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A Study on Phase and Microstructure of Reduced Graphene Oxide Prepared by Heating Corncobs

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Abstract
The purpose of this study was to find out reduced graphene oxide (RGO) phases by identifying the system, elemental system, and microstructure of corncobs. Characterization was carried out by XRD and SEM-EDX spectroscopy. The temperatures used in this work are 100, 200, and 250 ºC with a holding time of 1 hour. XRD analysis shows that the RGO phase is formed by the structure of the aromatic layer arrangement (graphite 002), and the widening comes from small-dimensional crystallites perpendicular to the aromatic layer (graphite 120). The elements contained in the three samples have a dominant proportion in the elements carbon and oxygen. From the SEM results obtained the morphology of corncobs powder that looks like sheets. The reduced graphene oxide phase is formed from the process of reducing corncobs powder.
Keywords: Phase structure, micro structure, RGO, corncob.

1. Introduction

Graphite is a type of carbon material formed from carbon atoms in the sp2 orbital. Microscopically, graphite consists of thin sheets resulting from bonds between carbon atoms called graphene [1]. Graphene is a developing material with a honeycomb-like two-dimensional crystal structure formed by carbon atoms at the sp2 hybrid junction, exhibiting unmatched electronic, thermal, and mechanical properties [2]. The most important problem for realizing graphene's potential is to achieve mass and controlled production [3]. There are various names for graphene, such as chemically modified graphene, functionalized graphene, chemically altered graphene, or simply graphene [4], [5].

Reduced graphene oxide (RGO) is a promising material for many applications such as in the development of energy storage capacitors [6], field-effect transistor sensors [7]. Materials that can be used as a basis for the manufacture of carbon materials are corncobs and other organic materials containing cellulose, hemicellulose, and lignin because most of them are composed of the element carbon [8]. As one of the graphene derivatives, graphene oxide consists of oxygen (O) and hydrogen (H) atoms bonded to carbon atoms in a hexagonal structure [9]. RGO is a reduction in graphene oxide which loses oxygen and hydrogen atoms so that it becomes a graphene structure.

Indonesia as an archipelago with a tropical climate has abundant natural resources in the form of corncobscorncobs. However, the use of corncobs as raw material is still not optimal and there is still huge potential, so a solution is needed to utilize and increase the economic value of corncobs. The results of the chemical analysis of corncobs contained 30.91% hemicellulose, alpha-cellulose 26.81%, lignin 15.52%, carbon 39.80%, nitrogen 2.12%, and water content of 8.38% [10]. Previous research has revealed that corncobs were widely used as activated charcoal [11] and it has not yet been developed into graphene. Thus, it is necessary to carry out further studies on the phases and microstructure of corncobs that have undergone a heating process to study the graphene oxide content.

2. Method

2.1. Preparation of Corn Cob Powder

The materials used in this study were corncob powder and distilled water. The equipment used in this research is a mortar, the crucible, oven, blender, beaker glass, digital scale, cutter, spatula, and aluminum foil.

2.2. Characterization of Samples

Sample preparation was done by cleaning the corncobs. It was then crushed using a powder grinder and using a mortar then sieved using a 200 mesh sieve. Corncobs powder was weighed per 5 grams which was placed into the crucible lid. It was then heated using an oven at a temperature of 100, 200, and 250 ºC for 1 hour in free air. Samples were characterized using X-Ray Diffraction (XRD) to identify the sample phase. SEM-EDX found out the elements contained and the microstructure of corncobs powder after treatment with variations in heating temperature.

3. Results and Discussion

3.1. Analysis of X-Ray Diffraction

XRD measurements were carried out with a small angle between 2θ = 10º–90º. XRD data were obtained using CuKα λ = 1.54056 Å radiation with step data of 0.02º. The XRD pattern described in Figure 1, shows that the RGO phase is formed by a turbostatic structure which is a random stack of parallel layers that compose a graphite structure with a cliftonite phase at temperatures of 100, 200, and 250 ºC. The diffraction pattern shows that it consists of two wide peaks located approximately at a position angle between 2θ = 15º and 30º, indicating the formation of an RGO phase with a mountain-like pattern with plane reflections (002). From search Match identification, the XRD pattern of corncobs powder at a heating temperature of 100 ºC refers to the structure of the aromatic layer arrangement (graphite 002), and the widening comes from small-dimensional crystallites perpendicular to the aromatic layer (graphite 120) [12]. From the results of the tests that have been carried out, it is obtained a graph of the diffraction pattern between intensity and 2θ. The peak of RGO refers to the diffraction pattern where this phase is included in the carbon-graphite type which has a hexagonal crystal system with a space group of R-3m. Corncobs fine powder heating to a temperature of 100, 200, 250 ºC with a holding time of one hour has an interesting characteristic, namely, it has an amorphous peak at an angle of 15º–30º where the peak formed at an angle of 15º is a graphene oxide phase [13], while the peak which is formed in the sample is at an angular position of the range 20º–25º which is RGO [14].

The amorphous peaks that are formed are quite interestingbecause the particles are not normally distributed at low-temperature heating and the holding time is quite short so that the amorphous peaks appear to have fractured when heading towards the 20º angle position [15]. The structure formed in the XRD test is a RGO phase. This is based on the results of tests conducted using EDX. EDX testing on corncobs powder that has been heated at a temperature of 100, 200, and 250 ºC for one hour to determine the dominant elements that are still contained in the sample and determine the percentage of carbon elements as a reference in determining the phase formed. Based on the EDX test results, the dominant elements in the carbonized sample are carbon and oxygen

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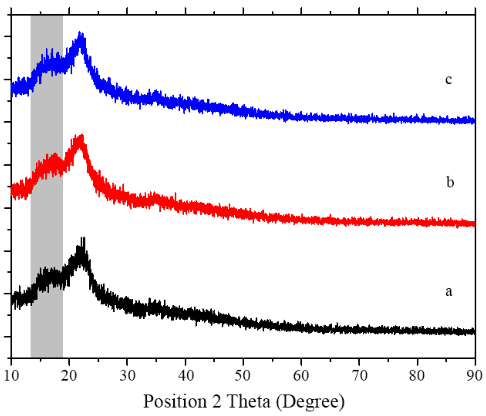


Figure 1. XRD of corncob powder heating at (a) 100, (b) 200, and (c) 250 ºC.

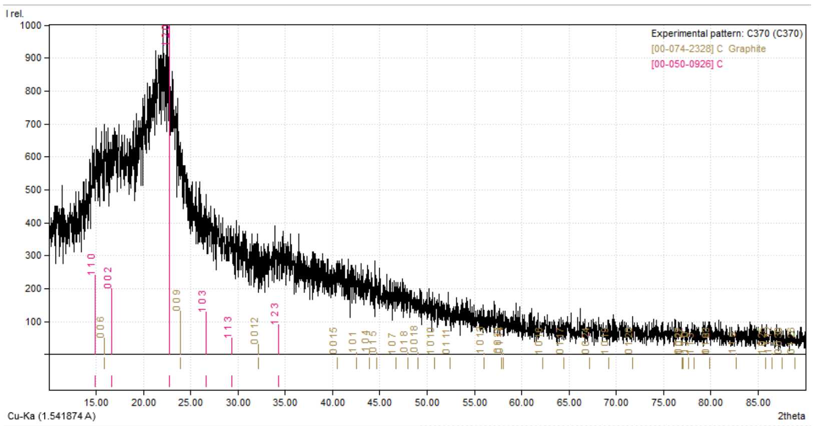


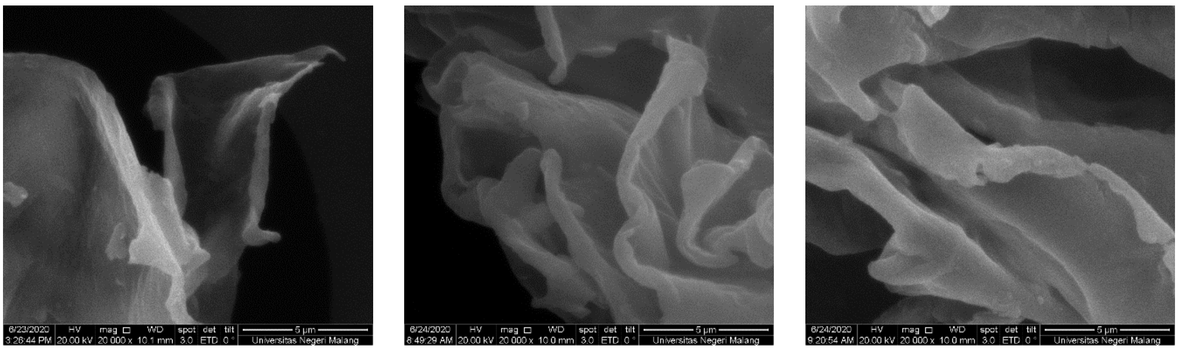
Figure 2. Phase identification of corn cob powder heating at 100 ºC using Match software.

Corncobs are an organic material from the nature which has different elements depending on environmental conditions. So it is necessary to do an EDX analysis to determine the elements found in corncobs powder. The results of the corncob EDX test shown in Table 1. The EDX test provides information that the percentage of the element carbon (C) is the highest with a mass percentage of 59.10% and an atomic percentage of 65.81%. And in the three samples were not found other elements. So that the EDX test results can be used as the basis for determining the phase formed in the XRD pattern [16].

SEM testing on corncobs heated at 100, 200, and 250 ºC for one hour. From the SEM results, the morphology of corncobs powder looks like sheets (shown in Figure 3). As can be seen, RGO features a smooth surface and a hierarchical structure consisting of micro-sized sheets [17], [18]. Samples that have been carbonized at 200 ºC look like stacked RGO sheets, which means that RGO starts to distribute evenly due to the increase in temperature (Figure 3.b). Meanwhile, the material which is carbonized at 250 ºC seems to be forming a wrinkled cloth. From the three SEM images showing a surface in the form of a sheet in micrometer size, which means that corncobs powder on heating temperature 100, 200, 250 ºC has formed an RGO phase structure [19], [20].

Table 1. EDX test results corn cob powder heating for one hour.

Element	Temperature 100 ºC		Temperature 200 ºC		Temperature 300 ºC	
	Wt (%)	At (%)	Wt (%)	At (%)	Wt (%)	At (%)
CK	58.31	58.31	58.83	65.56	59.10	65.81
OK	41.69	41.69	41.17	34.44	40.90	34.19



(a) (b) (c)

Figure 3. SEM results of corncob powder heating at (a) 100, (b) 200, and (c) 250 ºC.

4. Conclusion

The reduced graphene oxide (RGO) phase is formed by heating the corn cob powder at a temperature of 100, 200, and 250 ºC for one hour. Based on the XRD results, it is known that the phase formed is the RGO phase at an angle of $2\theta = 15º–30º$. This is also supported by the elemental content in the sample based on the results of the EDX test, which are elements of carbon and oxygen. Based on the results of the SEM test, all the samples produced to show the sheet morphology in microstructure size, which means that corn cob powder on heating 100, 200, 250 ºC has formed an RGO phase structure.

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