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Structural Characterization of Amorphous Carbon Films from Palmyra Sap

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Abstract. Carbon-based material and amorphous carbon film were extensively investigated for use in the solar cell. In this work, the amorphous carbon has synthesized from bio-product namely palmyra wine and palmyra brown sugar that taken from palmyra sap. The amorphous carbon was prepared by carburizing at 250°C. Then, the samples were calcined with varied at 400 °C and 600 °C for 2 h. Each sample was dissolved into a solvent. The solvents used in this research were aquades and DMSO (Dimethyl Sulfoxide). The solutions were exfoliated by using an ultrasonic cleaner for 2 h and then centrifuged at 4000 rpm for 30 min. The amorphous carbon in the liquid was deposited by a spin coating method on the ITO substrate. The XRD data showed that the patterns of the powder samples have a wide spectrum range at 2θ around 15-30° so the samples were amorphous. The FTIR absorption spectra in transmittance mode show that the sample dissolved into aquades (PBA) has functional group bonds such as O-H, C=C, C-C, C-H, C-O, C-N, N-H and C-I. On the other hand, the sample dissolved into DMSO (PBD) has functional group bonds such as O-H, C-H, O-H, -C=C-, C-H, C=C, C-C, C-N, N-H, and C-I in the range 500-4000 cm^{-1} . Microstructural characterization using SEM shows that the surface morphology of the PWA and PB samples were a lump and nodular, respectively.

INTRODUCTION

The requirement of energy for human increases with increasing civilization and technology. The energy sources from fossils used as electrical energy in this generation still very dominant. Although, in reality, the use of fossils as non-renewable energy will be exhausted if it continues to be exploited. This is the basis and motivation for researchers to find renewable energy, one of which is solar power. Indonesia's potential in developing solar cell energy is quite good because it is located on the equator.

Solar cells that are often used silicon-based solar cells as the result of the rapid development of electronic semiconductor technology. These solar cells are dominated by silicon material. However, the cost of consumption is more expensive than fossil energy sources. In addition, the other weakness of silicon-based solar cells is the use of harmful chemicals in the fabrication process. There is one material that has the potential to be an alternative material in the development of solar cells, i.e. carbon. Carbon has the same group as silicon in the periodic element table. It is possible for carbon to have similarities with silicon, so it is hoped that carbon can also be utilized in solar cell devices [1].

In recent years, amorphous carbon (a-C) has become a material developed by researchers as a basic material for solar cells. This is because it can be used to control the ratio of hybrid bonds. a-C is a non-crystalline form of carbon that only has a short-range structure. While the two main carbon crystals form 100% sp^3 (diamond) or 100% sp^2 (graphite) bonds, a-C films contain a mixture of sp^3 and sp^2 bonds. This is the ability to control the ratio of sp^3 bonds to sp^2 so that they can affect the mechanical, electrical, and optical properties of amorphous carbon. In addition, the hydrogen content also plays an important role in determining the nature of the film [2].

Palmyra tree which has the Latin name *Borassus flabelliferis* is a type of palm that grows in Southeast Asia and South Asia. In Indonesia, palmyra trees grow in East Java and eastern Central Java, Madura, Bali, West Nusa

Tenggara, East Nusa Tenggara and Sulawesi [3]. Palmyra trees have many benefits ranging from leaves, stems, fruits, and cob flowers. Palmyra flower cob if tapped can produce sap [4]. Palmyra sap can be obtained from the palmyra tree through stems of flower bunches that are cut or tapped. Palmyra sap contains brown sugar which is a source of carbon and there are sufficient nutrients as a growth medium for lactic acid bacteria. Besides, palmyra sap is also available in relatively large quantities and is cheap in price, but has not been processed optimally [5]. By heating and special treatment, palmyra sap can be processed into wine and brown sugar.

Mahtani [6] synthesized amorphous carbon layers by using the Radio Frequency Plasma Enhanced Chemical Vapor Deposition (RF-PECVD) deposition technique to analyze the effect of power and temperature on the percentage of sp^2 bonds and the optical properties of carbon thin films. Saleh [7] with the same deposition technique synthesizes carbon layers to determine the ratio of sp^2 bonds and sp^3 bonds by testing FTIR, Pamungkas [8] succeeded in fabricating amorphous carbon from coconut sap and obtaining energy gaps such as semiconductor materials in the range of 0.1585 eV-0.4873 eV

In this study, we used palmyra wine and palmyra brown sugar that taken from palmyra sap as a carbon-based material in development the study of solar cells. We investigated the characteristic in variations of solvents, i.e. aquades and DMSO (Dimethyl Sulfoxide) and in variations of the carbonization temperatures. In addition, the deposition technique of carbon was carried out using a simple method, namely spin coating.

EXPERIMENTAL DETAILS

Preparation of Carbon Solutions

The carbon materials were taken from the palmyra wine and palmyra brown sugar. The palmyra wine was processed by heating at a temperature of 100 °C and stirring at a speed of 500 rpm for 20 h. The palmyra wine after this process and palmyra brown sugar, respectively, are heated at a temperature of 250 °C for 2 h. Then both were further calcined at temperatures of 400 °C and 600 °C for 2 h, and thus the samples were designated as PW400, PW600, PB400, and PB600, respectively. The samples are then crushed and washed using aquades until it is clear to eliminate impurity and reduce the presence of KCl. Afterwards, the exfoliating process is carried out on PW400 and PB400 using an ultrasonic cleaner (Bransonic Cleaner) for 2 h with 2 solvents of aquades and DMSO which are denoted as PWA, PWD, PBA, and PBD, respectively. The ratio of the sample and solvent is 1 g:10 ml. The next step in the exfoliating process is the separation process using the centrifuge with a speed of 4000 rpm for 30 min.

Deposition of Amorphous Carbon Films

The substrate used in the deposition process of the carbon thin layer is a glass material with an ITO conductive layer in the dimension of 1×1 cm². The thing that needs to be considered before the deposition process is the cleaning of the glass with the aim to eliminate the impurity that is attached to the substrate because it can affect the properties of the deposition sample. The cleaning process of the substrate is done by washing with 50 ml of alcohol using an ultrasonic cleaner for 1 h. Then the substrate is dried on a hotplate at 40 °C for 30 min. The palmyra wine and palmyra brown sugar after the exfoliating process are coated on the substrate that has been cleaned using the spin coating method with a speed of 2000 rpm for 50 sec.

Characterization

Phase identification in this work was performed by X-Ray Diffraction (XRD, Philips X'Pert Multi-Purpose Diffractometer). In order to identify the functional groups of samples were carried out by testing using Fourier Transform Infrared (FTIR, Bruker Vertex 7.0v). The morphological structure of the amorphous carbon layer was tested using Scanning Electron Microscopy (SEM, FEI Inspect-S50).

RESULT AND DISCUSSION

Diffraction Pattern Analysis

The diffraction pattern of PW400 and PW600 can be seen in Fig. 1. The diffraction patterns show a wide range of formation at around 15-30° and the sharp peaks. The width peak identified the formation of an amorphous carbon phase. While the sharp peaks are the KCl phase formed in PW400 namely at 14.36°, 29.59°, 31.68°, 49.06°, and 53.84° with the crystal fields of (100), (110), (200), and (210). Whereas for PW600, KCl peaks formed at positions 25.27° and 31.41° with the crystal fields of (100) and (110).

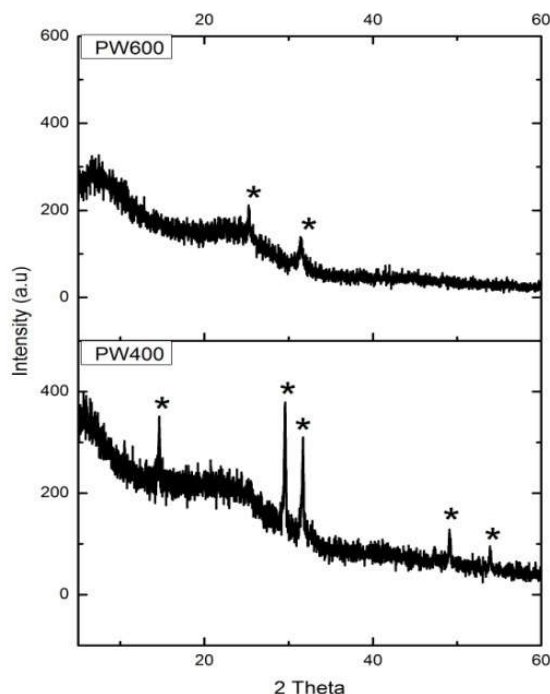


FIGURE 1. Diffraction Pattern of PW400 andPW600

The X-ray scattering pattern of PB400 and PB600 are shown in Fig. 2. The XRD patterns of PB400 and PB600 show a wide and irregular peak at around 15-30° which are identified as the formation of amorphous carbon. The high peaks formed of these samples, i.e. at 28.35°, 40.53° and 50.24°, is the peak of KCl. The KCl peaks formed in the crystal fields of (200), (202) and (222). The sharp small of PB600 is formed at 28.30° and 40.53° with the crystal fields of (200) and (202).

The presence of elements K and Cl in palmyra wine and palmyra brown sugar is derived from natural ingredients, namely palmyra sap. Nutrients and types of soil where palmyra trees grow are factors that influence the content of elements in wine and brown sugar. Based on the amount needed, nutrient elements are divided into two groups, namely macro and micronutrients. Macro nutrient elements are essential nutrient elements needed in large quantities. Micronutrients are essential nutrients needed in small amounts. Nutrient elements contained in the soil are Sodium (N), Phosphorus (P), Potassium (K), Calcium (Ca), Magnesium (Mg), and Sulfur (S). Micronutrient elements, for example, Iron (Fe), Manganese (Mn), Copper (Cu), Molybdenum (Mo), Boron (B), and Chlorine (Cl) [9].

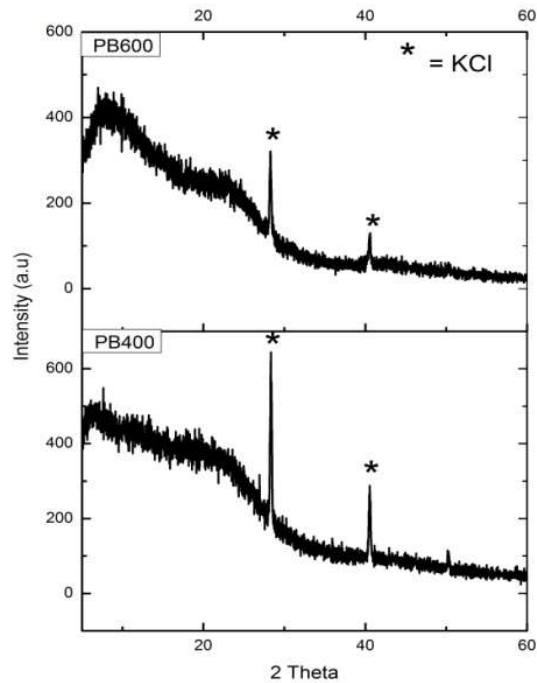


FIGURE 2. Diffraction Pattern of PB400 and PB600

The calcining process affects the KCl phase content on carbon powder. At a temperature of 600 °C, the KCl peaks from the palmyra wine sample and palmyra brown sugar decreases. The content of KCl in this sample can be further reduced by washing using aquades.

Morphology Analysis

Morphology characterization of amorphous carbon film can be identified using SEM (Scanning Electron Microscopy). In this study, SEM testing was carried out on amorphous carbon films deposited by carbon solution of palmyra wine with aquades solution and carburizing at 400°C (PWA) and amorphous carbon films deposited by carbon solution of palmyra brown sugar with DMSO solution and carburizing at 600°C (PBD) samples. Fig. 3(a) showed that the surface morphology of the PWA sample was elongated lump size of about 0.58 μm . Spread-free chunks indicate that the carbon layer deposited on the ITO substrate is uneven. Fig. 3(b) is a morphological structure of the PBD sample. The SEM test results showed that the surface morphology of PBD was nodular structures. The surface morphology of PBD was agglomerated and the grain boundaries were not clearly visible between each other.

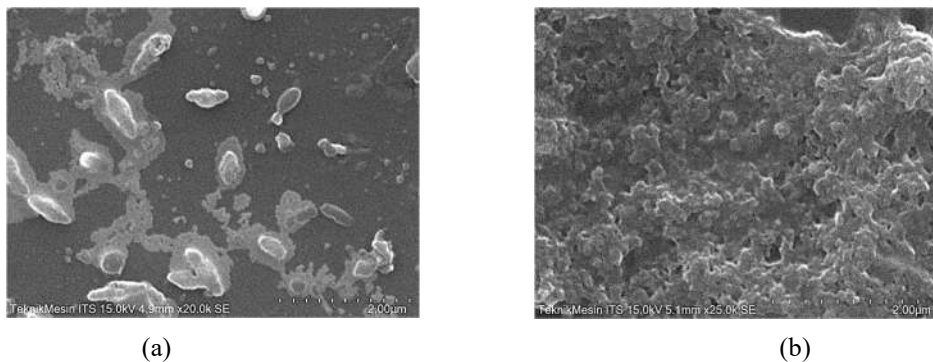


FIGURE 3. SEM Image of PWA and PBD

FTIR Analysis

Figure 4(a) showed that the FTIR spectrum of PWA. The infrared spectra of the PWA showed the presence of functional group bonds, namely O-H, C-H, N-H, C-Br, and C-C. Figure 4(b) showed the FTIR spectrum of PWD. The functional group bonds formed from the PWD are O-H, C-H, O-H, C=C, C-H, C-C, C-N, N-H and C-I.

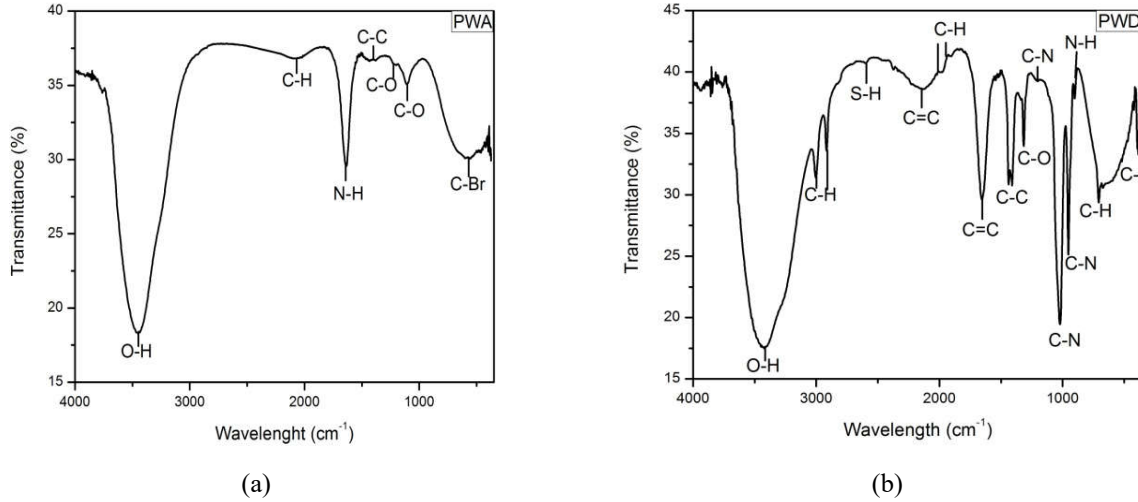


FIGURE 4. (a) FTIR Spectra of PWA and (b) FTIR Spectra PWD

FTIR testing was carried out to determine the functional groups in this work. Figure 5 (a) shows FTIR spectrum of PBA which explained the presence of functional group bonds namely O-H, C=C, C-C, C-H, C-O, C-N, N-H and C-I. Fig. 5 (b) shows the infrared spectrum of PBD. The functional group bonds formed from the PBD are O-H, C-H, O-H, -C=C-, C-H, C=C, C-C, C-N, N-H and C-I.

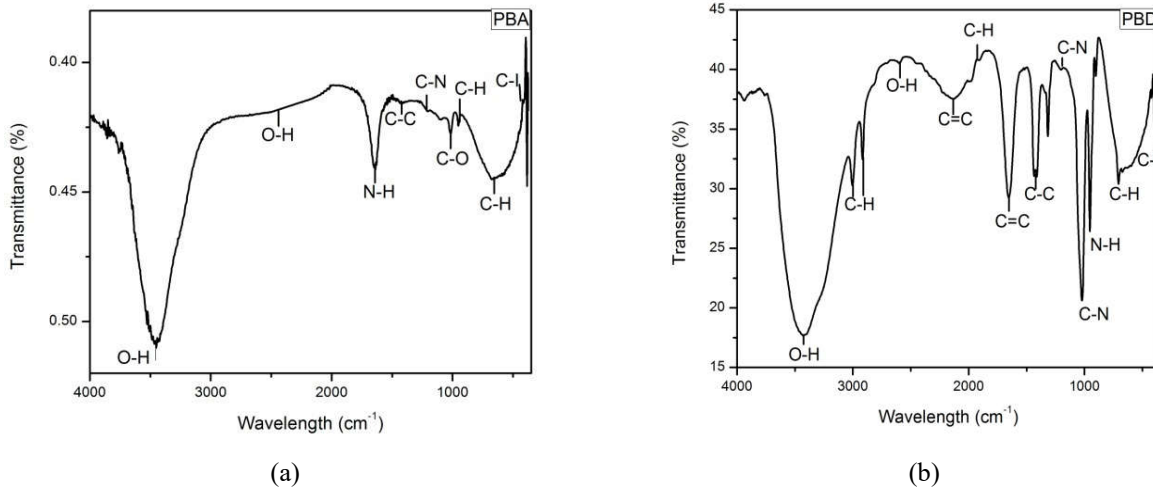


FIGURE 5. (a) FTIR Spectra of PBA and (b) FTIR Spectra of PBD

The data above shows that the use of aquades and DMSO solutions affects the functional group bonds formed in the sample. The functional group bonds formed in the sample using DMSO were greater than those with aquades. This is because of the bond in the solvent itself. The use of different temperatures also affects molecular bonds. The use of higher temperatures results in the breakdown of several molecular bonds.

SUMMARY

The carbon produced from carburizing of palmyra wine and palmyra brown sugar at 400 °C and 600 °C formed an amorphous carbon phase and the presence of impurities in the form of KCl. The morphology characterization of a thin layer of amorphous carbon from the PWA sample in the form of elongated lump and PBD sample in the nodular structure was agglomerated so that grain boundaries were not clearly visible between one another. Aquades and DMSO solutions affect the functional group bonds formed in carbon solution samples. Carbon solutions with DMSO solvents have more functional group bonds than those using aquades.

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