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Pore structure and specific surface area of the carbon electrodes from Coconut Shell Charcoal sintering up to temperature of 1500°C

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Abstract : Study pore structure and specific surface area of the carbon electrodes from charcoal coconut shell sintering up to temperature of 1500 C has been conducted. This research was aimed to study the effects of PVA as agent in the from of either powder or solution on pore structure and specific surface area and the effect of temperature up temperature of 1500 °C on the characters of the carbon electrodes. Several methods of characteristics were performed including specific surface area, total volume of pore and averaged pore radii the component of material. The product characteristics of carbon electrodes showed that there was a change on the specific surface area and averaged pore radii has been sintered up to 1500 °C, the pore diameter declined by the increasing of the temperature.

Keywords : coconut shell charcoal, pore structure, specific surface carbon area, electrode.

Introduction

Carbon material is one kind of material that has enough potential use in the field of engineering and construction. Carbon material used is usually allotrop of macromolecules composed of carbon atoms. The carbon atoms form a unique molecular structure. The structure of the chemical bonds formed contribute to the superior properties of carbon materials. The functions and properties of a material is closely related to how the structure of chemical bonds are formed.

Carbon materials have several types allotrop (a form of carbon material of different chemical bond structures), including graphite, diamond, carbon black, fullerene, carbon nano tube (CNT). Graphite is a type of carbon material formed from carbon atoms that form the orbital sp^2. One carbon atom bonded to three other carbon atoms ([1]). When seen in a microscopic scale, this graphite material consisting of flat sheets of carbon atoms are bonded, called Grafen. Grafen-Grafen each other to form a bond with each other through bonding weak van der Waals bonding. The nature of the bond structure and the dynamic interaction between the sheets Grafen generate large electrical conductivity properties as well as the nature of the lubricant (lube). Applications in the field of this graphite material acts as a conductor of electricity and material for lubricants.

Carbon in the industry is a snippet of carbon that has been heated to a temperature of 1000 - 1300 °C, while graphite is a snippet of carbon that has been heated to a temperature of 2500 °C and above. Graphitization is the provision of heat to the material that has been experiencing carbonization where unwanted material such as hydrogen, sulfur and other elements that are still lagging behind slowly will be expelled to the outside. In this process changes the structure of turbostatik into a structure with a degree of regularity better, the crystal structure of graphite ([2,3,4,5]). Graphitization heat treatment is more often performed at a temperature of not less than 2500 °C. In general, the manufacture of graphite done by heating carbon aromatic compounds at elevated temperatures up to 3000 °C.

Heating at high temperatures, up to 3000°C cause carbon has an irregular structure will evolve towards a more regular graphite structure by removing the volatile pollutant material. The structure of a pure substance shows the potential energy content stored on such material. During heating the carbon content increases, on the other hand oxygen, hydrogen and nitrogen decreases. Coke contains approximately 97% carbon ([6,7,8,9]) which was instrumental to the nature of the solid fuel is the calorific value associated with the chemical composition and structure molecular, ash content, sulfur content, and carbon bonded.

The main composition of coconut shell consists of cellulose, lignin, hemicellulose containing atoms of C, O, H, and N. These materials are organic-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 '), cluster ether linear and cyclic (RO-R') with varying amounts ([4,5,6]). The most common chemical reaction is combustion, which is a combination of a fuel with oxygen to form the product compound. This chemical transformation is the potential energy at the molecular scale, in this case related to the position of atoms and molecular structure.

Charcoal is a porous solid material and the heating of the material containing carbon. Most of the pores are still covered with hydrocarbons, tar and other organic compounds and components consist of bonded carbon, ash, water, nitrogen and sulfur. Charcoal can be made by direct or indirect heating in a pile or furnace. In this decomposition process in addition to the charcoal can produce other products such as distillate and gas. Products that have commercial value especially is charcoal.

The heating process is carried out on a coconut shell carbon will result in material changes gradually. The first phase is known as carbonization, ie carbon footage into coke ([10, 11]). The second phase is known as a stage of graphitization is to convert the coke into graphite so that the carbon structure of irregular tend to evolve towards graphite structure regularly ([12,13,14]). Properties of carbon is very dependent on the origin of the raw material of carbon materials were obtained, in addition to the methods and conditions of production. Properties of carbon materials is very important in a variety of uses, mainly as an adsorbent, the carrier of the catalyst, molecular sieve (molecular sieves), the electrode material, the carbon structure and so on ([15,16,17,18,19,20]).

The treatment of the heating temperature and reaction time (long detention) in the process of synthesizing the carbon material is intended to determine the relationship of microstructure, chemical composition, physical properties of the resulting carbon material. Data obtained in the form of system crystallography (structure crystal) through the technique of X-ray Diffarction (XRD), the spectrum of surface topography (micro structure) via analytical techniques Scanning Electron Microscopy (SEM), the spectrum shows the composition of the elements by the energy level of the technique of Energy Dispersive Spectroscopy (EDS), the functional groups by analysis of Fourier Transform Infra Red (FTIR), physical properties and thermal ([20, 21, 22, 23]).

Important properties of important properties of carbon depend on the micro structure, crystallographic system (crystal structure), composition, porosity, and particle size. Therefore, in this study developed the synthesis of carbon electrodes using coconut shell charcoal pyrolysis result, the powder metallurgy method (dry) and the solvent method using a modified carbon technology process. Studied the effect of sintering temperature and polyvinyl alcohol (PVA) to the pore structure and surface area.

Experimental

Material

Raw materials used in this study is charcoal result of pyrolysis of coconut shell of PT Tropica Nucifera Industry Bantul, Yogyakarta, Indonesia as a raw material source of carbon, HCl (pa Merck), PVA (pa Merck), ethanol (pa Merck), acetone (pa Merck) , universal indicators, paper Whatmann 42, Nitrogen gas from PT Aneka gas Putera Mandiri gas, argon gas, and distilled water.

Tools

The tools used in this study includes a number of glassware commonly used in laboratory, mortar agat, sieve of 100 mesh (USA Standard Testing Sieve), oven models gravity vonvection, electric furnace Carbolite models 2132 (Max Temperature 1200°C), Tube Furnace -Thermolyne (Sybron) Type 21100, Balance aND GR-200, thermometers, clamps, magnets, prints pellet, disk mill, hot plate (stir and heat), Stuart Scientifik (Rotator drivestry) to mix the material, Tarno Grocki 312 models max 20 ton, Carbolite-Edwards Pirani 501 A6d for maximum sintering temperature 1600°C, Buehler Ltd. to finishing, Quantachrome Instruments for testing pore structure and surface area.

Procedure

Setting up carbon

Coconut shell pyrolysis process is done using a pyrolysis reactor contained in PT Tropica Nucifera Industry Bantul- Yogyakarta. Coconut shell of coconut types-in the main raw material sample. The technique of random sampling.

Coconut shell charcoal from the pyrolysis is purified of carbon fiber which is attached with a knife, then made pieces of a smaller size by grinding using porcelain mortar. Delicate pieces of charcoal and then crushed into powder and sieved with a 100 mesh sieve to uniform particle size ([20]). Obtained carbon powder with a particle size of 100 mesh sieve passes.

Furthermore charcoal powder passes a 100 mesh sieve calcination reactor tube inserted into the furnace. Charcoal calcined at a temperature of 600°C for 3 hours is calculated when reaching that temperature, the gas flowing N2 ([15])

Charcoal calcination results do purification of 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. Then the charcoal is washed with distilled water until the washing water shows a constant pH, and then dried in an oven at 110 ° C for overnight ([25, 26]).

Furthermore charcoal purified carbon powder passes a 100 mesh sieve calcination reactor tube inserted into the furnace. Carbon calcined at a temperature of 750°C for 3 hours is calculated when the achievement temperature, with gas flowing N_2 ([2, 15]).

Synthesis Cokes done in 2 ways: first way: the dry method and means II: method solvent. Method I: a dry method (method of powder metallurgy), 0.25 to 1 gram (2.5-10 wt%) polyvinyl alcohol (PVA) from 9 to 9.75 grams of carbon mixed with calcination results using Scientifik Stuart (Rotator drivestry) speed 20 rpm for 3 hours. Method II: solvent method, as much as 2.5-7.5% by weight (0.25 to 0.75 grams) polyvinyl alcohol (PVA) with calcination results coconut shell carbon dissolved in distilled water (2.5-10 mL) at temperatures 80°C, stirred for 60 minutes until a perfect hydrolyzed. System solution of polyvinyl alcohol (PVA) is mixed with carbon results calcination, the mixing process until a homogeneous mixture, then dried at room temperature overnight.

Furthermore, the results of both methods of mixing is printed using a cylindrical mold with an inside diameter of ~ 15 mm. Compaction is done with an emphasis on one direction by means Tarno Grocki 312 models with a press force of 10 tons. This process results in a sample of pellets (green compact). Samples were obtained in this way is then dried at room temperature for 24 hours, and continue the process of drying in an oven at a temperature of 110°C for 4 hours. Samples were obtained in this way are then put into the furnace for sintering. Samples undergo a sintering process at a temperature of 1000°C and the hold time for 3 hours in the furnace Carbolite, Edwards Pirani 501, A6d, cooling in the furnace (annealing) ([11, 17].

Synthesis of carbon electrodes

Coke reheated to a temperature sintering up tp temperatue of 1500 ° C; with a heating rate of 10 oC / min, argon gas atmosphere and hold time 3 hours in the furnace Carbolite, Edwards Pirani 501, A6d; by cooling in the furnace (annealing).

Surface area

Quantachrome Instruments version 2.0 is used for the analysis of pore structure and surface area. Gas analysis used nitrogen gas with a molecular diameter (D) 3.54 Å, at a temperature of 77.35 K. bath Determination of surface area by the BET method to calculate the surface area of the carbon and the carbon material products produced from coconut shell. BET method (Brunauer-Emmett-Teller) with two types of measurement namely single point and multi point. Measurements carried out when the profile single point isotherms have been understood and implemented at a value of partial pressure of adsorbate where isotherm linear profile. While the multi-point measurement is done if the profile is not yet known isotherm done by varying the value of adsorbat partial pressure in the range of $0.05 <(P / Po) <0.35$. When adsorbate used is nitrogen gas, then liquid nitrogen is used as coolant. In addition, with the help of the development of the BET equation can determine the total pore volume and average pore diameter.

Results and Discussion

Van der Waals forces or chemical bonding causes the solid surface molecules adsorb gases or liquids. The main chemical bond between the surface and the adsorbed gas occurs in the adsorption-chemistry, and composition and surface structure changed. Van der Waals interactions cause the surface to physically absorb the gas below the critical temperature of the gas, is rapid and reversible, which can be eliminated by lowering pressure or increasing temperature ([22, 27]).

Table 1 shows the data mean pore fingers and surface area of coconut shell charcoal and carbon products sintering at a temperature of 1500 °C. Specific surface area and pore fingers mean products that have sintering at a temperature of 1500 °C with a concentration of 5%, respectively for 18.1034 $m^2 g^{-1}$; 0.3070 nm and 2,079 Å. Figure 1 shows the characters carbon sintering at a temperature of 1500 °C. Furthermore, the specific surface area and mean pore finger carbon electrode product with powder sintering method at a temperature of 1500 °C is equal to 9.785 $m^2 g^{-1}$ and the average pore finger of 0.6102 nm; listed in Table 1

Table 1 Properties of porous carbon material sintering at a temperature of 1400 (a) and 1500 °C

Sample	**Averaged pore radii (nm)**	**Specific surface area ($m^2 g^{-1}$)**
Solvent-5 % PVA	0.3070	18.1034 (a)
Solvent-5 % PVA	0.6102	9.785 (b)

Furthermore charcoal sintering at a temperature of 1500 °C provides specific surface area and mean pore finger is 4.486 $m^2 g^{-1}$ and 6.5177 Å. The application of high temperatures up to 1500 °C a change in the surface area decreases and increases the average pore finger. Reduced specific surface area resulting from the processing of sintering temperature is high then there is a change in the chemical composition, namely the elimination of pollutant material such as the elements contained in the surface of the material (Mendez et al., 2008), subsequently occur rearrangement within the framework of the carbon structure resulting in the shrinkage of pores and changes in pore particle ([10, 19]).

The specific surface area is the surface area of particles per unit volume or time, which can be determined by physical adsorption or chemical adsorption gas. The specific surface area carbon materials experimentally determined depending on the size of molecules adsorbed relative to the size of the pore. Small gas molecules can penetrate the pores smaller than 2 nm. Physical adsorption gas is used to determine the specific surface area of the carbon material powder.

Table 2 Pore Structure and specific surface area carbon materials that have structures sintering at a temperature of 1400 to 1500 °C.

Sample	Pore Structure		Spesific surface area, $m^2\ g^{-1}$
	Pore radius, Å	Total Pore Volume, cc s^{-1}	
Charcoal-1500	6,518	1,462	4,486
Solvent-5-1400	3,070	2,079	13,541
Powder-5-1500	6,153	3,010	9,785

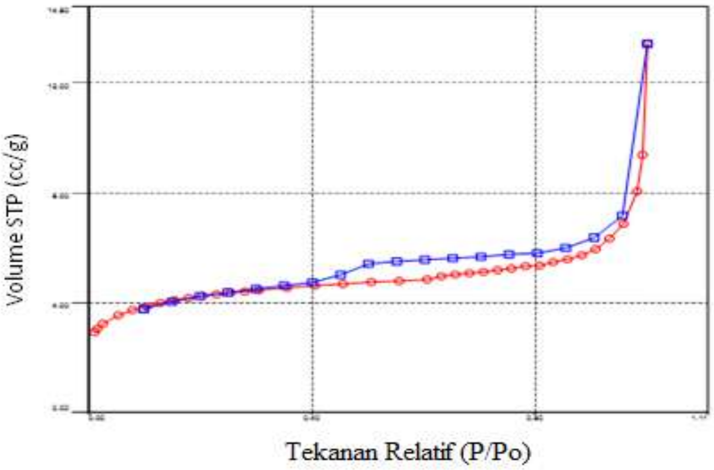

Figure 1 Adsorption and desorption isothermal N_2 to a carbon material that has been disinterring at a temperature of 1400 °C with a stream of argon gas.

In figure 1 above pattern of adsorption and desorption isothermal N_2 for carbon materials with a solvent method which has sintering at a temperature of 1400 °C with argon gas flow patterns show the adsorption and desorption in which the value of pressure increase followed by an increase in the value of P / Po to approach the value of 1 . In this condition the adsorption and desorption system shows the maximum process.

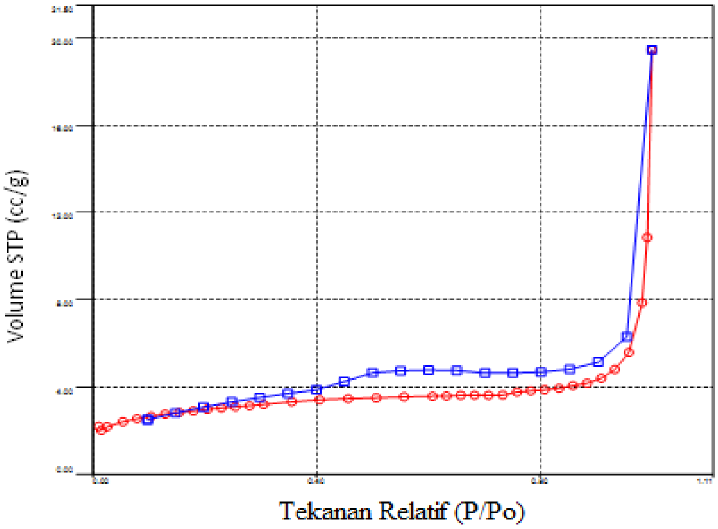

Figure 2 Adsorption and desorption isothermal N_2 to a material product carbon from coconut shell charcoal-PVA 5% by mass of the powder sintering method at a temperature of 1500 °C.

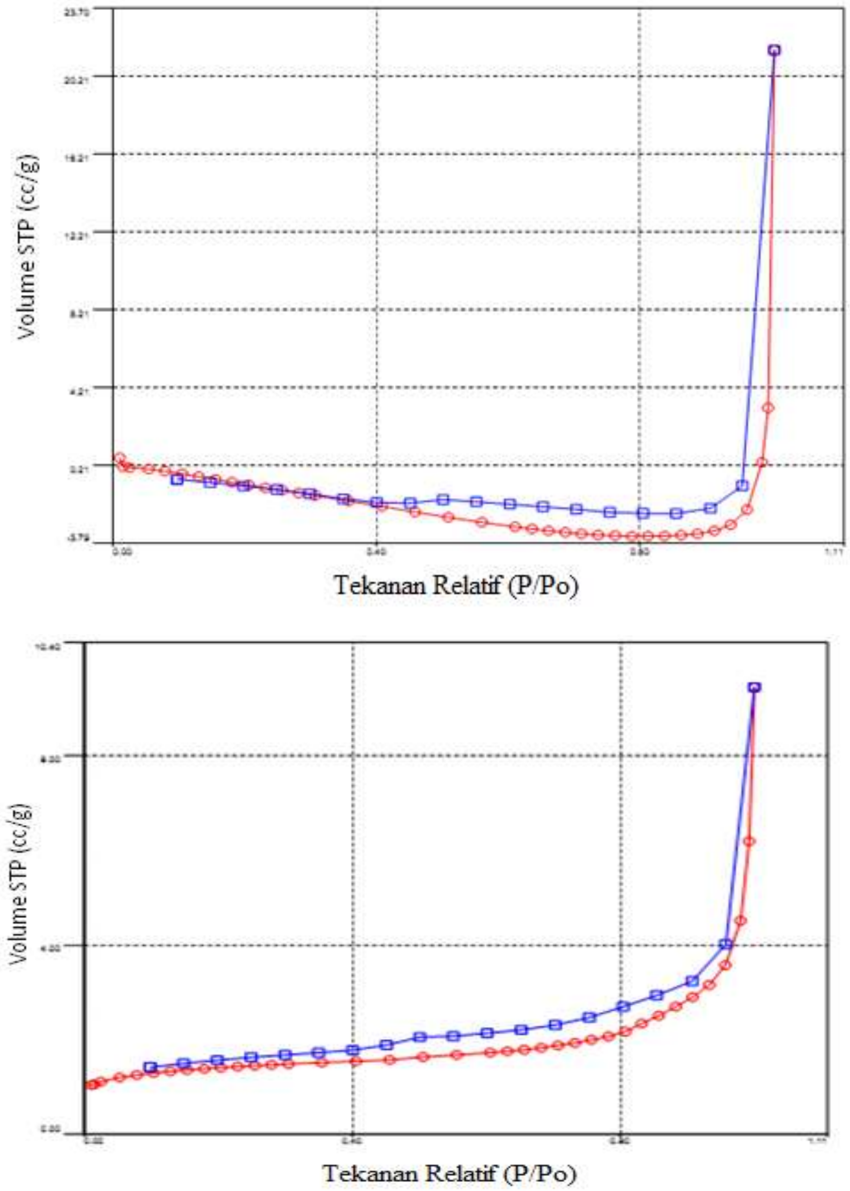

Figure 4 Adsorption and desorption isothermal N2 carbon products sintering at a temperature of 1500 °C: (a) carbon base material, and (b) of coconut shell charcoal-PVA 5% by mass with solvent method.

Analysis of pore structure is used to obtain information about the size and pore structure of calcined aggregate, soft agglomerates and granules, and applied high temperature material. Specific surface area of the powder is very depending on size and shape, and the content of submicron particle size and fine pores or fissures on the surface of the particles. The specific surface area nonpori powder containing particles can be used as an index of relative absorptivity for surface modifying processing approach is required in particle processing operations ([27]).

Table 2 and Figure 4 shows the data mean pore fingers and surface area of coconut shell charcoal and carbon sintering product to a temperature of 1500 °C. Charcoal specific surface area and mean pore finger calcined at a temperature of 750 °C with a stream of nitrogen gas in the amount of 106.589 m2 g-1 and 1.6298 Å. Furthermore charcoal sintering at a temperature of 1500 °C provides specific surface area and mean pore finger is 4.486 m2 g-1 and 6.5177 Å. The application of high temperatures up to 1500 °C a change in the surface area decreases and increases the average pore finger. Reduced specific surface area caused by high temperature sintering processing the changes in the chemical composition of materials, namely the elimination of impurities such elements contained on the surface of the material, then going back arrangements within the framework of the carbon structure resulting in the shrinkage of pores and pore particle changes.

Figure 5 shows the character of coke and carbon which has sintering temperature of 1000 to 1500 °C. The specific surface area and mean pore finger coke carbon particles and carbon products which have sintering at temperatures of 1000 and 1400 °C with a concentration of 5%, respectively for 196.338 m2 g-1; 13.541 m2 g-1 and 1.41511 Å; Å 3.0700. Figure 5.33 shows the characters carbon sintering at a temperature of 1500 ° C. Furthermore, the specific surface area and mean pore finger carbon products sintering at a temperature of 1500 ° C is equal to 9.785 m2 g-1 and 6.153 Å.

Characterization of surface area by the BET method to measure the surface area of the carbon produced from high-temperature heating. BET method developed by Brunauer-Emmet-Teller in 1938 with two types of measurement, single-point and multi-point. Single point measurement is done when the isotherm profile has been understood and implemented at a value of partial pressure of adsorbate where isoterm unknown profile is done by varying the value of the partial pressure of the adsorbate in the range of $0.05 < (P / Po) < 0.35$. When adsorbate used is nitrogen gas, then liquid nitrogen is used as coolant media. Additionally, through the BET characterization we also can know the total pore volume and averagepore diameter ([22, 27]).

Large pore of the carbon material increases after sintering at a temperature of 1400 to 1500 °C, where the temperature rise occurs pore increase of 3,070 into Å 6.152. The pore volume of carbon materials also increases with temperature rise, then the BET surface area is reduced.

The sintering temperature to a temperature of 1500 °C shows the changes in the reduced surface area of the carbon material. This is due to the increase in temperature then decreased porosity and pore volume, this is due to happen rearrangement of the carbon atoms in the carbon crystal structure of the arrangement.

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