Processing and Characterization of Activated Carbon from Toddy Fruit Husk

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Abstract

The focus of this research work was the processing and characterization of activated carbon from toddy palm fruit husk. To identify the purity of raw material, the properties of toddy palm fruit husk in terms of moisture, ash, volatile matter, fixed carbon content and bulk density were determined. Activated carbon was prepared by chemical activation of toddy palm fruit husk and then by carbonization. The effect of carbonization temperature and time on the appearance and properties of processed activated carbons were investigated. The physico-chemical properties of processed activated carbon such as moisture, ash, volatile matter and fixed carbon content were determined. The highest fixed carbon content was obtained with 25 % w/w zinc chloride as chemical activating agent at carbonization temperature 350°C for carbonization time 45 min. For the assessment of the quality of the product, the properties such as surface area, iodine sorption capacity and methylene blue number were also investigated. Furthermore, the phase and microstructure of the prepared activated carbons were determined by X-Ray Diffraction Spectroscopy (XRD) and Scanning Electron Microscopy (SEM). The high iodine sorption capacity and fairly high methylene blue number indicated that the processed activated carbon had large surface area and well developed mesoporosity. Therefore toddy palm fruit was the potential to be a promising precursor for the processing of activated carbon.

Keywords: activated carbon, toddy fruit husk, carbonization, activation, fixed carbon content

1. Introduction

Global demand for activated carbon is increasing and there is a strong need for finding a new starting material for the preparation of activated carbon, which should be cost effective. A variety of raw materials such as coal, coconut shell, wood, almond shell, rice husk banana empty fruit bunch, groundnut shell, corn cob, and agricultural waste were used for the production of activated carbon. Researchers are great interest in exploring new precursors depending on the availability and suitability for activated carbon [1].

Activated carbons are made from carbonaceous materials by activation and carbonization process. In physical activation, the precursor is first pyrolysed at high temperature and then activated using carbon dioxide or oxygen. In chemical activation, the starting material is impregnated with a chemical activating agent such as zinc chloride, calcium chloride, phosphoric acid and potassium hydroxide and then carbonized at 300-700°C [2]. The lower activation temperature and shorter activating time are the advantages of chemical activation. The activated carbon with mesoporous and larger surface area is obtained by chemical activation [3].

Activated carbon is applied widely in a variety of fields such as food industries, wastewater treatment, chemical industries and metallurgy [4]. The adsorption capacity of activated carbon depends on the nature of starting material and the manufacturing process. It is influenced by activating temperature, activating time and activating agent [3].

Toddy palm or Palmyra palm (*Borassus flabellifer* L.) is a native of tropical region in Myanmar. Toddy palm fruit pulp is consumed fresh, whereas the fruit fibers are waste by-product. [5]. The female tree can produce the fruits and the outer covering is smooth, thin, leathery, and brown. [6]. The fruits contain cellulosic semisolid flesh, which is armored by fibers. The toddy fruit fibers are inexpensive, abundantly available and eco-friendly, and hence it is essential to explore the potential utility of these fibers to the technical world [7].

The main objective of this study was to investigate the potential of toddy palm fruit husk as a raw material for the preparation of activated carbon and to study the effect of carbonization parameters on the properties of prepared activated carbon.

2. Materials and Methods

2.1. Preparation and Characterization of Toddy Palm Fruit Husk

Toddy fruit husks were collected from Patheingyi Township, Mandalay Region. The collected toddy palm fruit husks were cut into small pieces (2.5 cm in length and 1 cm in thickness). They were washed with water to remove foreign matter and then dried by sun drying at 32 to 35°C for 72 hr. The dried toddy palm fruit husks were put into a suitable plastic container for further work.

The physico-chemical characteristics such as moisture, ash, volatile matter, fixed carbon content and bulk density of toddy palm fruit husk were determined by ASTM standard test methods for activated carbon, 2000.

2.2 Processing of Activated Carbon from Toddy Palm Fruit Husk

Chemical Activation

(10) g of cleaned and dried toddy palm fruit husk was impregnated in 20 mL of 25 % $ZnCl_2$ solution and then soaked overnight at room temperature. Then the sample was sieved and put into the crucible and dried in a hot air oven at 105˚C for 30 min before pyrolyzed in a muffle furnace (HYSC, MF-05, Korea).

Carbonization

(10) g of chemically treated toddy palm fruit husk was placed in the porcelain crucible and covered. It was carbonized in a muffle furnace at 400˚C for 60 min carbonization period. After carbonization, activated carbon was cooled down and taken out carefully from muffle furnace. Then, it was washed with water for three times to remove trace chemicals. After washing, it was kept on tray for draining water and transferred into an oven at 105˚C for an hour. The activated carbons were stored in screw capped bottle at room temperature for further studies.

Effect of Carbonization Temperature

The effect of carbonization temperature on the quality of prepared activated carbons from toddy palm fruit husk was determined using the various temperatures ranging from 250ºC to 450ºC for 60 min.

Effect of Carbonization Time

In the carbonization of toddy palm fruit husk, the effect of carbonization time on the quality of prepared activated carbons was determined using the temperature 350ºC for various time ranging from 30 min to 90 min.

2.3 Characterization of Processed Activated Carbon

The physico-chemical properties of processed activated carbons from toddy palm fruit husk were determined using the same procedures described in Section (2.1).

Determination of pH

(1) g of processed activated carbon was added into the Erlenmeyer flask. After that 100 mL of distilled water was poured and it was gently boiled for 5 min. Then the solution was diluted to 200 mL, cooled to room temperature and pH of activated carbon was measured by using pH meter (JENWAY, 3510).

Determination of Surface Area

The specific surface area of activated carbon was determined as follows: 1.5g of activated carbon powder was mixed with 100ml of dilute hydrochloric acid at pH 3 and thoroughly agitated. After that, 30g of sodium chloride was added into the mixture and continuous agitation was kept constant. Deionised water was then added to make the volume up to 150 ml. The solution was titrated with 0.1N NaOH until pH was from 4 to 9 and volume V was recorded. The surface area according to this method was calculated as [1]:

$$S = 32V - 25 \quad (1)$$

Where *S* is surface area of activated carbon and *V* is volume of sodium hydroxide required to raise the pH from 4 to 9.

Determination of Iodine Number

1 g of activated carbon was thoroughly mixed with 10 ml of 5% HCl in the 250 ml flask. 100 ml of iodine solution was then added into it. Then mixture was made sure to be mixed thoroughly and filtered. After filtering, 50 mL of filtrate was taken and titrated with 0.1 M sodium thiosulphate until

changing pale yellow color. 1 ml of 1% starch indicator solution was then used to record the end point by disappearing pale yellow color. The blank titration without activated carbon was also carried out as the same procedure. The percent of iodine adsorbed by carbon was calculated by following equation [4].

$$Iodine\ number\ = \frac{V_{sb} - V_{ss}}{V_{sb}}\ x\ 100 \qquad (2)$$

where, *Vsb* is sodium thiosulphate used for blank (mL) and *Vss* is sodium thiosulphate used for sample (mL).

Determination of Methylene Blue Number

Methylene blue was measured by soaking the sample (10 mg) with different concentrations (10, 25, 50, 100, 250, 500 and 1000 mg/L) of methylene blue solution for 24 hr at room temperature. After 24 hr, the solution was analyzed by UV/Vis spectrophotometer (UV-160A, Shimadzu, Japan) at 645 nm wavelength. Amount of methylene blue adsorbed in each solution was calculated as follows [4].

$$Methylene\ blue\ number\ = \frac{(C_o - C_e)V}{M} \qquad (3)$$

where, *Co* and *Ce* (mg/L) are the concentrations of methylene blue at starting and equilibrium time, *V* is the volume of solution (L) and *M* is the mass of activated carbon used (g).

Determination of Microstructure

The microstructure of processed activated carbons was examined by Scanning Electron Microscopy (SEM) (JSM-5610).

Determination of Phases

The phases of processed activated carbons were analyzed by X-Ray Diffraction Spectroscopy (XRD), (MDI/JADE6).

3. Results and Discussion

The characteristics of waste materials such as toddy palm fruit husk were determined and shown in Table (1). The toddy fruit husk having low bulk density 3.3 g/cm^3 was found to be the valuable raw materials for the production of highly porous activated carbon. Other important characteristic was their high carbon content 37.15%. The effects of carbonization temperature and time on the appearance and properties of processed activated carbons are shown in Tables (2) to (5) respectively. The results in Tables (2) and (4) show that increase the carbonization temperature and time gave the ash occurred on the outer surface of carbonized toddy palm fruit husk. And then, lower carbonization temperature and time gave the incomplete carbonization of toddy palm fruit husk. Therefore, 350ºC of carbonization temperature and 45 min of carbonization time indicated that the bright black colour and good appearance of toddy palm fruit husk activated carbon.

Table 1. Physico chemical properties of Toddy Palm Hush

Sr. No.	Properties	Toddy Palm Fruit Husk
1	Moisture (%w/w)	19.42
2	Ash (%w/w)	6.29
3	Volatile matter (%w/w)	37.14
4	Fixed carbon (%w/w)	37.15
5	Bulk density, (kg/m^3)	3.3

From the results in Tables (3) and (5), it was found that all the processed activated carbons furnished a good percentage of fixed carbon. The fixed carbon content was increased with increase in carbonization time to certain value and then decreased.

Table 2. Appearance of Processed Activated Carbons with Different Carbonization Temperatures

Sample No.	Carbonization Temp. (°C)	Carbonization Time (min)	Appearance of Activated Carbons
1	250	60	Black, outer surface soft and interior hard
2	300	60	Black, outer surface soft and interior hard
3	350*	60	Bright black, porous surface
4	400	60	Soft, porous surface covered with ash layer
5	450	60	Soft, porous surface covered with ash layer

* *the most suitable temperature*

Table 3. Effect of Carbonization Temperature on the Properties of Processed Activated Carbons

Carbonization time = 60 min

C.T (˚C)	Properties of Activated Carbon: Moisture (%w/w)	Ash (%w/w)	Volatile Matter (%w/w)	Fixed Carbon (%w/w)	Yield (%w/w)
250	6.20	19.98	15.37	58.45	54.2
300	6.09	17.09	15.68	61.14	55.1
350*	5.96	15.42	14.34	64.28	69.8
400	5.95	16.64	13.53	63.88	64.8
450	5..95	16.85	13.30	63.90	61.5

* *the most suitable carbonization temperature* (C.T)

The highest fixed carbon content, 66.40 % w/w and the highest yield percent, 69.88 % w/w were obtained at carbonization temperature 350˚C for 45 min. Therefore, the carbonization temperature 350˚C for carbonization time 45 min were chosen as the most suitable condition for carbonization of toddy palm fruit husk.

Table 4. Appearance of Processed Activated Carbons with Different Carbonization Time

Sample No.	Carbonization Temp. (ºC)	Carbonization Time (min)	Appearance of Activated Carbons
1	350	30	Black, outer surface soft and interior hard
2	350	45*	Bright black, porous surface
3	350	60	Bright black, porous surface
4	350	75	Soft, porous surface covered with ash layer
5	350	90	Soft, porous surface covered with ash layer

** the most suitable time*

The physico-chemical properties of processed activated carbon at optimum conditions are shown in Table (6). From the data, it was found that processed activated carbon had less ash content than the commercial activated carbon. The less ash content attributed to lower inorganic content and higher fixed carbon content. Higher ash content is undesirable for activated carbon because it affects adsorptive capacity. The moisture content of activated carbon was slightly higher than commercial carbon, but the moisture content of carbon has no effect on its adsorptive power. The carbons of pH range 6 to 8 are useful for most applications [8]. The pH of processed activated carbon from toddy palm fruit husk was agreed with literature.

Table 5. Effect of Carbonization Time on Properties of Processed Activated Carbons

Carbonization temperature = 350˚C

C.T (min)	Moisture (%w/w)	Ash (%w/w)	Volatile Matter (%w/w)	Fixed Carbon (%w/w)	Yield (%w/w)
30	6.09	14.35	19.75	59.81	54.64
45*	5.49	14.11	14.00	66.40	69.88
60	5.96	15.42	14.34	64.28	69.80
75	5.95	16.03	14.03	63.99	68.96
90	5.95	16.05	14.18	63.82	68.50

** the most suitable carbonization time* (C.T)

The performance of activated carbon was commonly examined by iodine number and methylene blue number. For the estimation of surface area of activated carbon, the iodine number and methylene blue number are important, since the carbon possess different pore sizes. It was also observed that the prepared activated carbon which had more iodine value than the literature, which indicated that it capacity of carbon to adsorb smaller molecules. The higher iodine number of these carbons is due to the presence of large micropore structure which attributed to chemisorption taking place in the pores of the carbons during activation.

The surface morphology of processed activated carbon and commercial activated carbon were studied using SEM and their photographs are shown in Fig. (1). The commercial activated carbon showed aggregates with rough surface. The processed activated carbon form toddy palm fruit husk had rod like structures with many holes on carbon surfaces and open porous structure. The surface morphology of processed activated carbon revealed cavities, pores and rough surface. It was verified that the prepared zinc chloride impregnated activated carbons occupied better possibilities for adsorption onto the surface of the pores.

Table 6. Physico-chemical Properties of Processed Activated Carbon

Carbonization temp. and time - 350˚C for 45 min

Properties	Processed AC	Standard AC*
Moisture (%w/w)	5.49	5.23
Ash (%w/w)	14.11	15.34
Volatile matter (%w/w)	14.00	18.33
Fixed carbon (%w/w)	66.40	61.10
Bulk density (%w/v)	0.2	0.34
pH	6.7	7.4
Surface area, (m^2/g)	432	401.42
Iodine number, (%w/w)	79.1	75.95
Methylene blue number, (mg/g)	224	232
Yield (%w/w)	69.88	-

*AC= Activated Carbon, ** [3]

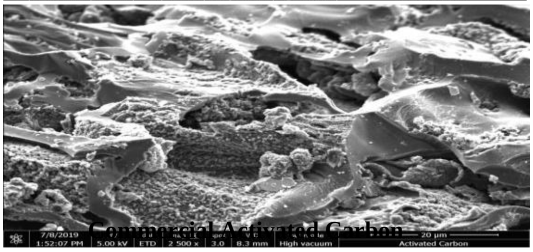

Figure 1. Microstructure of Activated carbon from Toddy Palm Fruit Husk

The nature of processed activated carbon and commercial activated carbon were analyzed by X-ray diffraction and the results are shown in Figure (2). X-ray diffraction analysis demonstrated that both processed activated carbon and commercial activated carbon possess graphitic character and low content of inorganic constituents.

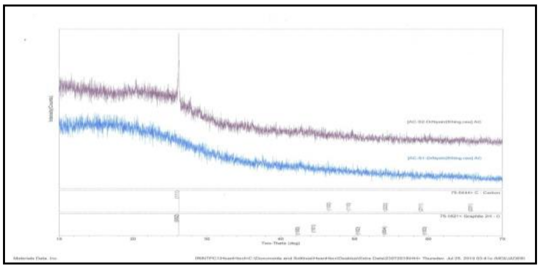

Figure 2. XRD Patterns of Commercial Activated Carbon and Processed Activated Carbon

4. Conclusion

In this research work, the activated carbon was prepared from chemical treated toddy palm fruit husk by carbonization method. The most suitable parameters for full carbonization of toddy palm fruit husk are found to be 350ºC for 45 min. At temperature lower and shorter time than the above condition, there is an inefficient carbonization due to incomplete removal of volatile products resulting in pore blockade and less adsorptive capacity. And also at temperature higher than 350º C and longer time, the carbonized products are generally reduced in activity due to excessive or post-carbonization process. The toddy palm fruit husk, lignocellulosic waste can be converted to a value- added product. Processing toddy palm fruit husk activated carbon is a way of making use of waste materials, thus reducing disposal and environmental polluted problem.

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