



Proximate Analysis of Cane Bagasse and Synthesizing Activated Carbon: Emphasis on Material Balance

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Abstract

This study aims for proximate analysis of cane bagasse and preparation of activated carbon using a suitable sugar cane bagasse. Sugarcane bagasse was examined to produce an activated carbon through the chemical activation process. The bagasse has longer availability and inexpensive material with high carbon and low inorganic content. The activated carbon produced from pyrolysis of sugar cane bagasse was chemically activated with sodium hydroxide (NaOH). A systematic investigation of the effect of activation temperature on the properties of the activated carbon was done. The chemically activated carbon yield was investigated and the maximum yield percentage of the activated carbon prepared in this study was 14.5%.

Keyword: Activated carbon, cane bagasse, material balance, proximate analysis

1 Introduction

Proximate analyses are used to determine the characteristics of carbonaceous materials that may be used as precursor for active carbons. Proximate characteristics include contents of moisture, volatile materials, ashes and fixed carbon. Adsorption characteristics of activated carbon from cellulosic wastes are affected by the type of carbon, which is governed by the source of the raw materials and preparation procedures. Different raw materials and manufacturing processes produce activated carbon of different physio-chemical properties, thereby resulting in different adsorption characteristics. Activated carbon is nothing but carbon produced from carbonaceous source materials like nutshells, peat, wood, coir, lignite, and coal and petroleum pitch [1, 2]. It can be produced by any one of the following described processes:

a) Physical reactivation: By this process, precursor is developed into activated carbons using gases. This is generally done by using one or a combination of the following processes:

Carbonization: Material having appreciable carbon content is pyrolyzed at temperature ranging between 600–900 °C, in the absence of oxygen (usually in inert atmosphere with gases like argon or nitrogen)

Activation/Oxidation: In this process, raw material or carbonized material is exposed to oxidizing atmospheres (carbon monoxide, oxygen, or steam) at temperatures above 250 °C, usually in the temperature range of 600–1200 °C [1].

b) Chemical activation: Before carbonization, the raw material can be impregnated with certain chemicals. The chemical needs to be typically an acid, strong base, or a salt (phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride). After impregnation, the raw material needs to be carbonized at lower temperatures (450–900 °C). It is believed that the carbonization / activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material [1].

2 Methodology

2.1 Chemicals and materials

Chemicals and materials used for the research were hydrochloric acid, sodium hydroxide, distilled water, bagasse, filter paper, funnel, sieve, knife cutter, plastic bag, stove, conical flask, beakers, oven, spoon, desiccators, furnace and mass balance.

2.2 Raw material collection and pretreatment

Precursor (raw material) selection for the production of activated carbon was obviously the first step of the project. Conventionally, activated carbon is produced from carbonaceous source material such as sugar cane bagasse, wood, peat, coal, and wastes of vegetable origin (e.g. nutshells, fruit stones) [2]. Sugarcane bagasse is a new alternative as a replacement to existing product of activated carbon. Bagasse pitch is a waste product from sugar refining industry. It is the name given to the residual cane pulp remaining after sugar has been extracted. Bagasse pitch is composed largely of cellulose, pentose, and lignin. Sugar cane bagasse was collected from Wonji/Shoa Sugar factory and dried at a temperature of 115°C. The size was reduced to appropriate size of 5.6mm and sieved. After

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that, the bagasse was stored in plastic bag for further processing [2].

2.3 Proximate Analysis

The proximate analysis is a simple means of determining the distribution of products obtained when the coal sample is heated under specified conditions. As defined by ASTM D 121, proximate analysis separates the products into four groups: (1) moisture, (2) volatile matter, consisting of gases and vapors driven off during pyrolysis, (3) fixed carbon, the nonvolatile fraction of coal, and (4) ash, the inorganic residue remaining after combustion.

Proximate analysis is the most often used analysis for characterizing a material in connection with their utilization [3].

Moisture content determination: This method is used to determine the percentage of water in a sample by drying the sample to a constant weight.

Procedure: The weight of moistured sample was measured immediately and recorded as "wet weight of sample".

The wet sample was dried, at a temperature of 115°C using suitable drying equipment by putting the sample in an oven and then the oven was closed and the sample was dried for 3 hour [4].

Thereafter the oven was open and the dry sample was drawn into a container immediately.

Then the sample was cooled in desiccators to ambient temperature and the weight was measured. Finally the weight of cooled sample was measured again, and recorded as the "dry weight of sample".

To determine the moisture content of raw material, 30 grams of wet weight of bagasse have been taken [4].

Ash content determination: This method determines the total ash content of sugar cane bagasse.

Procedure

- 6gm sufficient dried bagasse was measured.
- Empty crucible was weighed.
- The sample was put into a crucible and the crucible was ignited in the furnace at 800°C for 1 hour 30 minutes.
- Then the crucible was placed in the desiccators.
- Finally, sample was cooled to room temperature and weighed

Volatile matter content determination: The sample was heated at 800°C for 8 minutes. The percentage of volatile matter was calculated from the loss in mass of the sample [5].

Procedure

- ✓ The temperature of the muffle furnace was adjusted to 800°C.
- ✓ The steady temperature condition in the furnace was maintained.
- ✓ The weight of crucible was measured.
- ✓ About 4g of the sample was weighed and the sample was put into a crucible. Aafter placing the lids on the crucible, it was transferred into the furnace and left there for 8 min at 800°C.
- ✓ Finally, it was allowed to cool, by placing the crucible in the desiccators, to room temperature and weighed.

Fixed carbon content determination

The fixed carbon content, FC, was determined by subtracting the sum of percentage compositions of moisture content, volatile matter content, and ash content from 100.

Preparation of activated carbon via chemical activation (NaOH)

- ✓ 50 gram of pretreated sugarcane bagasse (5.6 mm size) was soaked into a 750ml aqueous solution of NaOH solution for three different beakers overnight.
- ✓ The impregnation ratio between sugarcane bagasse and activating agent was maintained at 1:1(wt /wt). The sample was dried in an oven overnight at 120°C to remove water.
- ✓ The carbonization was carried out at three different activation temperatures 400, 500 and 600°C for a period of 90 min.
- ✓ After the activated carbon was cooled, it was temporarily stored in desiccators and then continuously washed using 0.1M hydrochloric acid in order to remove the activating agents and impurities.
- ✓ Then, the activated carbon was washed several times using distilled water in order to remove the HCl until clear filtrate was obtained.
- ✓ At the end, the product was dried in oven overnight and stored in plastic bags until it is needed for sugar decolorization process and further characterization [5].

2.4 Developed process flow diagram for activated carbon Production.

Process flow diagram for production of activated carbon was developed by identifying the major and minor equipment from laboratory experiments and literature.

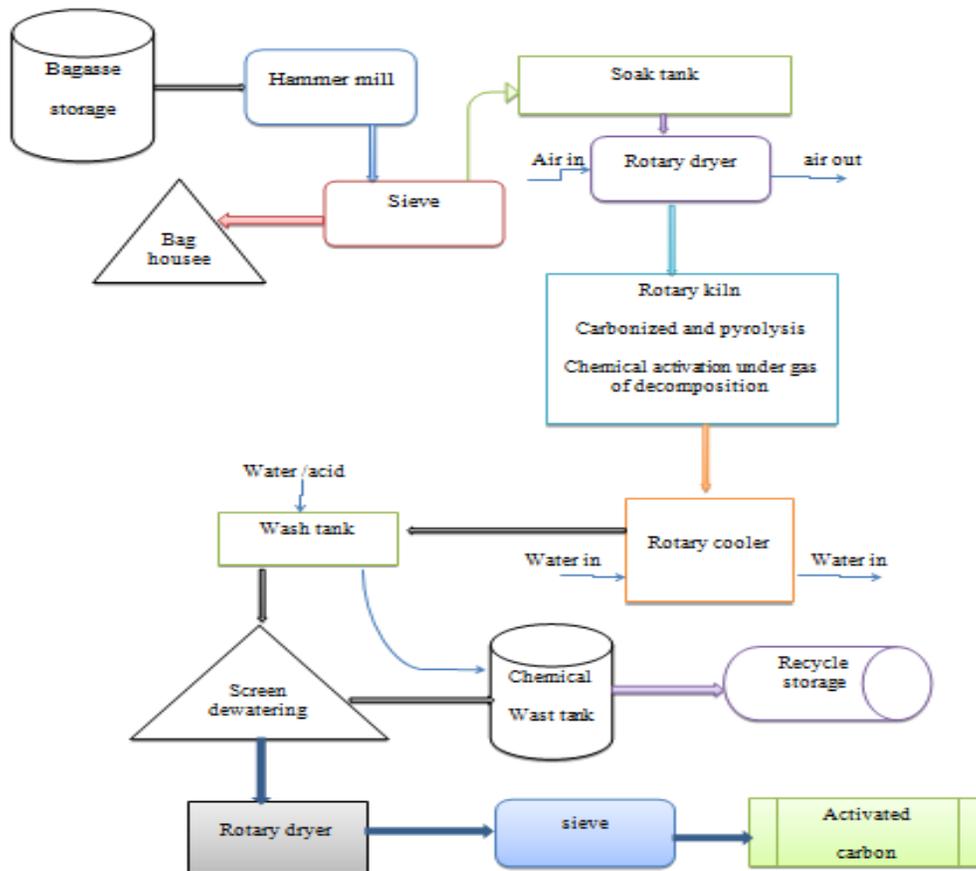


Fig 2. 1 Process flow diagram (PFD) for the production of activated carbon using chemical Activation.

3 Result and Discussion

3.1 Proximate Analysis

Moisture content determination: To determine the moisture content of raw material, 30 grams of wet weight of bagasse was taken. Out of these 22.8 grams of dried weight of bagasse have been found. The moisture content, MC, is given by:

$$Mc = \frac{ww - wd}{ww} * 100$$

where, Wd is dry weight bagasse, Ww is weight of bagasse before dry.

$$Mc = \frac{30 - 22.8}{30} * 100 = 24\%$$

Ash content determination:

Table 3.1 Ash content determination	
Mass of empty crucible(gram)	29.5
Mass of crucible and dry sample(gram)	35.5
Mass of crucible and ash(gram)	30.003

The ash content, AC, is given in % by:

$$Ac = \frac{Z - X}{Y - X} * 100$$

where, x is mass of empty crucible in gram, y is mass of crucible plus dried sample in gram and z is mass of crucible plus ash sample in gram.

$$Ac = \frac{30.003 - 29.5}{35.5 - 29.5} * 100 = 8.4\%$$

Volatile matter content determination: Mass of sample before heating was 4 gram and 2.19 gram was produced after heating. The volatile matter content on dry basis, Vd in% is given by:

$$Vd = \frac{F - G}{F} * 100$$

where, F is mass of bagasse before heating and G is mass of bagasse after heating.

$$Vd = \frac{4 - 2.19}{4} * 100 = 45.25\%$$

Fixed carbon content determination: The fixed carbon content was determined as follows

$$\%age Fc=100- Mc- Vd-Ac=100 - 24-8.4-45.25=23.35\%$$

Yield and % burn-off: % Burn-off is the percentage weight loss of pyrolyzed char on a dry basis, which occurs during activation, and yield is the mass of carbon left after pyrolysis and activation [7, 8].

These parameters were evaluated using Equations 1 and 2.

Burn off

$$Burr\ off = \left[\frac{(A - B)}{(A)} \right] * 100 \dots \dots \dots (1)$$

$$Yield = \left(\frac{C}{A} \right) * 100 \dots \dots \dots (2)$$

where, A is weight of sample before carbonization, B is weight of sample after carbonization and C is dried weight after wash

Table. 3.2. Activated carbon preparation data.

No.	Temp. °C	Weight of sample		Dried weight After wash	Yield %	%burn-Off
		Before carbonization	After carbonization			
1	400	50gm	34.3gm	7.25gm	14.5	31.4
2	500	50gm	29.5	6.7gm	13.4	41
3	600	50gm	27	6.0gm	12	46

The Proximate Analysis result of bagasse determines the distribution of its contents. It may be noted that the volatile matter present in bagasse, 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 69.25% of the contents tend to leave the sample when heated, of which 45.25% is volatile matter and 24% is moisture content. The values from Table 4.1 indicates that the ash content of the sample is 8.4% and also fixed carbon content of the sample is 23.35%. This gives an overview about the properties and components of bagasse.

Figure 3.1 shows the proximate analyses of bagasse. In general, biomass contains 3 main cellulosic components, that is, hemicelluloses, cellulose, and lignin. During the carbonization of biomass, the hemicelluloses and cellulose will decompose mostly into volatile products, while the lignin content is more difficult to decompose and contributes to the formation of a solid residue called char. It is observed that the fixed carbon and ash content decreased with increase in the volatile content.

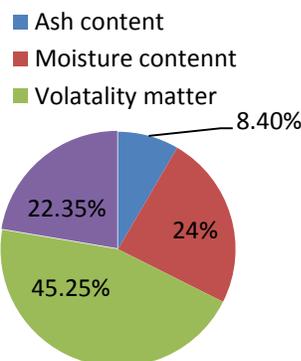
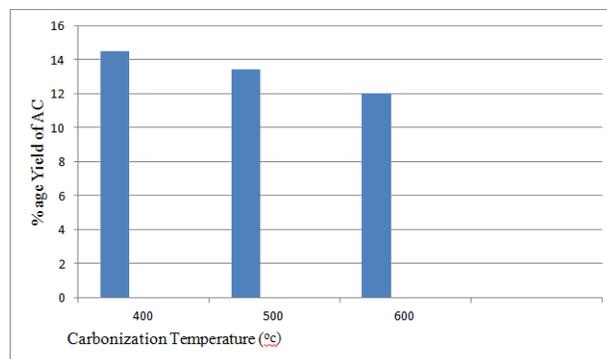


Fig 3.1 composition of bagasse.

3.2 Yield Percentage

The yield percentage result of chemical activation using sodium hydroxide is given in Table 2.2. From Figure 3.2, it can be clearly noted that the yield of activated carbon are 14.5%, 13.4%, 12% at 400°C, 500°C, 600°C, respectively. It is clearly evident that yield percentage is not the only sole criterion for optimization of activated carbon production.



From Figure 3.2, one can conclude that the activated carbon yield decreased with an increase in the carbonization temperature. The yield significantly decreased from 400 to 500°C and then gradually decreased at temperatures higher than 600°C. This result indicates that the lower yield obtained at a higher temperature is caused by a much larger release of volatile matters.

4 Mass Balance

Cane crashed per day at Wonji/Shoa sugar factory=6250 t/day.

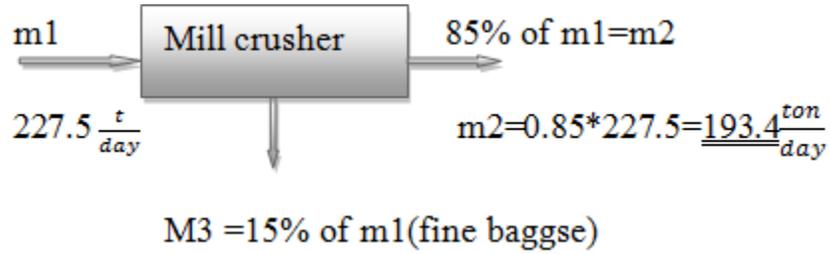
$$\begin{aligned} \text{Bagasse} &= 28\% \text{ of crushed cane} \\ &= 0.28 * 6250 \\ &= 1750 \text{ t/day} \end{aligned}$$

- Assume:
- ✓ Bagasse was sufficiently dried
 - ✓ Surplus=13% of bagasse

$$\text{Therefore amount of surplus} = 0.13 * 1750 = 227.5 \text{ t/day}$$

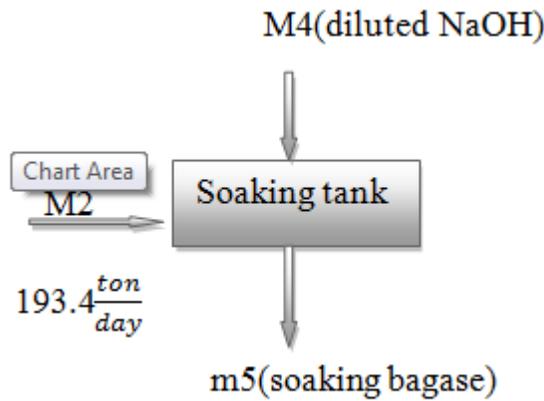
Fig.3.2. Percentage yield of activated carbon.

A. Mill crusher



$$\begin{aligned}
 M3 &= m1 - m2 \\
 &= 227.5 - 193.4 \\
 &= 34.125 \text{ ton/day}
 \end{aligned}$$

B. Soaking tank



The impregnation ratio is 1:1, that means $m2 = m4$ (NaOH) = 193.4 ton/day
 $M2 = m3 = 193.4$ ton/day
 For lab scale it requires 750 gram of H₂O to dilute 50 gram of NaOH, therefore

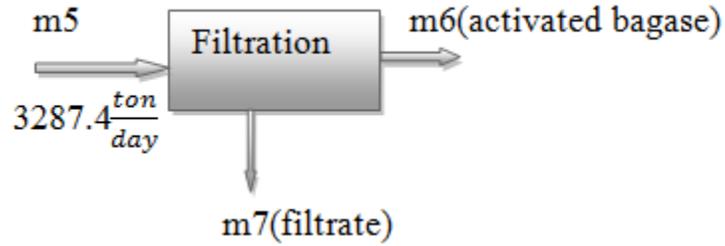
$$\begin{aligned}
 50 \text{g NaOH} &= 750 \text{g H}_2\text{O} \\
 193.4 \text{g} &= ?
 \end{aligned}$$

$$\text{mass of H}_2\text{O} = 193.4 \text{g} * \frac{750 \text{g H}_2\text{O}}{50 \text{g NaOH}} = M4 = 2900.6 \text{ ton/day}$$

$$M_{in} = M_{out} \rightarrow m5 = m4 + m2$$

$$\begin{aligned}
 M5 &= 3094 + 193.375 \\
 &= 3287.4 \text{ ton/day}
 \end{aligned}$$

C. Filtration



Assumption:

- No loss of activated bagase
- 95% of H₂O and 90% of NaOH will be filtered

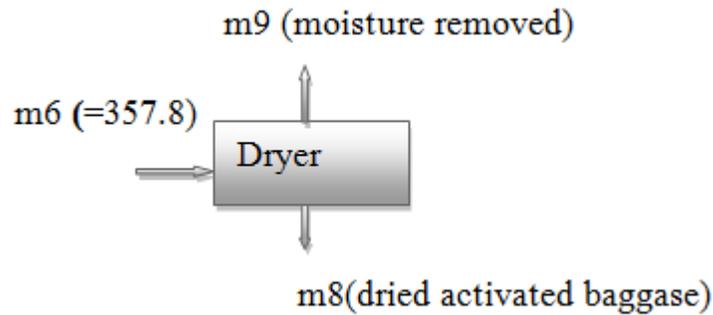
Thus, $m_7 = 95\%$ of H₂O + 90% of NaOH

$$= 0.95 * 2900.6 + 0.9 * 193.4$$

$$= 2929.6 \text{ ton/day}$$

$$M_6 = M_5 - M_7 = 3287.4 - 2929.6 = 357.8 \text{ ton/day}$$

D. Dryer



Assumption: 100% of moisture will be removed

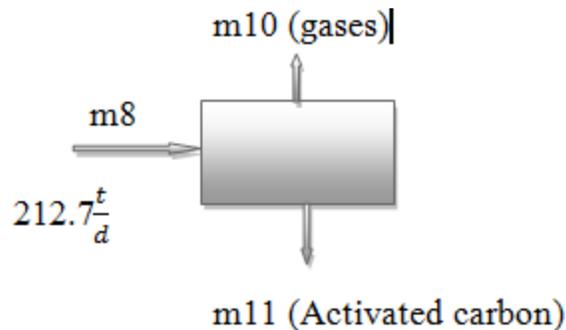
Therefore $m_9 = 5\%$ of H₂O = $0.05 * 2900.6$

$$= 145.03 \text{ ton/day}$$

Thus, $m_8 = m_6 - m_9$

$$= 357.8 - 145.03 = 212.7 \text{ ton/day}$$

E. Carbonization unit

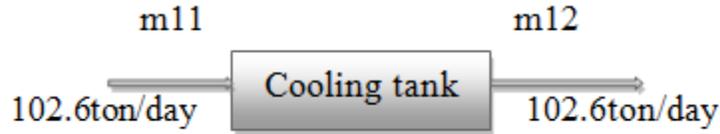


In lab scale 55g of activated bagase were pyrolyzed and we get 27g of activated carbon.

Assume activation temperature were 600°C, thus we can calculate m_{11} .

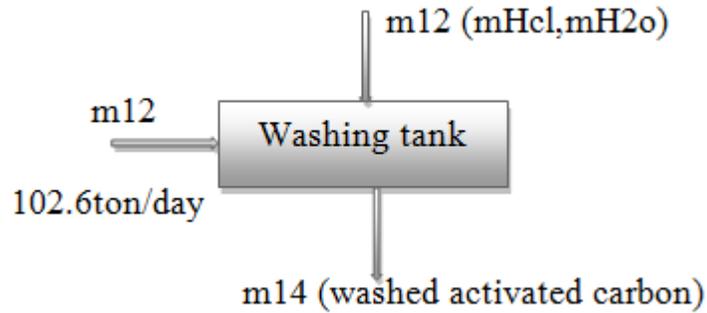
$$M_{11} = 212.7 * 27/56 \text{ t/d} = 102.6 \text{ ton/day}$$

F. Cooling



$M_{in}=m_{out}$, therefore $m11=m12$

G. Washing



Note washing includes both acid and water washing tanks.

In lab. Scale to wash 27g of pyrolyzed activated carbon it requires

$V=8.6\text{ml}$ of HCl , $HCl=1.18\text{g/ml}$

$=m/v$, $m= \square *v=1.18\text{g/ml} * 8.6\text{ml}$

$m=10.148\text{g}$ of HCl

And $v=1\text{litre}=0.001\text{m}^3$ of distilled water

$H_2O=1000\text{kg/m}^3$

$M_{H_2O}=\square *v=0.001\text{m}^3 * 1000\text{kg/m}^3$

$M_{H_2O}=1\text{kg}$ of distilled water

$m13=m_{H_2O}+m_{HCl}$

$M13=m12*(1000/27)/\text{day}$ of $H_2O+m12 (10.148/27)$ of HCl

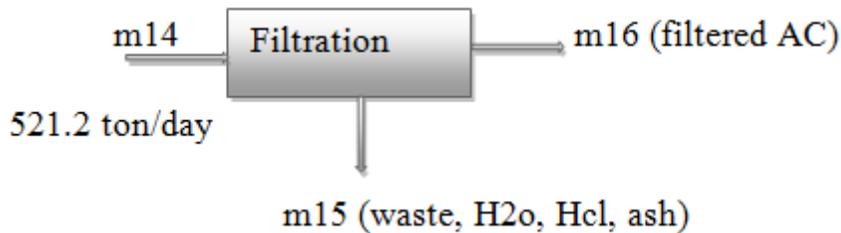
$M13=102.6*(100/27) + 102.6*(10.148/27)$

$M13= (380 + 38.6) \text{ ton/day}=418.6\text{ton/day}$

$M_{in} = M_{out}$, $m14 =m13 + m12$

$m14 = 102.6 \text{ ton/day} + 418.6 \text{ ton/day} =521.2 \text{ ton/day}$

H. Filtration unit



Assumption

✓ 100% of Hcl will be removed

✓ 95% of H_2o will be removed

In lab scale 50g of bagasse 6g of activated carbon were produced i.e $6/50*100 = 12\%$ of yield.

$m16 = 0.12m1 + 0.12m_{H_2o}$

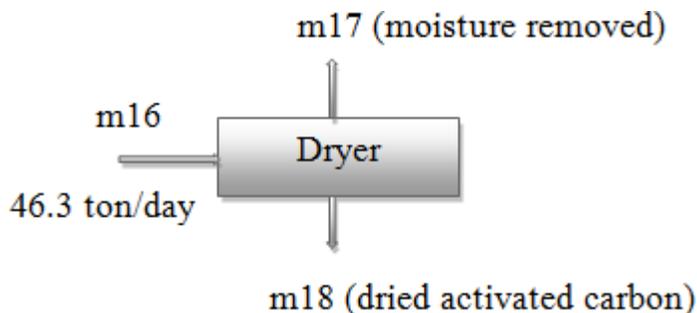
$= 0.12*227.5 + 0.05*380$

$m16 = 46.3 \text{ ton/day}$

From this $m_{14} = m_{15} + m_{16}$

$m_{15} = m_{14} - m_{16} = 521.2 \text{ ton/day} - 46.3 \text{ ton/day} = 475 \text{ ton/day}$ of wastes.

I. Dryer2



Assumption

- ✓ 100% of moisture will be removed
- ✓ In lab. Scale, from 50g of bagasse, 6g of activated carbon was produced (12% yields).

Thus, $m_{18} = 12\%$ of $m_1 = 0.12 * 227.5 \text{ ton/day}$

$m_{18} = 45.5 \text{ ton/day}$

$m_{16} + m_{18}$, $m_{17} = m_{16} - m_{18}$

$m_{17} = 46.3 - 45.5 = 0.8 \text{ ton/day}$

Therefore 45.5 ton/day of activated carbon is produced from 227.5 ton/day of sugar cane bagasse.

Table 5.1. Material balanceresult

Unit operation	Input(ton/day)	Output(ton/day)
Millcrusher	227.5	193.4
Soaking tank	193.4	3287.4
Filtration	3287.4	357.8
Dryer1	357.8	212.7
Carbonization unit	212.7	102.6
Cooling	102.6	102.6
Washing tank	102.6	521.2
Filtration unit	521.2	46.3
Dryer2	46.3	45.5

5 Conclusions

Based on the proximate analysis result, sugar cane bagasse has maximum volatile matter and minimum ash content. Therefore, the analysis gives an evidence for sugar cane bagasse as the precursor use for activated carbon. To determine the optimum temperature for maximum quantity of activated carbon, the sugar cane bagasse was activated at 400, 500 and 600 °C. As a result, the high percentage yield of activated carbon is recorded at 400 °C carbonization temperature. But, this does not mean that the adsorption performance of the activated carbon is the temperature at which the maximum yield is produced; this is due to the fact that low yield could also produce highly active pores. Therefore, the adsorption capacity of the activated carbon should be considered as a criterion for determining the optimum temperature for activation [9, 10].

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