* (2010). Therefore at a rice husk flow rate (for lab scale, of 33 g approximately 9.9 KJ) of 660 kg/day approximately 198 KJ could be required. During pyrolysis energy is required in the form of heat, to create the reactive conditions.

Thermodynamic properties for the energy balance

The enthalpy of evaporation and specific heat capacity for water were obtained from Cengel (Cengel (2003) and. The specific heat capacity of rice husk varies 2.1- 2.754 KJ/ kg.K at 650°C (Journal of Agricultural Engineering). The bio-gas specific heat capacity was estimated from CO and CO_2 be close to 1 KJ/ kg.K (www.engineeringtoolbox.com). Average C_P values were used over the temperature range 25°C to 650°C. From the references, for the thermodynamic properties are given as follows:

$Q_{in} = \Delta E_{total} = \Delta E_{heating} + \Delta E_{evap} + \Delta E_{rxn} = M_{(RH+water)} C_{P(avg)} \Delta T + M_{water} \Delta H_{evap} + (M_{(RH+water)} - M_{water}) \Delta H_{rxn} + M_{gas} C_{P(gas)} \Delta T$

For lab scale

=33 g*2.427J/g.K(650-25) K+1 g*2300J/g.

+32 g*9900J/g+16.094 g*1J/g.K (650- 25)K

= 379251.625 J = 379.216 KJ

For small scale plant

= (660 kg*2.427 KJ/ kg.K(650-25)K+ 20 kg*2300 KJ/ kg+640 kg*198 KJ/ kg+ 321.88 kg*1 KJ/ kg.K (650-25))/day

= 1372144 KJ/day =1372.144MJ/day =15.88 KJ/s = 15.88 KW

Therefore the design heating capacity for the reactor is 15.88 KW to account for losses. The design cooling capacity is also15.88 KW.

CONCLUSION AND RECOMMENDATIONS

Conclusion

The proximate analysis of the rice husk gave an insight of the nature of the precursor. Proximate results were very helpful in choosing the appropriate precursor. The quality of activated carbon is highly proportional to the dehydration rate of the sample and also on the process of removal of the volatile substances present in the precursor. According to proximate analysis, rice husk has a volatile matter of 68.06%, ash content 0.952%, fixed carbon content 20.988% and moisture content of 10%. This contributes to a total volatile content (easily escapable components) of about 68.06%. Therefore, proximate analysis served as an evidence for choosing rice husk as the precursor. To determine the optimum temperature of activation, the rice husk powder was activated at 650°C, 700°C and 800°C. The yield percentage of all the activation experiments was recorded and noted. This yield were plotted to determine a suitable optimum temperature. There are several methods for characterization of activated carbon using XRD, SEM and BET etc., but unfortunately on this project we could not perform any of the characterization due to the lack or absence of the necessary equipment and chemicals for the characterization in Wolkite University and Ethiopia.

This project was aimed at producing high quality powder activated carbon by an economically cheaper method. In this project rice husk was examined to produce an activated carbon through the chemical activation process using sodium hydroxide. Rice husk has longer availability and inexpensive material with high carbon and low inorganic content. The result obtained from this project will help in providing a solution for increasing demand of activated carbon with a minimum cost and more effective way.

Recommendations

A positive point of this project is that sample (rice husk) gets freely or for cheap cost. In the production of activated carbon in laboratory there are many equipment are required but most of the equipment are not available. Because of this limitation the expected final product couldn't be attained. The other one is characterization, characterization is very essential for determination of the quality of activated carbon specifically it helps to determine the adsorption capacity and specific surface area however these characterization were not performed and it is highly recommended to perform this using XRD analysis, SEM Photographs and BET for surface area etc., it would add quality on the product and also on the project.

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