International Journal of Advanced Educational Research
ISSN: 2455-6157; Impact Factor: RJIF 5.12
Received: 06-06-2019; Accepted: 09-07-2019
www.educationjournal.org
Volume 4; Issue 5; September 2019; Page No. 40-43

Making and characterization of active carbon from coconut shell charcoal

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Abstract

This study aims to determine the norit structure (Activated carbon) of coconut shell charcoal test results using Fourier Transform Infrared (FTIR) analysis, X-Ray Diffraction (XRD) and thermal reactions through analysis of Differential Scanning Calorimeter (DSC) and get norit (carbon active) from coconut shell charcoal with high purity X-Ray Fluorescence (XRF) analysis which can be applied as in the pharmaceutical field. The research method consists of 3 stages: (1) preparation of coconut shell charcoal, (2) synthesis of activated carbon, and (3) testing of activated carbon products. The results of FTIR analysis showed a decrease in impurity elements, XRD analysis showed semi crystalline structural activated carbon, DSC analysis showed thermal reaction with exothermic peaks at a temperature of 112.44 C, and XRF analysis showed impurities namely Cl, K, Ca, Px, and Fe.

Keywords: coconut shell charcoal, activated carbon, FTIR, XRD, XRF, DSC

Introduction

In Indonesia, especially the centers of coconuts, coconut shells, coconut husks and pulp are the by-products of copra and oil processing. The average coconut shell component is around 15% of whole coconut fruit. The chemical composition of coconut shell contains water, lignin, cellulose, and hemi-cellulose. In industry, coconut shells are not directly used, first processed into charcoal or powder. Charcoal powder is obtained by grinding. Charcoal powder is widely used in the chemical industry as a solution for insecticide thinners, and also functions as a filler in the plywood, asbestos and insect repellent industries (Jamaran, 2010) [12].

Coconut shell is the main ingredient in making charcoal and activated charcoal. Activated charcoal is widely used as a liquid adsorbent in the sugar industry, cooking oil industry, soft drinks and alcohol. In the chemical industry it is used to make acetone, methanol, phenol, and cresol. In the pharmaceutical field it is used as a bleaching material and ingredients for making norits (Josep and Layuk, 2010) [15].

If activated carbon is examined under Scanning Electron Microscopy (SEM), very large amounts of pores will be seen. These pores have various sizes. Pores of more than 50 nm are called macropores, 2 nm - 50 nm mesopores, and below 2 nm micropores. If we count, then 1 gram of activated carbon has a pore surface area of 0.5 to 1.5 square kilometers (Marsh and Rodrigues-Reinoso, 2005) [18].

These very wide pores are capable of capturing a variety of materials, including toxic materials, mainly through van der Walls (Marsh and Rodrigues-Reinoso, 2005) [18]. Therefore activated carbon can be used in cases of drug overdose, food poisoning, or ingestion of toxic substances. However, its ability to capture poisons only occurs in the stomach and intestines, when toxic substances have not been absorbed and enter the bloodstream. So that, the faster it is given, the more poisons that can be absorbed.

Activated carbon has been used as a catalyst carrier for several years. The most ancient use is as a bearer of precious metal catalysts, namely for organic reactions on a laboratory scale, because activated carbon is inert then the use as a catalyst can provide high selectivity (Marsh and Rodrigues-Reinoso, 2005) [18]. The main role of activated carbon is to facilitate the formation of the stability of the dispersed active phases. The use of activated carbon as a carrier of metal catalysts because it has a large surface area and low chemical activity (Fraga *et al*., 2002; Rampe *et al*, 2012) [9, 20].

In this study coconut shell was used as raw material in the process of synthesis of norit (activated carbon). To obtain carbon from coconut shell charcoal, the impurities in the form of tar and evaporating material are removed (Rampe *et al*., 2014) [22]. To remove impurities it is done by heating at a temperature of 500 oC to 1000 oC, and to help eliminate unwanted compounds carried out by steam or gas drainage. Because during the heating process, all material that is not carbon must be removed to obtain purer carbon (Gupta *et al*., 2005; Elsayed *et al*., 2007; Wiratmoko and Halloran, 2009; Rampe *et al*, 2013) [11, 7, 29, 22].

The treatment of heating temperature and duration of reaction (duration of detention) in the synthesis process of activated carbon is intended to determine the relationship of microstructure, chemical composition, physical properties of norit produced. Data obtained in the form of crystallographic systems (crystal structure) through X-ray Diffarction (XRD) technique, spectrum that shows elemental composition through X-Ray Fluorescence (XRF) technique, functional groups by Fourier Transform Infra-Red (FTIR) analysis and to determine the reaction thermal charcoal and starch using Differential Thelmal Analysis (DSC) (Fernandez and Fernandez, 2008; Calister, 2007; Lalena *et al*., 2008; Rampe, 2011) [8, 17, 21].

Research Methods

Material

The raw material used in this study is charcoal from coconut shell pyrolysis from Tomohon, HCl p.a, amylum p.a, ethanol p.a, acetone p.a, Na2S2O3 p.a, universal indicator, Whatmann paper No.42.

Tools

The tools used in this study include agat mortar, 100 mesh sieve, gravity vonvection oven model, Carbolite electric furnace model 2132 (Max Temperature 1200°C, thermometer, clamp, Fourier Transform Infrared (FTIR) for fu group analysis, Differential Scanning Calorimeter (DSC) Perkin Elmer's model for studying thermal reactions, X-Ray Fluorescence (XRF) models of Thermo Fisher Scientific analysis of material element types, X-Ray Diffraction (XRD) Goniometer type for identification of crystalline structures / molecular structures of material qualitatively.

Carbon preparation

The coconut shell pyrolysis process is carried out using a pyrolysis reactor which is designed by itself. Coconut shell is the main raw material for the research sample. The sampling technique is random.

The coconut shell charcoal from the results of the pyrolysis is cleaned from coir charcoal which is attached using a knife, then smaller pieces are made by mashing using porcelain mortar. Subtle pieces of shell charcoal are then mashed into powder and sifted with a 100 mesh sieve to homogenize particle size (Lalena *et al*., 2008) [17]. Carbon powder obtained with particle size passed the 100 mesh sieve.

Furthermore, the charcoal powder passes the 100 mesh sieve, purification from inorganic minerals such as: Mg, Al, K, Ca, and Fe. Where the charcoal powder is soaked for 24 hours with 3 M HCl at room temperature. Then the charcoal is washed with distilled water until the washing water shows a constant pH, then it is dried in an oven at a temperature of 110 oC overnight (Rampe, 2011) [20].

Furthermore, the charcoal powder is put into a tube furnace calcination reactor. Calcined charcoal at a temperature of 300oC for 3 hours was calculated when the temperature was reached, with gas N2 (Anirudhan *et al*., 2009; Concheso *et al*., 2009).

Activated carbon synthesis

Synthesis of activated carbon was carried out by the solvent method, as much as 5% mass (5 grams) starch with coconut shell powder washing results dissolved in distilled water (10 mL) at 100 oC, stirred for 60 minutes until completely hydrolyzed (Billmeyer, 2000) [3]. The starch solution system is mixed with charcoal powder, by mixing until the mixture is homogeneous, then dried at room temperature for one night.

Furthermore, the results of the drying are printed using cylindrical molds with a diameter of ~ 15 mm. Compaction is done by pressing in one direction with a compressive force of 5 tons. This process produces a sample in the form of pellets (green compact). The samples obtained in this way were then dried at room temperature for 24 hours, and continued with the drying process in an oven at 110 oC for 4 hours. Samples obtained in this way are then put into the furnace for sintering. Samples undergo a sintering process at a temperature of 300 oC and a holding time of 3 hours in a Carbolite furnace, cooling in annealing (Ebner *et al*., 2004; Jia-Yuan *et al*., 2008; Ebner *et al*., 2004; Rampe, 2011) [6, 6, 20].

Active carbon analysis

1. Fourier TransformInfrared Analysis (FTIR)

Analysis of the structure of the norit synthesis sample using the Fourier Tranform Infra-Red (FTIR) Shimadzu IR-Prestige-21 model for this purpose was made KBr pellets, wave numbers (v) 500 to 4000 cm-1; used to determine the functional group of norit carbon material (Miyazaki *et al*., 2005; Sikalidis *et al*., 2006; Rampe *et al*., 2012) [19, 27, 21].

Table 4.1: Coconut shell charcoal Infrared absorption bands

Wave number, cm^{-1}		**Functional groups**
Coconut shell charcoal	**Activated Carbon**	
748,39 m	756,12 m	
810,10 m	817,84 m	
864,12 m		NO_3 vibration and O-N-O vibration
1265,3 w	1226,75 w	C-O vibration
1373,34 w	1381,05 w	Phenol
1427,32 w	1427,34 w	
1581,65 s	1581,65 s	Aromatic structures (aromatic groups), vibrations of Carbonil C=O
1851,68 w		
2337,72 m	2337,74 w	
	2368,60 w	
		vibrasi ν.as$CH_{2,\ \nu.s}CH_2$& CH_3alifatik
2931,82 w	2924,11 w	
3425,60 m	3425,60 w	O-H
3757,35 w		
3873,08w	3803,65 w	

Description: vs = very strong, s = strong, m = medium, w = weak

Infrared spectrum data from coconut shell charcoal from pyrolysis and norit products activated carbon shows several missing functional groups. It can be concluded that the activation process can remove impurity groups on the surface of activated carbon material.

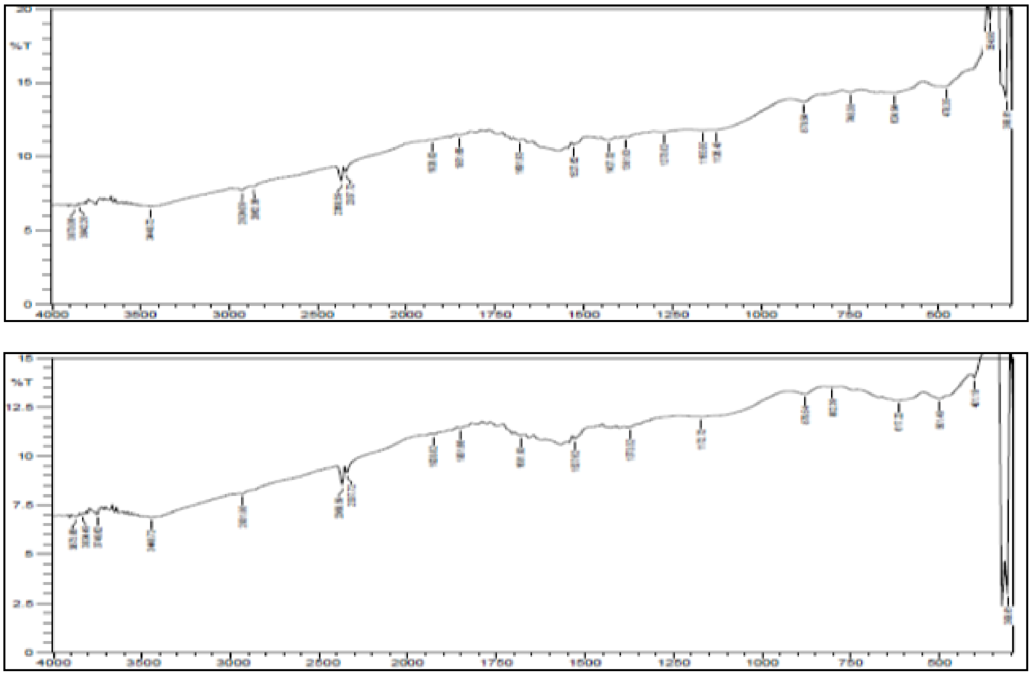

Fig 4.1: FTIR spectrum: (A) coconut shell charcoal, and (B) activated carbon.

2. Analysis *X-ray Diffraction* (XRD)

The diffractogram of activated carbon is presented and the recapitulation of data on changes in basal spasing is shown in Table 4.2. Qualitative analysis of X-ray diffraction data is carried out by further processing of data generated using the PCPDFWIN data base (PDF, Powder Diffraction File) issued by JCPDS (Joint Committee on Powder Diffraction Standard). Normal graphite (Graphite-2H) with PDF: 41-1487 with hexagonal crystal structures having 2 mempunyai of 26.38 and spacing between fields, d of 3.38 Ǻ (002) used as a standard for studying diffractogram patterns and X-ray diffraction data from carbon material. Determination of carbon crystal structures based on diffraction patterns is indexing patterns based on the angular position of the diffraction line.

Table 4.2: Value of activated carbon and dan grafit normal (literatur)

Nilai d, (Ǻ)	
Activated carbon	**Graphite normal***
4.506	3.38
4.241	2.04
3.364	1.68

Difraktogram norit (activated carbon) shows the characteristic reflection peaks which show that the atoms have a pattern that can be repetitive. The diffractogram of the norit product shows characteristic reflection peaks which indicate a turbostatic structure of carbon, which is at d_{002} = 4,506 Ǻ; d_{002} = 4.241 A and d_{002} = 3.364 Ǻ. In general graphite shows the distance d002 in the range of 3.38 38 and 2.04 4 which corresponds to the angle 2 sudut between 26.38o and 44.39o. Reflection intensity at $2\theta = 26.47^{o}$ gives a relative intensity of 45% and reflection d002 (3.36 Ǻ) according to the XRD pattern standards based on JCPDS data (PDF 41-1487) showing that the atoms begin to be arranged according to a pattern that is repeated in 2 dimensions. The results of this study as reported by Anirudhan *et al*. (2009) state that the pattern of peak width is centered at 2θ of 26^{o} with respect to the 002 graphite plane, as the peak characteristic of activated carbon.

4. X-Ray Fluorensence Analysis (XRF)

Data on the content of the elements of activated carbon are determined based on the results of the analysis using XRF. The data presented in Table 3 shows impurity elements namely chloride, Cl of 52.27% of mass and potassium, K of 37.80% of mass, Ca of 5.83% of mass, Fe of 1.23% of mass. The process of activation and extraction with HCl can increase the purity level of coconut shell charcoal. The results of this analysis are indicated by the results of FTIR analysis even with diffraction patterns of amorphous charcoal structures.

Table 3: Chemical composition of activated carbon

Compound	% m/m	Elemen	% m/m (1)	% m/m (2)
Cl	47,36	Cl	47,36	52,27
K2O	38,91	K	32,30	37,80
CaO	6,72	Ca	4,81	5,83
P2O5	4,81	Px	2,14	2,32
Fe2O3	1,45	Fe	1,02	1,23

4. Differential scanning Calorimetty (DSC) analysis

The standard differential Scanning Calorimetry (DSC) curve from the literature is used as a reference to discuss the coconut shell DSC curve. Differential Scanning Calorimetry (DSC) is based on changes in energy (ΔH).

The carbon activated DSC curve (activated carbon) is presented in Figure 4.5. The DSC curve in the temperature range of 30 to 428 ^{o}C, obtained by one peak exothermic reaction is shown in Figure 8. The curve shows an exothermic reaction at a peak temperature of 112.44 ^{o}C with a peak intensity of -17.9604 mW. The graph shows the net at 63.65 ^{o}C and ends at 162.96 ^{o}C, with an area of -3839.931 mJ and the reaction enthalpy of H = -191.9966 Jg^{-1} This exothermic peak is caused by a dehydroxylation reaction in the form of molecular loss water absorbed in the grille of the coconut shell norit product. Then there is a decrease in the endothermic reaction.

Conclusion

The results of FTIR analysis show that norit (activated carbon) of coconut shell has a functional group active as graphite and XRD shows a pattern of semi-graphite carbon material. The results of XRF analysis show that there are still impurities in norit products (activated carbon), namely Cl, K, Ca, Px, and Fe.

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