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Of Coconut Shellwaste Pyrolysis Poten as a Carbon Material From Minahasa Charcoal Cy, Indonesia

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Abstract

Coconut shell charcoal can be obtained through the pyrolysis of coconut shell. Coconut shell comes from Minahasa, North Sulawesi, Indonesia. Activation by heating at a temperature of 750ºC and immersion in HCl done for purification of charcoal as a base material of carbon. Charcoal's characters as carbon material were analyzed through Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM). The analysis showed after the activated charcoal absorption band is polar with -OH groups/cluster, C = C, C = O and C-O; particle distribution nearly similar/uniform, smooth and visible presence of pores on the surface of charcoal, and is semi crystal with a hexagonal crystal structure.

Keywords: coconut shell charcoal, FTIR, XRD, SEM

INTRODUCTION

Indonesia is a rich country in agricultural and plantation products, oil is one of it. Coconut (Cocos nucifera, L) is one of the plants belonging to Palmae family, where all parts of this plant, from the roots to the leaves and fruit of coconut are beneficial.

The shell are not optimally utilized, just piled, burned or abandoned because most farmers consider coconut shell as a waste with a minimum utilization and has a low mercantile value.

Coconut shell categorized as hardwood that has higher levels of lignin whereas cellulose content is lower. Coconut shell is hard because of the high silicate (SiO2) and high carbon bound content, whereas low mineral ash content. The main composition of coconut shell consists of cellulose, lignin, hemicellulose containing atoms of C, O, H, and N. These organic materials containing functional groups such as hydroxyl (R-OH), alkanes (R- (CH2) n- R '), carboxyl (R-COOH), carbonyl (R-CO-R'), ester (R-CO-O-R '), group linear ether and cyclic (RO-R') with varying amounts (Rampe et al., 2011). Charcoal is the main product produced through pyrolysis process that creates by-product in the form of liquid smoke and tar. Charcoal consists of several components which are carbon bonded, ash, water, nitrogen, and sulfur. Most of charcoal pores still covered with hydrocarbons, tar and other organic compounds (Rampe et al, 2014). Charcoal pyrolysis is very potential to be process into a carbon material such as activated carbon, molecular filter, briquettes, electrode, and others. Activation is a charcoal treatment aimed to enlarging the pore by breaking the bonds of hydrocarbons or oxidizing surface molecules that change the nature of charcoal, either in physics or chemical. The process of physics activation is done by heating at high temperature in a closed system without air while flowing inert gas. The characterization charcoal of coconut shell pyrolysis performed, in connection with the optimization on utilizing coconut shell that are only considered as a waste and the increasing needs of the use of carbon materials. Coconut shell used comes from Minahasa, North Sulawesi, Indonesia.

In this study, coconut shell used as raw material in the synthesis process of carbon material, polluters in the form of tar and evaporate material removed to obtain carbon from coconut shell charcoal. Eliminating polluters conducted by heating at a temperature of 500oC up to 1000oC, then steam or gas flow process helps to eliminate unwanted compounds. Because during the heating process, all non-carbon material should be removed to obtain pure carbon, at the same time arranging its structure (Wiratmoko and Halloran, 2009; Elsayed et al., 2007; Gupta et al., 2005). In order to eliminate existing metals done by soaking in HCl solution, then washing and drying (Fraga et al., 2002). Charcoal of the coconut shell pyrolysis were analyzed through Fourier Transform Infra Red (FTIR) to determine the functional groups, Scanning Electron Microscopy (SEM) to determine the morphology of the surface structure, and X-Ray Diffaction (XRD) to determine the crystal structure of carbon materials.

RESEARCH METHOD

A. Materials

The raw materials used are the charcoal of coconut shell pyrolysis from Minahasa, North Sulawesi, Indonesia; HCl (p.a merck), a universal indicator, Whatmann paper no.42, Nitrogen gas.

B. Tools

Tools used comprise of a glasses that commonly used in laboratory, pyrolysis reactor, mortar agat, sieve 100 mesh (USA Standard Testing Sieve), oven models gravity convection, Scale AND GR-200, an electric furnace Carbolite models 2132 (Max Temperature 1200oC), tube Furnace-Thermolyne (Sybron) Type 21100, thermometers, magnetic clamps, hot plate (stir and heat), Scanning Electron Microscopy (SEM) JEOL JSM-6360LA for testing the surface structure in the form of micro-structure, Fourier Transform Infra Red (FTIR) Shimadzu models of IR-Prestige-21 to determine the functional group, X-Ray Diffraction (XRD) Goniometer type to identify the crystal or molecular structure of the material in qualitative, Fourier Transform Infra Red (FTIR) Shimadzu models of IR-Prestige-21 to determine the functional group.

RESEARCH PROCEDURE

Charcoal Preparation

Coconut shell pyrolysis process is carried out using a pyrolysis reactor. Coconut shell from the inner part of the coconut is the main raw material sample. The research technique applied is random sampling. Charcoal of coconut shell pyrolysis is peeled out from fiber carbon attached using knife, then made pieces of a smaller size by grinding it using porcelain mortar. The delicate pieces of charcoal crushed into powder and sieved with a 100 mesh sieve to similar particle size (Lalena et al., 2008). Obtained carbon powder with a particle size of 100 mesh sieve passes.

Activation

Furthermore, charcoal powder passes a 100 mesh sieve inserted into the *tube furnace* calcination reactor. Charcoal calcined at a temperature of 600oC within 3 hours, measured when it reaching that temperature, while N2 gas flowing in(Anirudhan et al., 2009; Concheso et al., 2009). Charcoal calcination results purified from inorganic minerals such as Mg, Al, K, Ca, and Fe. Wherein, the charcoal powder is soaked for 24 hours with 1 M HCl at room temperature. Afterwards, the charcoal is washed with

distilled water until the swill shows a constant pH. Then, dried in an oven at 110 ° C overnight (Fraga, et al., 2002; Seok-Jin et al., 2005). Furthermore, charcoal purified carbon powder that passes a 100 mesh **sieve** inserted into the *tube furnace* calcination reactor. Carbon calcined at a temperature of 750oC within 3 hours, measured when reaching that temperature, the N2 gas flowing in (Anirudhan et al., 2009)

Carbon Material Analysis

*X-ray Diffractio*n (XRD) Analysis

Analysis of X-ray Diffraction (XRD) Goniometer type towards the powder synthesized sample conducted with X-ray source Cu / K-α1 with a wavelength, λ = 1.54056 Å. The analysis was performed with the angle 2θ ranging from 4o up to 60o. For identification of the diffraction pattern synthesized carbon materials, diffractogram resulted were compared with the data base JCPDS (Joint Committee on Powder Diffraction Standards), PDF 41-1487. (Ozaki et al., 2006).

Fourier Transform Infrared (FTIR) Analysis

Analysis of the structure of the synthesized sample using *Fourier tranform Infra Red* (FTIR) Shimadzu models of IR-Prestige-21. In this purpose made KBr pellet, the wave number (ν) 500 up to 4000 cm-1; it is used to determine the functional group of carbon materials (Sikalidis et al., 2006; Miyazaki et al., 2005).

Scanning Electron Microscopy (SEM)

Surface micro structure was analyzed using Scanning Electron Microscopy (SEM) JEOL JSM-6360LA .SEM for testing the surface topography of the surface structure in the form of micro-structure.

RESULTS AND DISCUSSION

Pyrolysis carried out in a reactor at a temperature of 350o C to get charcoal from coconut shell which is the basic carbon material. Figure 1 and 2 shows the pyrolysis reactor and charcoal of coconut shell pyrolysis. Charcoal pyrolysis were crushed and **sieved** using a 100 mesh **sieve** as shown in Figure 3.

Figure 1. Pyrolysis reactor

Figure 2. Charcoal of Coconut shell

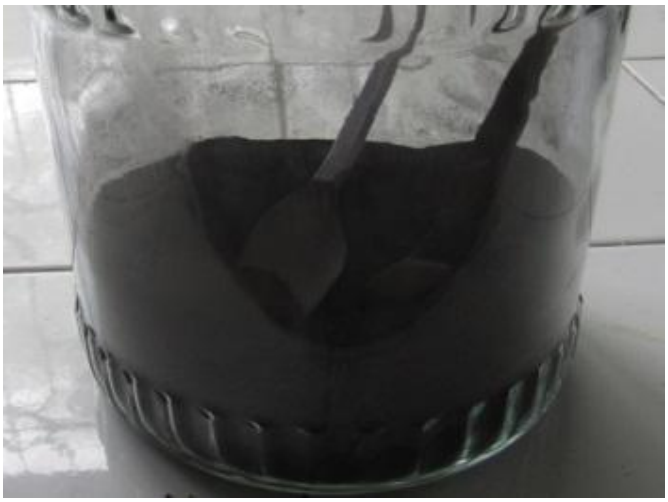


Figure 3. Charcoal Powder

FTIR Analysis

Figures 4 and 5 shows the FTIR spectra before and after charcoal activation. Furthermore, infrared absorption bands before and after the activated charcoal is given in Table 1. Numbers of wave absorption band of infrared spectra before and after the activated charcoal with the activation temperature and HCl showed an absorption band shifts. The emergence of new absorption band and the loss of long absorption band indicates that the activation process can eliminate the functional groups pollutant material on the surface of charcoal. Absorption band that shows the -OH stretching vibration, C = C, C = O and C-O indicates that the charcoal before and after activation tend to be polar. In addition, the preparation of aromatic compound shows a hexagonal structure on charcoal before and after activation.

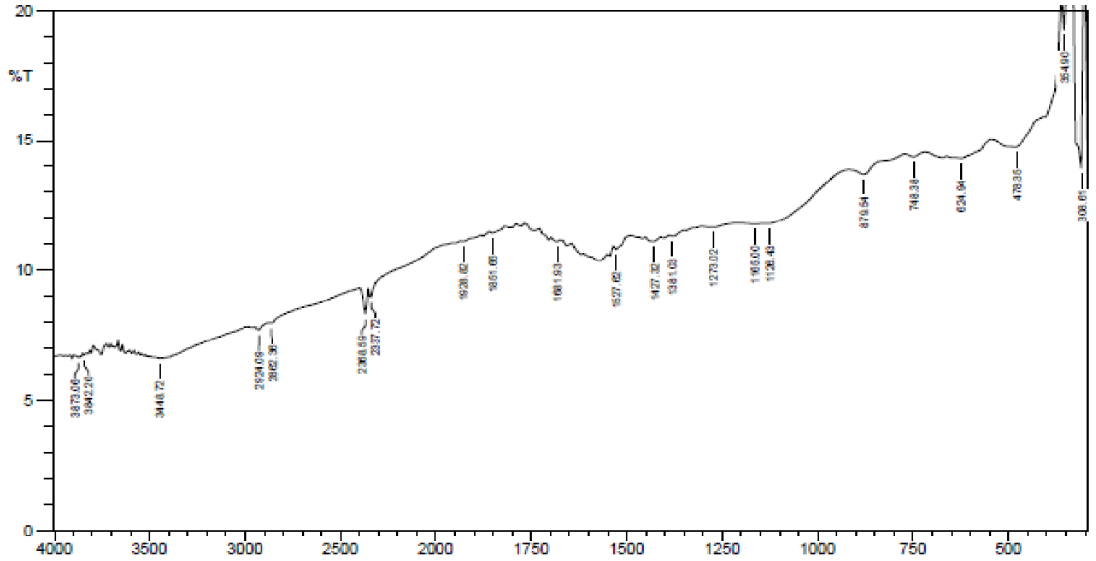


Figure 4. FTIR charcoal spectrum before activation

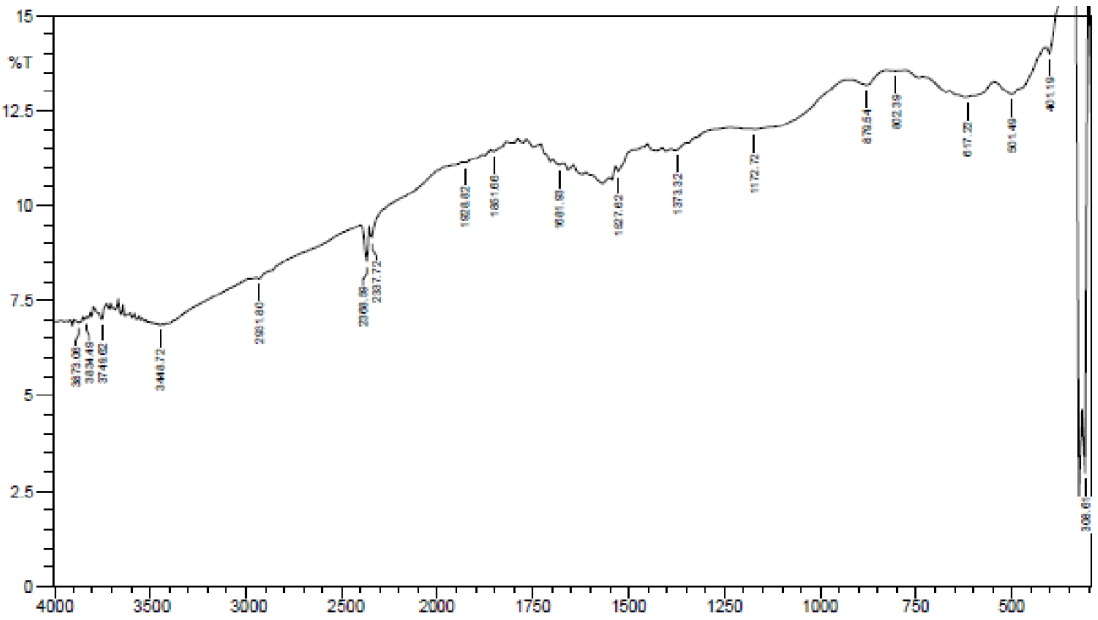


Figure 5. FTIR charcoal spectrum after activation

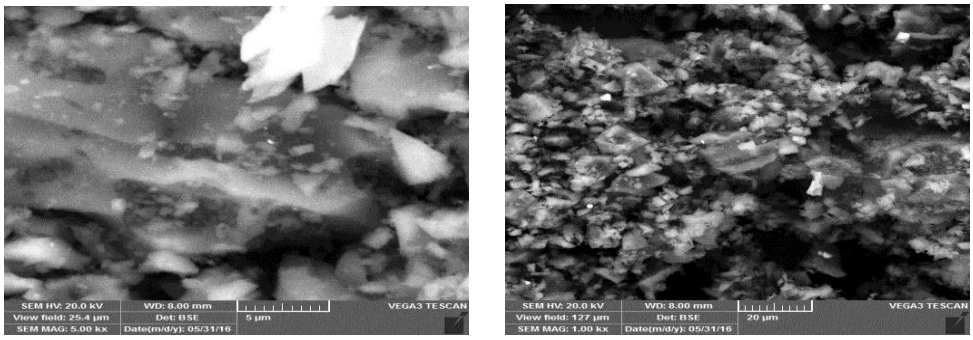
Table 1. Infrared absorption bands before and after the activated charcoal

Wave numberal cm^{-1}		**Functional Groups**
Before Charcoal Activation	**AfterCharcoal Activation**	
3448,72	3448,72	-OH
2924,09 **2862,36**	2931,80	C-H aliphatic stalling
2368,59 **2337,72** **1928,82** **1851,66**	2368,59 2337,72 1928,82 1851,66	C=O ester
1681,93 **1527,62** **1427,32**	1681,93 1527,62	C=C aromatic
1381,03 **1273,02** **1165,00** **1126,43** **879,54**	1373,32 1172,72 879,54 802,39	C-O
748,38 **624,94** **478,35**	617,22 501,49 401,19	C-H aromatic

SEM Analysis

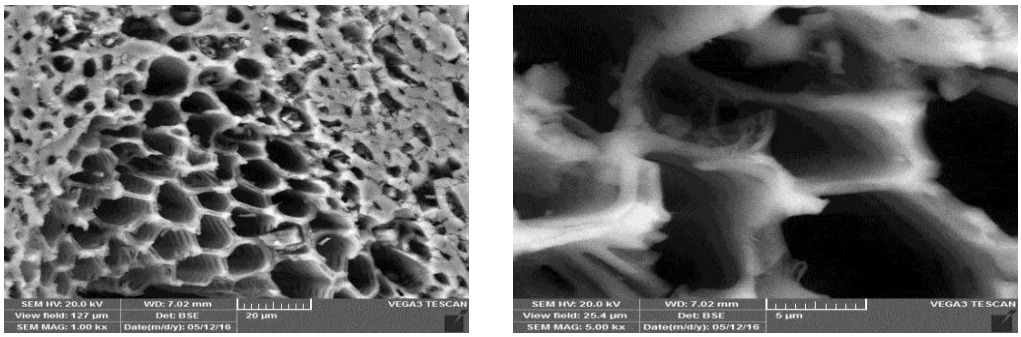
Figures 6 and 7 shows the morphology of charcoal before and after activation. Distribution of the particles were nearly similar, smooth charcoal surface and a visible presence of pores. This is because of the impurities contained on the surface of the charcoal had been lost during the activation process. Wherein, during the activation process, the activator reacts with the carbon, oxidizes and removes hydrocarbons, tar and the other compounds that attach to the surface of charcoal, causing structural

arrangement of carbon atoms and nearly similar particle distribution, charcoal surface becomes open, a new pore form.



(a) magnification of 1000x and (b) Magnification 5000x

Figure 6. Surface morphology SEM Charcoal Before Activation



(a) magnification of 1000x and (b) Magnification 5000x

Figure 7. Surface morphology SEM charcoal after activation

XRD Analysis

Figure 8 and 9 shows the diffractogram before and after charcoal activation. Diffraction basic material of coconut shell charcoal showed peaks reflection characteristics of the atoms that have a repetitive pattern that can be arranged. Diffractogram charcoal calcined showed peaks reflection characteristics leads to carbon turbostatic structure, namely on D002 = 3.85 Å; A D002 and D002 = 2.23 = 1.82 Å. Semi crystal with a hexagonal crystal structure. In general, graphite shows the distance of D002 in the range of 3.38 and 2.04 Å Å corresponding to the angle 2θ between 26,38o and 44,39o. The intensity of the reflection at 2θ = 26,38o give 100% and the relative intensity of the reflection D002 (3.38 Å) in accordance with the

standards of the XRD patterns based on data from JCPDS (PDF 41-1487) demonstrated the atoms are arranged in a pattern began repeating in two dimensions. This result is consistent as reported by Anirudhan et al., (2009) stated pattern width peak centered at 26o 2θ associated with the field of 002 graphite, as the characteristic peak of activated carbon.

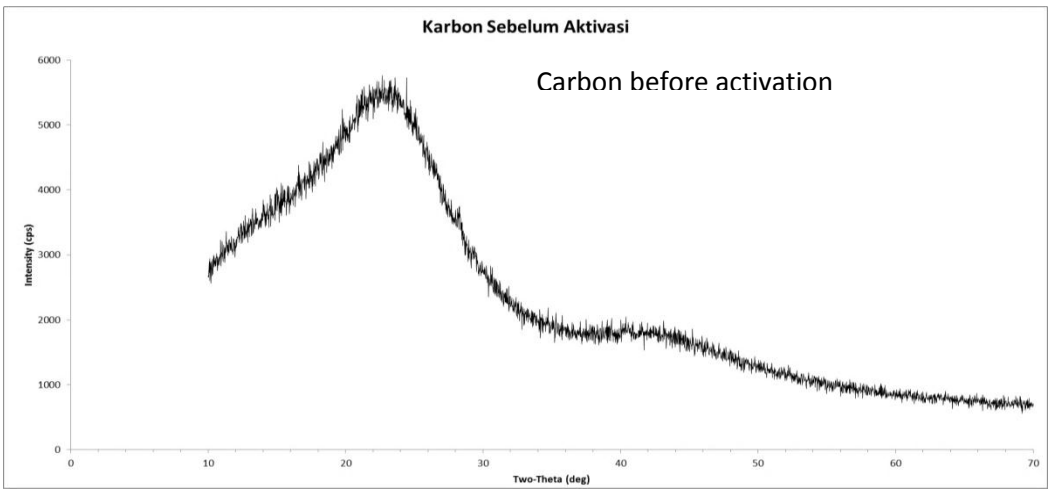


Figure 8. Diffractogram CaXRD charcoal before activation

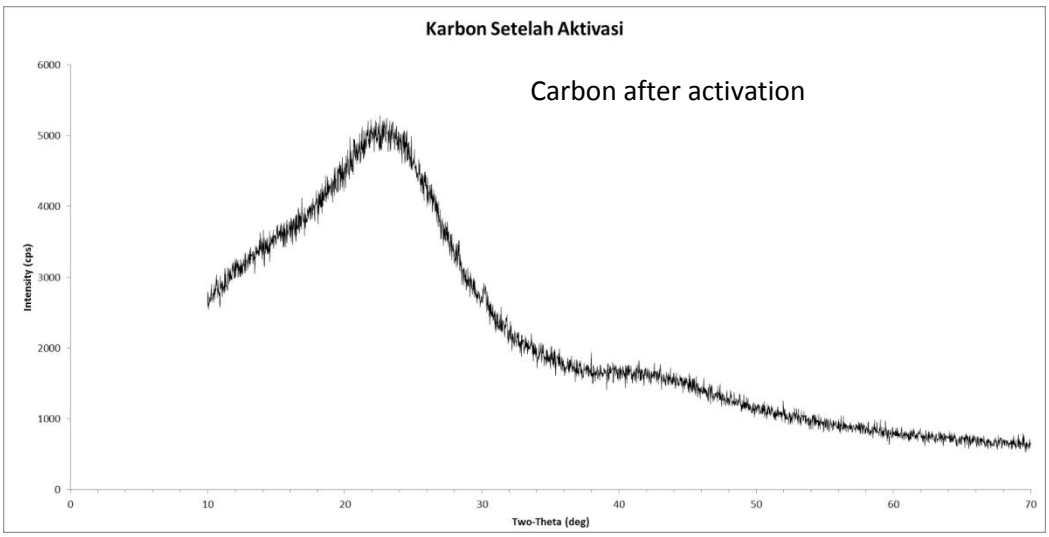


Figure 9. Diffractogram XRD charcoal after activation

CONCLUSION

The character of coconut shell charcoal before and after activated using FTIR, SEM and XRD analysis tend to be polar with absorption bands -OH roups, C = C, C = O and C-O; nearly **similar** particle distribution, smooth charcoal surface and a visible presence of pores, semi crystal with a hexagonal crystal structure.

REFERENCES

[1] Anirudhan, T.S., Sreekumari, S.S., dan Bringle, C.D., 2009, Removal of Phenol from Water and Petroleum Industry Refinery Effuents by Activated Carbon Obtained from Coconut Coirpith, *Adsorption*., 15, 439-451.

[2] Elsayed, M.A., Hall, P.J. dan Heslop, M.J., 2007, Preparation and Structure Characterization of Carbon Prepared from Resorcinol-Formaldehyde Resin by CO_2 Activation, *Adsorption*, 13, 299-306.

[3] Fraga, M.A., Jordao, E., Mendes, M.J., Freitas, M.M.A., Faria, J.L., dan Figueredo, J.L.., 2002, Properties of Carbon-Supported Platinum Catalysts: Role of Carbon Surface Sites, *J. Catal*., 209, 355-364.

[4] Gupta, S., Sahajwalla, V., Burgo, J., Chaubal, P. dan Youmans, T., 2005, Carbon Structure of Coke at High Temperatures and Its Influence on Coke Fines in Blast Furnace Dust, *Metall and Mat.Trans* B., 36B, 385-394.

[5] Lalena, J.N., D.A Cleary, D.A., Carpenter, E.E., dan Dean, N.F., 2008, *Inorganic Materials Synthesis and Fabrication*, John Wiley & Sons, USA.

[6] Miyazaki, K., Maskawa, N., ; Kobayashi, W., Kaku, M., Yasumaru, N. dan Kiuchi, J., 2005, Reflectivity in Femtosecond-Laser-Induced Structural Changes of Diamond-like Carbon Film, *Appl. Phys*., A80, 17-21.

[7] Ozaki, J.I., Nozawa, K., Yamada, K., Uchiyama, Y., Yoshimoto, Y., Furuichi, A., Yokoyama, T., Oya, A., Brown, L.J. dan Cashion, J.D., 2006, Structure, Physicochemical Properties and Oxygen Reduction Activities of Carbons Derived from Ferrocene-Poly(furfuryl Alcohol) mixtures, *J. Appl. Electrochem.*, 36, 239-247.

[8] Rampe, M.J., Setiaji, B., Trisunaryanti, W. dan Triyono, 2011, Fabrication and Characterization of Carbon Composite from Coconut Shell Carbon, *Indo.J.Chem*., 11:2, 124-130.

[9] Rampe, M.J., Setiaji, B., Trisunaryanti, W. dan Triyono, 2014, Study on Growth of Carbon Crystal from Charcoal of Coconut Shell and PVA, *IJETAE*, 6:1, 174-178.

[10] Seok, J.S., Jung. S.C., Ko. Y.C. dan Sun, D.S., 2005, Development of Carbon Dioxide Adsorbents Using Carbon Materials Preparated Coconut Shell,

Korean J. Chem. Eng., 22:291-297.

[11] Sikalidis, C., Zorba, T., Chrissafis and Paraskevo, K.M., 2006, Iron Oxide Pigmenting Powders Producad by Thermal Treatment of Iron Solid Wastes from Steel Mill Pickling Lines, *J.Therm.Anal.Cal.,* 86, 411-415 .

[12] Wiratmoko, A., dan Halloran, J.W., 2009, Fabricated Carbon from Minimally Processed Coke and Coal Tar Pitch as a Carbon-Sequestering Construction Material, *J. Mater. Sci.*, 44, 2097-2100.