



Synthesis and Characterization of Bio-Coke from Charcoal Coconut Shell

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Abstract: In this paper, This research was aimed to study the utilization of polyvinyl alcohol (PVA) as the stimulant agent in synthesis coke from the charcoal, to study the effects of PVA in the form of either powder or solution on the character of structural growth of crystal and its physical-chemical properties and to study the effect of temperature (1000°C) on the character of graphite carbon. Several methods were performed such as Fourier Transform Infrared (FT-IR) spectrometry to determine the functional groups of the carbon, X-ray diffraction (XRD) to identify the structure of carbon crystal, Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) to analyze the topography of surface and to analyze the component of material.

Several carbon technologies, such as calcinations, mixing and sintering at high temperature, were carried out by employing charcoal produced from coconut shell as the raw material and PVA as the stimulant. Characterization of carbon material showed that there was a change on the physical-chemical properties of raw material which has been sintered at 1000°C, at which coke was produced in this process.

Keywords : Coke; PVA; coconut shell; FTIR; XRD; SEM-EDS.

1. Introduction

The structure of chemical bonds that is formed contributes to the establishment of superior properties of carbon materials (Buchman and Bryant, 1999). Carbon contains allotrope which has different types of chemical bonding structure, including graphite, diamond, black carbon, fullerenes, carbon nano tubes (CNT).

Graphite is type of carbon material obtained from carbon atoms with sp^2 orbital. One of these atoms forms a new bond with 3 carbon atoms. The microscopic material consists of graphite flat sheet from carbon atoms which are bonded, as the grapheme. The characteristics of the bond structure and dynamic interactions between the grapheme layers produce strong electrical conductivity properties (Buchman and Bryant, 1999).

The heating process, using various methods, has conducted to accelerate the adsorption of electron (Hirose *et al.*, 2002). The mechanism can be as the simple as removal of surface impurities, however it may also involve the removal of oxide and even the structural rearrangement (Sahajwalla *et al.*, 2004). The use of temperature from 1000 °C to 1720 °C results in the increase in ratio of graphite layer in the crystallite from 4.5 to 3.3 and the growth in diameter from 1.8 to 6.3 nm.

Coke can be obtained by carbonizing coconut shell, and polyvinyl alcohol (PVA) in a thermal decomposition process (Scok *et al.*, 2005; Tae-Hwan *et al.*, 2002). The main composition of coconut shell consists of cellulose, lignite, and hemicelluloses with a formula C, O, H, and N. These organic materials contain functional groups such as hydroxyl (R-OH), alkanes (R-(CH₂)-R'), carboxyl (R-COOH), carbonyl (R-CO-R'), ester (R-CO-OR'), linear and cyclic clusters ether (RoR') with a variation of carbon (van der Marrel and Beutelspacher, 1976). In this process, the cellular anatomic features of coconut shell and polyvinyl alcohol are retained in the new carbon material (Inagaki, 2000). The structural changes during carbonization and the physical properties of the resulting chars have been investigated in several studies.

Polyvinyl alcohol (PVA) as an inert and organic adhesive is used to obtain high density products. PVA was chosen due to its homogeneity in functional groups, linearity and solubility in water (Ilcin *et al.*, 2010). With increasingly high-temperature treatment of adhesive polyvinyl alcohol (PVA), it further affects the growth of grain to be larger in size.

Heating process of coconut shell will produce a gradual change. In the first stage of the carbonization, the carbon samples become coke. The second stage is graphitization, in which coke is changed into graphite carbon whose irregular structures tend to grow (over the graphite whose structure is irregular) (Gupta *et al.*, 2005; Miyazaki *et al.*, 2005). The nature of carbon depends on its source, and on the method and conditions of carbon materials gives impact on their various uses, primarily as carbon electrodes and carbon materials structure.

This study discusses the use of, coconut shell and polyvinyl alcohol (PVA) to produce coke (carbon bio-composite). The main purpose of this research is to study the crystal structure as affected by using parameters such as concentration of polyvinyl alcohol and sintering temperature.

2. Material and Method

2.1 Materials

This study used coconut shell charcoals as the raw material. This charcoal is the source of carbon and polyvinyl alcohol. Further, Meck stimulant and argon gas were used as inert atmosphere to get air free.

Calcinations was conducted using a tube-Thermolyne Furnaces (Sybron) Type 21100 with a maximum temperature of 1200 °C. Calcinated charcoal as raw material was obtained through 100 mesh grinding and sifting. For carbon materials, Tarno compacted Grocki 312 model a maximum of 20 tons was used. Sintering was carried out by using a Carbolite furnace- Edwards Pirani 501 A6D 1600 °C maximum temperature with argon gas as the atmosphere, Scanning Electron Microscopy (SEM) JEOL JSM-6360 LA and Energy Dispersive Spectroscopy (EDS) system JEOL, JED-2300, and Fourier Transform Infra Red (FTIR) Shimadzu model IR-Prestige 21 was used while the Goniometer model diffractometer with Cu K α ($\alpha = 1.54056 \text{ \AA}$) radiation was used for X-ray Diffraction (XRD).

2.2 Methods

1. Charcoal calcinations

Charcoal powder 100 mesh sieves was calcined in an inert state through two stages. First, was the calcinations at temperatures of 873 K for 3 hours with the existence of nitrogen gas then followed by the purification of charcoal powder which was extracted for 24 hours with 1 M hydrochloric acid at room temperature (Gupta *et al.*, 2005). The sample was washed with distilled water until it reached constant pH and was dried in an oven at temperatures of 823 K with nitrogen.

2. The process of carbonization

The carbon powder was calcined with 2.5 to 7.5% wt. polyvinyl alcohol then suspended in distilled water at 353 K and stirred for one hour. Binder system was mixed with charcoal powder, forming a paste with the density in different systems. The next step was molding by using a cylindrical mold with a diameter of ~ 15 mm. Compaction was performed with one direction force by the means of Tarno Grocki with the thrust of 5 tons. In this stage, the process produced a sample of pellets (green compact). The samples were then dried at

room temperature for one day before oven drying for 4 hours at 383 K. The rate of temperature was 10 K/min and reaction time was 3 hours. Then, these samples were cooled in the furnace at a rate of 8 K/min, with a flow of argon gas. This process produced dense coconut shell coke or carbon-carbon composite materials (Wiratmoko and Halloran, 2009; Miyazaki *et al.*, 2005; Mothe and de Miranda, 2009).

3. Result and Discussion

SEM-EDS analysis

Figure 1 showed the SEM photographs of coke (PVA 5% wt.) sintered at 1273 K. Figure 1 showed that coke was non-homogeneous particles distribution. EDS analysis for the coke, carbon/PVA (5% wt.) sintered at 1273 K were shown in Figure 2, it was apparent that chemistry composition of the coke consisted of C element was 97,35% wt. Heat treatment ranging from 873 to 1273 K, as demonstrated in this study, caused the aromatic layers grow and coalesce each other (Rampe, *et al.*, 2011a; Inagaki, 2000). This is due to the evolution of volatile matter (Rampe, *et al.*, 12) and the breakage of cross-linking bonds such as CH₂ bridge.

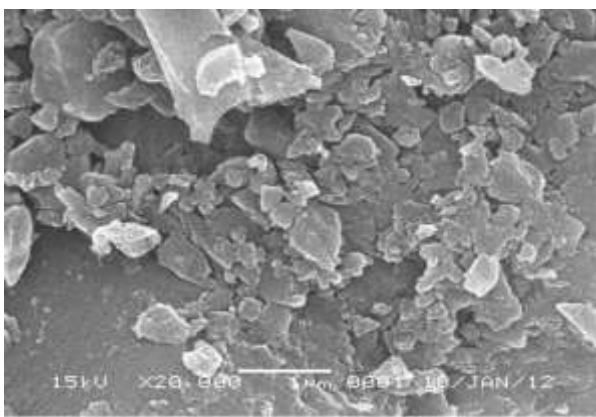


Figure 1. SEM micrographs of coke (PVA 5% wt), sintered at 1273 K

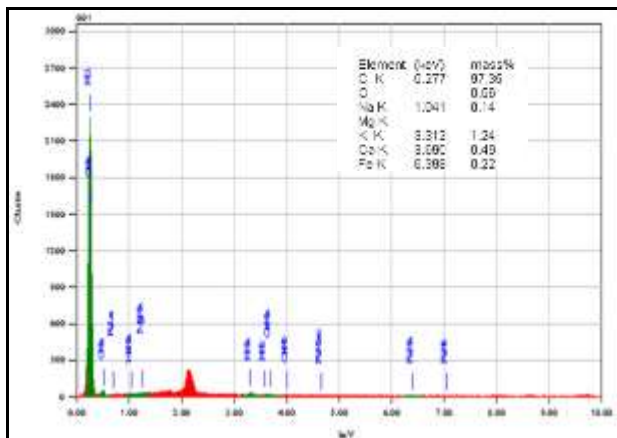


Figure 2. EDS analysis of the coke (PVA 5% wt) at 1273 K atmosphere argon

XRD analysis

Figure 3 showed a typical X-ray diffraction (XRD) pattern of testing coke carbon material gave of interlayer spacing (d) (Å) as a graphite semi-crystalline structure. Yin *et al.* (2009) reported that the decrease of the (d) and the increases of the crystalline diameter and average stacking height of the aromatic carbon sheets (L_c) with increasing the temperature suggested the development of stacking structure, increased the size of crystallite as well as removal of defects and increased order in carbon materials structure. The increase of L_c with increasing temperature resulted from crystallite growth in-plane and coalescence of crystallites. In this process, the structure of amorphous carbon structure was changed into semi-crystalline structure with a better

degree of order, namely turbostratic structure (Rampe, *et al.*, 2010). There was the change of internal structure by setting the position of equilibrium carbon atoms.

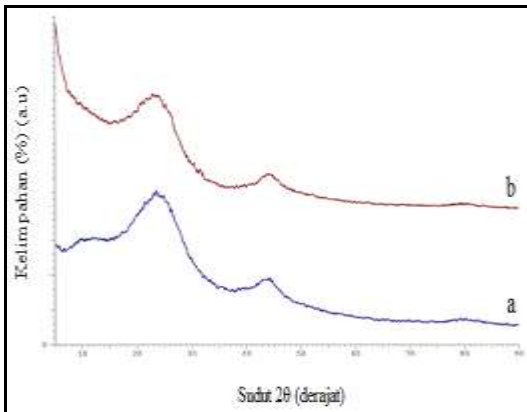


Figure 3 XRD pattern of coke sintered at 1273 K (Ar) : (a) 2,5% wt. PVA and (b) 5% wt. PVA

FTIR Analysis

Figure 4 showed that three spectra between coke 2.5% wt. PVA, 5.0% wt. PVA, and 7.5% wt. PVA (atmospheric argon gas) that has been sintered at a temperature of 1273 K. All three spectra gave the peak absorption bands that basically have the same pattern. The spectra showed a broad band near 3425 cm^{-1} which indicated the presence of hydroxyl groups on the carbon surface. The stretching was attributed to the absorbed water on the surface of coke and carbon black. The stretching frequencies of the aromatic C=C and aromatic C-H groups gave rise to peak at 2924 and 2862, respectively, which were originally existed as the support for the coke and graphite (van der Marrel and Beutelspacher, 1976); Rampe *et al.*, 2011c). The peak at near 1600 cm^{-1} ($\nu_{\text{C=O}}$) along with another peak at 1635 cm^{-1} ($\nu_{\text{C-O}}$) indicated the presence of carboxylic acid groups. The peak at 1635 cm^{-1} was assigned to a conjugated hydrogen bonded carboxyl groups, assigned by several authors on coke. The peak at 2924 cm^{-1} was due to C-H stretching of CH_2 groups (Maria and Teixeira, 2009). The bands near 1600 cm^{-1} indicated the fingerprint region of C=O, C-O and C-H groups that exist as functional groups of coke and carbon black. The band near 972 cm^{-1} was due to out-of-plane bending of C-H group in benzene derivatives.

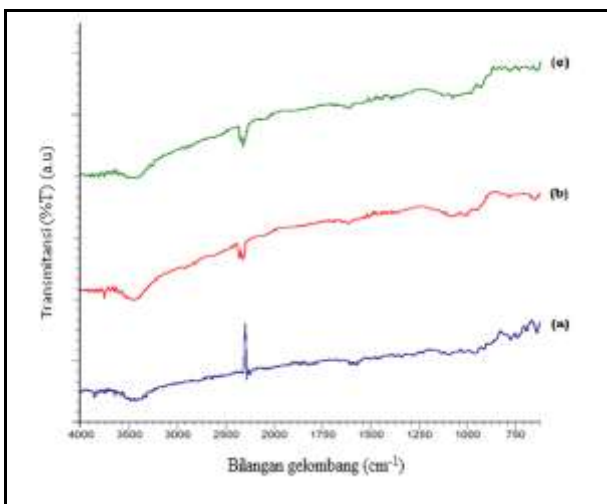


Figure 4. FTIR spectra pattern of coke (carbon/PVA) sintering at 1273 K, argon atmosphere: (a) 2.5% wt. PVA, (b) 5.0% wt. PVA, and (c) 7.5% wt. PVA.

4. Conclusion

Coke can be prepared from carbonized coconut shell and polyvinyl alcohol (PVA). The results showed that the products were in uniform particle size of micrometer dimensions and spherical particles in shape.

Increasing sintering temperature, the structure of amorphous carbon structure was changed into turbostratic structure with a higher purity level.

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