Study of the Formation of Amorphous Carbon and rGO-like Phases from Palmyra Sugar by Variation of Calcination Temperature

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Abstract: We have processed biomass from palmyra sugar to produce allotrope carbon by heating process with the variation of calcination temperature. The formation of amorphous carbon (a-C) was confirmed from the XRD result heated at 400\(^\circ\)C with the observation of the peak at the position of 24\(^\circ\). By increasing the temperature at 700\(^\circ\)C, the presence of two peaks at 24\(^\circ\) and 43\(^\circ\) were observed, indicating the formation of rGO-like phase. The functional groups detected by FTIR spectra consist of C=C, C-O, C=O, C-H and O-H. The conductivity measurement confirmed that the conductivity for a-C and rGO samples at room temperature are 4.50 S/m and 6.53 S/m, respectively. The result of conductivity measurement exhibits that the material can be classified as semiconducting materials.

Keywords: amorphous carbon; heating temperature; palmyra sugar; rGO.

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I. INTRODUCTION

The study of carbon becomes attractive since the nobel prize award on graphene material in 2010 received by two scientists, Andre Geim and Konstantin Novoselov [1]. Carbon is one of the elements that is most widely studied and applied in various fields such as: supercapacitors, batteries, fuel cell electrodes and adsorbent materials [2,3]. Carbon has two main allotropes: crystalline (diamond, graphite and fullerene) and amorphous carbon [4]. Graphite consists of thin sheets of molecular bonding between carbon atom called graphene. As one of graphene derivatives, the graphene oxide (GO) consists of oxygen (O) and hydrogen atoms (H) which are bonded to carbon atoms in the hexagonal structure as shown in Fig. 1. The reduced graphene oxide is the reduction of graphene oxide that experiencing the loss of oxygen and hydrogen atoms [5]. Diamond and graphite, which are abundant allotropes of carbon, are diamagnetic materials owing to their orbital diamagnetism [6]. Amorphous carbon (a-C) and reduced graphene oxide (rGO) contain mixtures of sp\(^2\) (graphitic) and sp\(^3\) (diamond) hybrids. They show semiconducting behavior and conduct electricity which can be used in some applications including its function for increasing the conductivity in secondary ion batteries, such as: Li- and Na-ion batteries [7,8].

Coal based activated carbon products are commonly used across a large number of industries for a variety of applications. However, in order to make green alternatives to replace coal as a source of carbon, a carbon-based biomass is now being extensively studied. Carbon can be produced from heating of organic materials (waste recycling into advance products).

The production of allotropes carbon such as a-C and rGO using biomass has been studied in the past [9-14]. The formation of a-C and rGO are found by heating the coconut shell by variation of temperature [11,12]. In this study, we use another type of biomasses named palmyra sugar to produce a-C and rGO-like materials which has not been studied in the past. Indonesia becomes one of countries that has a large source of palmyra sugar [15,16]. The heating treatment with the variation of calcination temperature was used in this study to obtain allotropes carbon from biomass.

II. METHODOLOGY

The starting material was palmyra sugar from Lamongan, East Java, Indonesia. The palmyra sugar was heated at a temperature of 250\(^\circ\)C at the ambient pressure for 2 hours to eliminate water contents. After obtaining the precursor, the sample was then calcinated at 400\(^\circ\)C henceforth called as “sample 1”. The other sample was obtained by calcinating the precursor at higher temperature of 700\(^\circ\)C (called as "sample 2"). The products of calcinated samples were characterized by some experimental techniques. The thermogravimetric analysis and differential scanning calorimetry (TGA/DSC) were carried out to determine the decomposition temperatures. To analyze the composition of phases, the samples were characterized by the XRD (Philips XPert Multi Purpose Diffractometer) using CuK\(_\alpha\) radiation (\(\lambda = 0.154056\) nm) at the angle of 5-60\(^\circ\). Functional groups presented in the samples were identified by the Fourier Transform Infrared measurement (8400S Shimadzu FTIR) in the range of 4000-500 cm\(^{-1}\). Conductivity measurement using four-point probe apparatus was also
FIG. 1: The chemical route to the reduced graphene oxide (rGO) [5].

Fig. 2 displays TGA/DSC of the precursor from palmyma sugar, which is useful to determine the decomposition temperatures. The TGA/DSC curve of heated palmyma sugar shows thermal degradation at three stages. The weight loss in the first stage is about 3%, within the temperature range 150°C to 300°C, coincidence with the endothermic reaction observed by DSC curve. The endothermic reaction at this stage can be attributed to the removal of adsorbed water. The second stage at 300°C to 500°C with 46% weight losses, followed by the third stage from 500°C to 876°C is observed. At this temperature range, the weight losses are attributed to the thermal degradation of hemicellulose and cellulose, forming a carbonaceous residue (rGO-like phase) from above 500°C.

The formation of amorphous carbon (a-C) is confirmed from the XRD result of sample 1 with the observation of the peak at the position of ~24° as shown in Fig. 3. A broadening XRD pattern is an indication of amorphous state of this sample. By increasing the temperature at 700°C, the presence of two peaks at 24° and 43° were observed in sample 2, indicating the formation of rGO-like phase. The XRD pattern of a-C and rGO-like phase obtained in this result are similar to that of reported in Ref. [17-20]. As mentioned in Ref. [20], the broad peak of ~24° is (002) plane which can be attributed to the amorphous carbon structure. The weak and broad diffraction peak of 43° can be attributed to (101) or (102) plane of rGO structure [20,21].

As shown in Fig. 4, the functional groups detected by the FTIR spectra are consisted of C=C, C-O, C=O, C-H and O-H. The C-H stretch is observed at 870 cm⁻¹, 1440 cm⁻¹ and 2917 cm⁻¹. A broad OH vibration peak is observed at 3436 cm⁻¹ which is similar to that of in Ref. [19], displaying that absorption bands in the range between 3373-3546 cm⁻¹ are associated to the symmetrical stretching of hydroxyl groups (OH). Other transmittance peaks in the FTIR spectrum are observed at 1157 cm⁻¹ (CO stretching vibrations), at 1689 cm⁻¹ (CO stretching vibrations) and at 1582 cm⁻¹ (CC stretching vibrations). The C≡C are attributed to sp² config-
FIG. 4: FTIR results of the powders from sample 1 (400°C) and sample 2 (700°C).

TABLE I: Comparison of conductivity in various allotrope carbon in different temperature.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Conductivity (S/m)</th>
<th>Temperature (K)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>4.50</td>
<td>300</td>
<td>Present work</td>
</tr>
<tr>
<td>Sample 2</td>
<td>6.53</td>
<td>300</td>
<td>Present work</td>
</tr>
<tr>
<td>rGO film</td>
<td>$6.3 \times 10^5$</td>
<td>300</td>
<td>[23]</td>
</tr>
<tr>
<td>GO film</td>
<td>$5.4 \times 10^5$</td>
<td>2</td>
<td>[23]</td>
</tr>
<tr>
<td>rGO nanosheets</td>
<td>$5.32 \times 10^2$</td>
<td>230</td>
<td>[25]</td>
</tr>
<tr>
<td>a-C: H</td>
<td>0.3</td>
<td>348</td>
<td>[26]</td>
</tr>
<tr>
<td>a-C thin films</td>
<td>$1.88 \times 10^{-2}$</td>
<td>300</td>
<td>[27]</td>
</tr>
</tbody>
</table>

IV. SUMMARY

We have processed biomass from palm sugar to produce allotrope carbon by heating process with the variation of calcination temperature. The formation of amorphous carbon (a-C) was confirmed from the XRD result heated at 400°C with the observation of the peak at the position of 24°. By increasing the temperature at 700°C, the presence of two peaks at 24° and 43° were observed, indicating the formation of rGO-like phase. The functional groups detected by FTIR spectra consist of C=C, C-O, C=O, C-H and O-H. The conductivity measurement confirmed that the conductivity of a-C and rGO samples at room temperature are 4.50 S/m and 6.53 S/m, respectively. The result of conductivity measurement exhibits that the material can be classified as semiconducting materials. This report is able to show the alternative to produce allotrope carbon from biomass.

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