

Investigating The Effects of Activation Temperature on The Crystal Structure of Activated Charcoal From Palm Bunches (Arengga Pinnata Merr.)

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Abstract

This research aims to determine the effect of activation temperature the crystal structure of activated charcoal. The material used activated charcoal bunches (Arengga Pinnate Merr). The process of making activated charcoal divided into two, namely the carbonization stage at a temperature of 250-400 °C and the activation stage at a temperature variation of 600 - 800 °C. To find the crystal structure, the sample characterized by X-Ray Diffraction. The results of analysis the dominant elements diamond before activation with a percentage of 90.2% and an orthorhombic crystalline structure, where the lattice parameter $a = 4.12700 \text{ \AA}$; $b = 4.93700 \text{ \AA}$; $c = 4.81900 \text{ \AA}$. Peak Carbon has a hexagonal crystal structure in all temperature variations. Peak Graphite an orthorhombic crystal structure and at a temperature of 700 °C a hexagonal crystal structure formed. Therefore, giving temperature variations the activated charcoal of the bunches affects the structure of the formed Crystal. Wherein increasing the activation temperature, the crystal structure that forms look more amorphous marked by a widening diffraction peak intensity decreased crystals.

Keywords: Crystal structure, temperature activation, XRD, bunches of palm

I. Introduction

Bunches of the palm is a plant with many stems, which measure about two feet. All filled with fruit that is green when young and yellowish brown when ripe. Then, empty bunches where fruits used to process into food (Indonesia names kolang-Kaling) usually left dry or used as firewood. According to [1] his research component of chemical compounds included in waste palm bunches lignin is 27.74%, 68.11% (hemicellulose), 33.79% (α -cellulose), 11.10% water content extractive, so palm bunches are very high potential to be used as active charcoal.

Bunches of palm production function produces a variety of commodities that have high economic value and potentially serious export if cultivated, such as palm stems produce flour if palm sugar not intercepted and palm wood used as raw material for making furniture. All parts of the sugar plant have a production function and the function of conservation highly effective one to reduce Characterization directly into the ground to prevent erosion [2].

Some researchers have examined active charcoal made from palm bunches. Some of them [1] arguing

that the activated charcoal of aren bunches consists of α -cellulose isolation elements from palm bunches about 20-26% and 16-21.33% microcrystalline cellulose, with microcrystalline structures consisting of particles with a form irregular, odorless, white, tasteless, with pH 7.4 and drying shrinkage 8.6%. Microcrystalline cellulose powder of palm bunches has good flow properties, with a resting angle of 20.47 Ha, Hausner index of 1.19, and compressibility index of 18.6%. Moreover, Hafiz et al., inform that microcrystalline cellulose pulp derived from oil palm empty fruit bunches filtered at room temperature and then dried in a vacuum oven at 105°C to constant weight reached. SIEMENS Difraktometer D5000 used to study the crystallinity of composite samples. XRD results explained that microcrystalline cellulose has its own or single cellulose structure with an 87% crystallinity index and a relatively high index of crystallinity with oil palm empty fruit pulp [3].

The yield of palm sugar leaves more bunches of sugar palm into the garbage. The trash from aren bunches is often considered useless and only becomes trash which turns out to have many

benefits for the community because its utilization to date only limited to tapping flower bunches for processing palm sugar juice. One of the benefits of palm bunches rarely known the public it can use as activated charcoal or briquettes which can be used to absorb the waste solids dissolved in water, remove odors, absorption of dyes, purification agents [4].

Utilization of palm bunches intended in addition to tackling the buildup of waste and palm bunches expected to produce products that safe and environmentally friendly [5]. Research on activated charcoal from palm bunches is very rarely found in various literature because currently the most developed is research on active charcoal from oil palm bunches, although palm bunches made into charcoal as a raw material for making activated charcoal. However, the activation process technology to produce activated charcoal to the characteristics of the crystalline structure of the corresponding standards still faces many obstacles. By knowing the crystal structure of an element, it will get a little information about the origins of the elements, physical properties, and mechanical.

One of them is the method of X-ray diffraction (X-RD) where samples will be shot using X-rays with wavelengths λ scale corresponding to the lattice spacing of a material that is the measurement result is a diffractogram — considering the importance of knowing the information about the structure of the activated charcoal crystal. So that researcher interested in researching with the title "effect of activation temperature on the crystal structure of activated charcoal from palm bunches." This research aims to learn the effect of activation temperature on the crystalline structure of activated charcoal formed from palm bunches.

II. Theory

The process of making activated charcoal

Activated charcoal making process made employing carbonation pyrolysis method. Pyrolysis is a heating process in the absence of oxygen [6]. [7][8] stated that the pyrolysis is a process of incomplete combustion of a carbon-containing compound that not oxidized to CO_2 . Primary pyrolysis divided into a slow process at a temperature of $150\text{ }^\circ\text{C}$ - $300\text{ }^\circ\text{C}$ which produces charcoal, H_2O , CO and CO_2 , and a fast process, transpires at temperatures of $300\text{ }^\circ\text{C}$ - $400\text{ }^\circ\text{C}$, which produces charcoal, gas, and H_2O [9]. After pyrolysis occurs at temperatures above $600\text{ }^\circ\text{C}$, which produce carbon monoxide,

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hydrogen gas, and hydrocarbon gases [10][11]. Next, [12] said states that when fast pyrolysis used (fast pyrolysis), heating for 0.1–0.5 seconds at a temperature of $400\text{ }^\circ\text{C}$ – $600\text{ }^\circ\text{C}$.

Authoring or carbonization process divided into four stages: (a) The evaporation stage of water, which occurs at temperatures of $100\text{ }^\circ\text{C}$ – $150\text{ }^\circ\text{C}$; (b) Stage of decomposition of hemicellulose and cellulose at a temperature of $200\text{ }^\circ\text{C}$ - $240\text{ }^\circ\text{C}$ into pyrogenic solution which a low boiling point organic acid such as acetic acid, formic acid, and methanol; (c) The stage of depolymerization and termination of C-O and C-C bonds, at a temperature of $240\text{ }^\circ\text{C}$ - $400\text{ }^\circ\text{C}$. In addition, the lignin begins to decompose to produce tar, decreasing the pyroglut and CO solution and increasing CO , CH_4 , and hydrogen gas; (d) The stage of forming an aromatic layer, which occurs at temperatures more than $400\text{ }^\circ\text{C}$ and lignin, continues to decompose to a temperature of $500\text{ }^\circ\text{C}$, whereas at temperatures over $600\text{ }^\circ\text{C}$ there is a process of enlarging the surface area of charcoal. Furthermore, charcoal can be purified or turned into activated charcoal at a temperature of $500\text{ }^\circ\text{C}$ - $1000\text{ }^\circ\text{C}$ [13][14][15].

The crystal structure

Crystalline structure is a condition or condition in which a typical arrangement of atoms in a crystal arranged specially and periodically repeats in three dimensions on a crystal lattice due the heating process resulting in more regular changes in the atomic structure [16]. The arrangement occurs because of geometric conditions that must meet the permanence of trending atomic bonds and tight arrangement. For knowing the crystal structure of an element, it will get a little information about the origins of the elements, physical properties, and mechanical.

Ideally, the most stable arrangement of polyhedra coordination is one that allows minimum energy per unit volume. This situation achieved if: a) electricity neutrality fulfilled; b) discrete and directed covalent bonds fulfilled; c) the repulsion force of ions becomes minimal; d) the arrangement of atoms as close as possible [17]. Lattice is an arrangement of points in three-dimensional space in which each point has a similar environment. The point with a similar environment named the lattice points. Knot lattice arranged only in 14 different configurations, named lattice Bravais. Based on the theory of the higher temperatures will increase the

degree of crystallinity carbonized charcoal because of the high temperature capable of breaking the carbon chains that increasingly disordered arrangement of carbon atoms [18]. The degree of crystallization seen from the peaks of XRD which viewed from the value of the microcell origin/match three software

III. Methodology

This research conducted at the physics education laboratory Universitas Halu Oleo and XRD sample characterization at the energy laboratory of the Sepuluh Nopember Institute of Technology. The

materials used are Arenga Pinnata Merr and using XRD (X-Ray Diffraction) characterization tools. The research stage divided into two stages, namely the carbonation stage at temperatures of 250-450°C and the activation phase of the sample starting from 600 - 800°C. The result of the analysis XRD in the form of an image of the activated crystalline charcoal structure of each bunch tested based on a comparison of temperature variations and a graph of the relationship of intensity and angle 2θ contained in each sample. The stages of the research described in Figure 1.

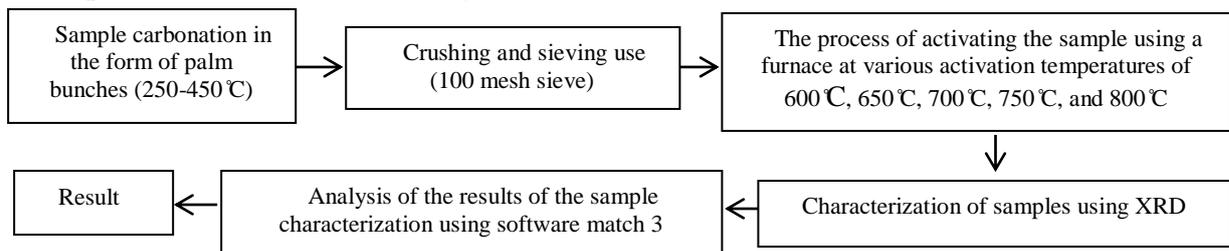


Figure 1. Research procedure

IV. Results and Discussion

Result

In this study to determine the crystal structure of the activated charcoal from palm bunches before heated, 600°C-800°C analyses by XRD, the source used Cu with a wavelength of $\lambda = 1.54060 \text{ \AA}$. The direct information obtained from the XRD

test of palm bunches for various temperature variations the form of a spectrum or graph between scattering angles (2θ) taken at a scattering angle of 5°-60°. The diffractogram graph of the XRD results seen in Figure 2.

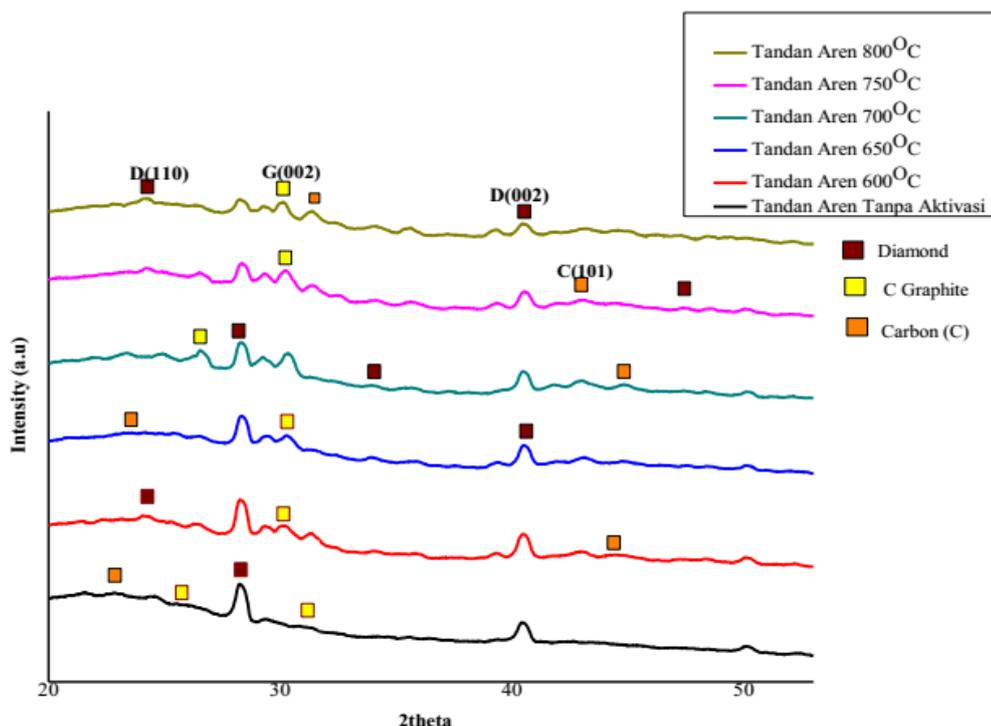


Figure 2. Graph of the Relationship of Intensity and 2θ in the Characteristics of Samples of Arsenic without Activation and Temperature of 600 °C to 800 °C

Figure 2 shows the change in diffraction peak as the activation temperature increases. A crystal structure assumed with a peak image where the position of the diffraction peak provides information about the lattice parameter, the shape of the crystal, while the relative intensity of the diffraction peak provides information about the position of the atom in a unit cell. To see the

relationship between angles 2θ and counts, Match three software used, which then analyzed the peaks formed. From Match 3 software results obtained in the form of structure or shape of crystals, elements contained, percentage of content and lattice parameters (Å). The results of the analysis of match-three software seen in Table 1.

Table 1. Results of testing by the method of X-Ray Diffraction (XRD) analysis using software Match

Temperature (°C)	Crystal Structure	Component	Persentase	2θ	The lattice parameter (Å)
Without Activation	Orthorhombic	Diamond	90,2%	28,35°	a= 4.12700 Å b=4.93700 Å c=4.81900 Å
	Orthorhombic	Graphite	5,7%	25,70°	a= 4.52500 Å b=5.33400 Å c=5.92500 Å
	Hexagonal	Carbon	4,1%	23,12°	a= 4.89000 Å c=3.88000 Å
600	Orthorhombic	Diamond	38,1%	24,23°	a= 4.87000 Å b=5.56500 Å c=4.40600 Å
	Orthorhombic	Graphite	15,8%	30,07°	a= 4.57500 Å b=5.30400 Å c=5.63500 Å
	Hexagonal	Carbon	46,1%	43,67°	a= 2.52210 Å c=12.35570 Å
650	Orthorhombic	Diamond	57,4%	40,54°	a= 4.96400 Å b=5.16300 Å c=4.38700 Å
	Orthorhombic	Graphite	21,4%	30,02°	a= 4.52500 Å b=5.33400 Å c=5.92500 Å
	Hexagonal	Carbon	21,1%	22,66°	a= 4.89000 Å c=3.88000 Å
700	Orthorhombic	Diamond	49,3%	28,34°	a= 4.12700 Å b=4.93700 Å c=4.81900 Å
	Hexagonal	Graphite	8,7%	26,69°	a= 2.45600 Å c=6.69600 Å
	Trigonal	Carbon	42,0%	44,58°	a= 2.52210 Å c=43.24500 Å
750	Orthorhombic	Diamond	66,3%	47,31°	a= 4.12700 Å b=4.93700 Å c=4.81900 Å
	Orthorhombic	Graphite	7,0%	30,22°	a= 4.57500 Å b=5.30400 Å c=5.63500 Å
	Hexagonal	Carbon	26,7%	42,86°	a= 2.52210 Å c=8.23710 Å
800	Orthorhombic	Diamond	84,6%	24,23°	a= 4.87000 Å b=5.56500 Å c=4.40600 Å
	Orthorhombic	Graphite	12,5%	30,09°	a= 4.52500 Å b=5.33400 Å c=5.92500 Å
	Hexagonal	Carbon	2,9%	31,20°	a= 4.89000 Å c=3.88000 Å

Furthermore, the characteristics of the crystalline material determined by atomic bonds in the crystal, the orientation of the crystal plane. The distance between the crystal plane and the crystal system. The typical arrangement of atoms in crystals called a crystal system or crystal structure. Diffraction is the process of X-ray scattering by

crystal material. The XRD method uses an X-ray which diffracted as a ray reflected from each plane, formed with crystal atoms from the material each sample with different temperature variations, it will produce a different diffraction pattern from the samples.

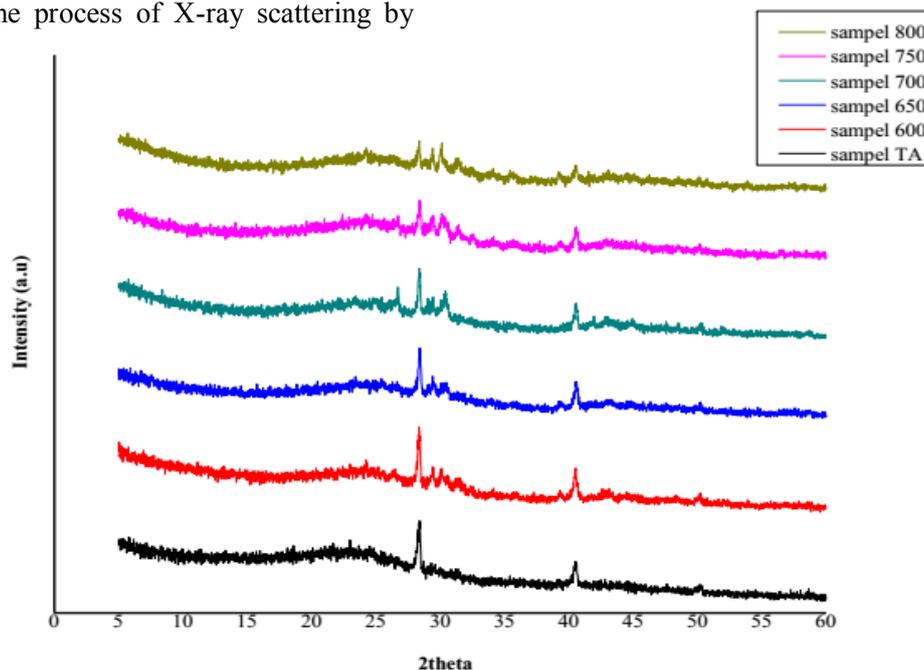


Figure 3. Diffractogram samples palm bunches on pre-heating and temperature of 600°C-800°C

Based on the graph (Figure 3) of measurement results with the XRD with the assisted of software match 3 before heated, 600°C, 650°C, 700°C, 750°C, 800°C shows that the resulting diffraction peak contains diamond elements, graphite, and carbon which not periodic and the actual diffraction peaks formed leads to crystalline graphite. Although the higher the activation temperature diffraction peaks, more dominant amorphous form characterized by irregular peaks generated. Crystal diffraction peaks formed at each temperature change the intensity of the diffraction peak, either increasing or decreasing.

Discussion

This study aims to determine the description of the crystalline structure formed in activated charcoal bunches using XRD (X-Ray Diffraction). The results of the analysis explain that there is a shift in the diffraction peaks towards the angle 2θ higher near the peak of graphite (26°) and new peaks appear around the 30° and 40° angles (Figure 3). Peak 26° and 40° characterize graphite in a flat plane (Planar L), whereas at angle 27° - 28° is a graphite crystal structure in the vertical plane. At different temperatures, the diffraction peak of the crystal formed at an angle of 28° still appears, what changes is the intensity of the peak diffraction. Manocha argues that the arrangement of activated carbon particles is allotropic, which can change from one crystal cell unit structure to another at a specific temperature [19].

The statement confirmed from the analysis of the samples sixth graph, the crystal structure changes, and the angular position of each element, obtained as a result of the given temperature. After the search Software match 3, on active charcoal samples, bunches of palm-based diffractogram (Figure 2) with different temperature variations arise diffraction peaks typically. The highest diffraction peak around the corner 2θ : 28.39° from the peak formed seen that in some of the top structure already shows the crystal structure although not periodic. The second known that the crystal structure has energy peaks sharp and narrow and at some of the top chart above shows the characteristics mentioned crystal structure located around the corner $28,35^\circ$, 30° , $40,54^\circ$. Although a relatively small diffraction intensity showed, an amorphous structure is more dominant than the crystal structure.

The crystal structure formed at a temperature of 700 °C more transforms the diffraction peak shifts due to the vibration of atoms where the graphite

element forms a hexagonal crystal structure which initially at a temperature of 650 °C with an orthorhombic structure. It evidenced by the percentage of graphite content from the sample before heated, which is 5.7% to 15.8% at a temperature of 600°C and increases to 21.4% at a temperature of 650°C, but the decline in temperatures 700°C is 8.7%. This data reinforced from the study of [20-21] that on X-ray investigation showing that microscopic crystalline activated charcoal is similar to graphite structure, where graphite consists of some plates arranged in parallel, and each plate has a hexagonal system with six carbon atom. With increasing temperatures given, the type of the element at some angle 2θ obtained will vary from one temperature to another temperature. Other materials also have a hexagonal crystal structure is CdSe or compounds of cadmium and selenium that widely used in solar cell technology. In the study [22-23] the thin layer of CdSe formed a polycrystalline crystal with a hexagonal crystal system. Changes in crystal structure with different materials have also studied [24] stated that the transformation of pyrolusite minerals with a tetragonal structure a cubic bixbyite structure than to tetragonal hausmannite occurred due to increased sintering temperature causing structural changes in manganese seeds.

Based on XRD analysis (Figure 2) when this diamond element fitting process found at the angles of 2θ (28° and 40°), but further research is needed to see if in certain circumstances diamonds can be amorphous or vice versa and still need to be verified by the existence of diamonds with other characterizations such as Raman Spectroscopy. The principle of Raman Spectroscopy is the interaction between light and the sun that uses a beam of monochromatic light such as a laser. Scattering through the sample cause the frequency shift and yielding information on vibration, rotation, or other low-frequency transition in the molecule and capable of providing a fingerprint where the molecules identified. Because generally the making of diamond elements not easy, so other proofs needed with different characterizations. Based on research conducted by [25] regarding the Raman Spectroscopy study of diamond and graphite in ureilite and the origin of diamonds explain that Raman Spectroscopy is a useful and non-destructive tool for checking the properties of carbon materials. Raman Spectroscopy studies to obtain information about diamond and graphite properties in ureilite. Graphite quickly produced with diamonds when the proportions of H_2 and

CH₄ change slightly. Notably, high CH₄ content seems to support the growth of graphite. Graphite in ureilite is well arranged compared to carbon material in chondroitin carbon. Domain dimensional graphite is estimated at 45-110 Å. Shifting the position the diamond peak to higher wave numbers seem to support the chemical vapor deposition of diamond origin ureilite containing carbon matrix. Its silicon coarse-grained (millimeter) and regarded as the accumulation of magma ultrasonic or partial melting residues.

However, impressive results appear where at a temperature of 700°C at an angle of 30.38°, the diffraction peak does not experience a decrease in intensity after the diffraction peak temperature of 650°C widens. The decline in the intensity of the diffraction peaks are clues to start the formation of aromatic compounds [26]. The compound is a constituent of hexagonal crystalline charcoal and activated charcoal structures. In Smallman's and [27-28] explained the effect of heating on the structure of minerals that formed is that the properties of the material will change if given treatment such as heating on the resources of manganese seeds. These events can be explained by physics when seed manganese samples heated at a precise temperature, the mineral structure changes in manganese ore samples into a new mineral which is also beneficial for the industry. Changes in the structure of manganese ore minerals can occur due to temperature sintering.

The changing structure of the material due to the temperature also caused when a material is heated; there will be an increase in energy, which allows the atoms vibrate at interatomic distances higher [29]. The higher the energy applied, the distance between atoms would be getting away. For a certain level of energy (temperature), atoms can get away from each other more quickly and are more difficult to suppress. The influence of energy on the distance between atoms, namely the more significant the energy yielded, the distance between atoms will be further away. Changing the spacing between atoms will affect the structure of the material.

V. Conclusion

From the results of the analysis of sugar palm bunches samples with six types of temperature treatment obtained by the dominating element is diamond found in the sample without activation with a percentage of 90.2% with an orthorhombic crystal structure. Giving the heating temperature variation or activated charcoal palm bunches affect

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the structure of the crystals formed. Where the temperature of activation increases, the structure of the formed Crystal is more amorphous, which is characterized by the irregularity of the resulting peak.

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