



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

K W Mas'udah^{1*}, A taufiq², and Sunaryono²

- ¹ Department of Mathematics, Faculty of Science and Technology, Universitas Pesantren Tinggi Darul Ulum, Jl. Rejoso, 61481, Jombang, Indonesia.
- ² Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Malang, Jl. Semarang 5, Malang, 65145, Indonesia.

*E-mail: kusumawardhani.m@mipa.unipdu.ac.id

Received
13 September 2020

Revised
19 October 2020

Accepted for Publication
21 October 2020

Published
23 October 2020



This work is licensed under a [Creative Commons Attribution-ShareAlike 4.0 International License](https://creativecommons.org/licenses/by-sa/4.0/)

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 °C 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 corncobs. 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 °C 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 $2\theta = 10^\circ$ – 90° . XRD data were obtained using $\text{CuK}\alpha \lambda = 1.54056 \text{ \AA}$ radiation with step data of 0.02° . The XRD pattern described in Figure 1, shows that the RGO phase is formed by a turbostratic 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 °C. The diffraction pattern shows that it consists of two wide peaks located approximately at a position angle between $2\theta = 15^\circ$ and 30° , 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 °C 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 °C with a holding time of one hour has an interesting characteristic, namely, it has an amorphous peak at an angle of 15° – 30° where the peak formed at an angle of 15° is a graphene oxide phase [13], while the peak which is formed in the sample is at an angular position of the range 20° – 25° which is RGO [14].

The amorphous peaks that are formed are quite interesting because 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 20° 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 °C 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

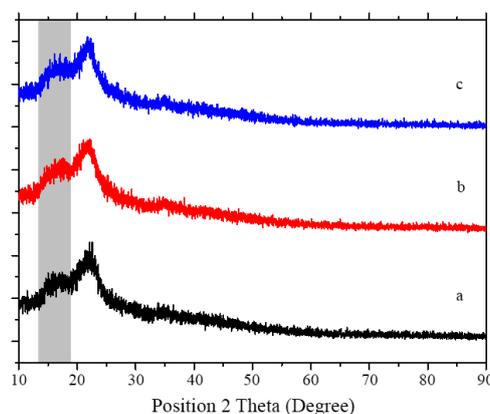


Figure 1. XRD of corncob powder heating at (a) 100, (b) 200, and (c) 250 °C.

4. Conclusion

The reduced graphene oxide (RGO) phase is formed by heating the corn cob powder at a temperature of 100, 200, and 250 °C for one hour. Based on the XRD results, it is known that the phase formed is the RGO phase at an angle of $2\theta = 15^\circ\text{--}30^\circ$. 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 °C has formed an RGO phase structure.

Acknowledgement

The authors are grateful to the KemenRistek Dikti research funding institution for the PKPT (Higher Education Collaboration Research) grant scheme for the 2019 and 2020 fiscal years that have supported this research. Besides, the authors would like to express their deepest gratitude to the Department of Physics, Faculty of Mathematics and Natural Sciences (Universitas Negeri Malang) for collaborating in the use of corncobs into graphene film as a semiconductor material.

References

- [1] Q. Jiang, Z. Zhang, S. Yin, Z. Guo, S. Wang, and C. Feng, "Biomass carbon micro/nano-structures derived from ramie fibers and corncobs as anode materials for lithium-ion and sodium-ion batteries," *Appl. Surf. Sci.*, vol. 379, pp. 73–82, 2016.
- [2] B. Mortazavi, M. E. Madjet, M. Shahrokhi, S. Ahzi, X. Zhuang, and T. Rabczuk, "Nanoporous graphene: a 2D semiconductor with anisotropic mechanical, optical and thermal conduction properties," *Carbon N. Y.*, vol. 147, pp. 377–384, 2019.
- [3] Y. Liu, X. Ge, and J. Li, "Graphene lubrication," *Appl. Mater. Today*, vol. 20, p. 100662, 2020.
- [4] N. R. Chodankar, A. K. Nanjundan, D. Losic, D. P. Dubal, and J. B. Baek, "Graphene and molybdenum disulphide hybrids for energy applications: an update," *Mater. Today Adv.*, vol. 6, p. 100053, 2020.
- [5] D. Zhang, J. Tong, and B. Xia, "Humidity-sensing properties of chemically reduced graphene oxide/polymer nanocomposite film sensor based on layer-by-layer nano self-assembly," *Sensors Actuators, B Chem.*, vol. 197, pp. 66–72, 2014.
- [6] G. B. A. Putra, H. Y. Pradana, D. E. T. Soenaryo, M. A. Baqiya, and Darminto, "Synthesis of green Fe³⁺/glucose/rGO electrode for supercapacitor application assisted by chemical exfoliation process from burning coconut shell," *AIP Conf. Proc.*, vol. 1945, no. 1, p. 020040, 2018.
- [7] S. W. Howell *et al.*, "Graphene-insulator-semiconductor junction for hybrid photodetection modalities," *Sci. Rep.*, vol. 7, no. 1, pp. 1–9, 2017.
- [8] K. W. Mas'udah, P. E. Yuwita, A. Taufiq, and Sunaryono, "Fabrication of nanocrystalline carbon based on corncobs charcoal," *AIP Conf. Proc.*, vol. 2231, no. 1, p. 040020, 2020.
- [9] P. E. Yuwita, K. Wardhani, Sunaryono, and A. Taufiq "Investigation of carbon phase structure of corncob charcoal powder investigation of carbon phase structure of corncob charcoal powder," *AIP Conf. Proc.*, vol. 2231, no. 1, p. 040083, 2020.
- [10] K. W. Mas'udah, P. E. Yuwita, Y. A. Haryanto, A. Taufiq, and Sunaryono, "Effect of heat treatment on carbon characteristic from corncob powders prepared by coprecipitation method," in *AIP Conf. Proc.*, vol. 2251, no. 1, p. 040044, 2020.
- [11] S. Saputro, M. Masykuri, L. Mahardiani, and D. Kurniastuti, "The synthesis of corncobs (zea mays) active charcoal and water hyacinth (eichornia crassipes) adsorbent to adsorb Pb(II) with it's analysis using solid-phase spectrophotometry (sps)," *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 333, no. 1, 2018.
- [12] Y. Liu, X. Zhao, J. Li, D. Ma, and R. Han, "Characterization of bio-char from pyrolysis of wheat straw and its evaluation on methylene blue adsorption," *Desalin. Water Treat.*, vol. 46, no. 1–3, pp. 115–123, 2012.
- [13] A. Y. Nugraheni, M. Nashrullah, and F. A. Prasetya, "Study on phase, molecular bonding, and bandgap of reduced graphene oxide prepared by heating coconut shell," vol. 827, no. 1, pp. 285–289, 2015.

- [14] I. Khambali, I. S. Ardiani, A. R. Kurniawan, Triwikantoro, M. Zainuri, and Darminto, “Synthesis of n-doped reduced graphene oxide from coconut shell as supercapacitors,” *Mater. Sci. Forum*, vol. 966, pp. 437–443, 2019.
- [15] H. Ma, J. Li, W. Liu, M. Miao, B. Cheng, and S. Zhu, “Bioresource technology novel synthesis of a versatile magnetic adsorbent derived from corncob for dye removal,” *Bioresour. Technol.*, vol. 190, pp. 13–20, 2015.
- [16] K. W. Mas’udah, F. Astuti, and D. Darminto, “Study on physical properties of reduced graphene oxide from heating coconut shell,” *JPSE (Journal Phys. Sci. Eng.)*, vol. 1, no. 1, pp. 1–6, 2016.
- [17] Y. Zhang, M. M. Umair, S. Zhang, and B. Tang, “Phase change materials for electron-triggered energy conversion and storage: a review,” *Journal of Materials Chemistry A*, vol. 7, no. 39, pp. 22218–22228, 2019.
- [18] Y. Li *et al.*, “Graphene-CoO/PEG composite phase change materials with enhanced solar-to-thermal energy conversion and storage capacity,” *Compos. Sci. Technol.*, vol. 195, p. 108197, 2020.
- [19] A. S. K. Kumar, S. S. Kakan, and N. Rajesh, “A novel amine impregnated graphene oxide adsorbent for the removal of hexavalent chromium,” *Chem. Eng. J.*, vol. 230, pp. 328–337, 2013.
- [20] A. S. K. Kumar and N. Rajesh, “Exploring the interesting interaction between graphene oxide, Aliquat-336 (a room temperature ionic liquid) and chromium (VI) for wastewater treatment,” *Rsc. Advances*, vol. 3, no. 8, pp. 2697–2709, 2013.