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# Graphene Oxide Production Via Exfoliation Process of Low-Temperature Synthetic Graphite from Oil Palm Trunk Waste

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The exfoliation process via the modified Hummers' method has been well established as the main method to produce graphene oxide. Previously, however, the process only involved commercial graphite as a precursor material for the production of graphene oxide. In this study, synthetic graphite has been successfully produced using oil palm trunk waste as potential carbon sources. Different values of the heating parameter, i.e, 500, 800, and 1000°C, have been applied. The heating also varied from 5 to 10°/min and to 20°/min to control the heating condition. After heating treatment, the samples were characterised using X-ray diffraction and analysed by X'Pert HighScore Plus software. The graphite nature of the synthetic graphite produced was additionally supported by Raman analysis. Morphological study was carried out using a scanning electron microscope. Based on the analysis, the optimum processing parameters were found, namely the temperature of 800°C and the heating rate of 20°/min. The as-produced synthetic graphite was then subjected to further exfoliation to form graphene oxide via the modification of Hummers' method. The graphene oxide produced was then characterised to confirm its graphitic nature.

topics: synthetic graphite, low temperature, oil palm trunk waste, graphene oxide

## 1. Introduction

The modified Hummers' method is a chemical process that can be used to generate graphite oxide. Previously, the preparation of synthetic graphite always required temperatures above 2500°C, which means a high energy cost [1].

In this project, it was possible to produce synthetic graphite at a much lower heating temperature, which was less than 1000°C at 3 various soaking times and heating rates, using oil palm trunk waste as a starting material. The synthetic graphite used in this paper as a precursor material for the production of graphene oxide is different from the commercially available synthetic graphite that was previously made.

The utilisation of oil palm trunk waste as a primary source for the production of synthetic graphite begins only when issues with the disposal and handling of the oil palm trunk waste arise.

## 2. Experimental details

### 2.1. Graphitisation of the oil palm trunk waste and graphene oxide exfoliation via the modification of Hummers' method

Heat treatment takes place with various heating parameters. After heat treatment, the samples will be characterised using different characterization tools and the best synthetic graphite from the best parameter will be further exfoliated to produce graphene oxide.

The first step in the modified Hummers' method was to produce pre-oxidised graphite and use all chemicals [2]. The obtained pre-oxidised graphite was then oxidised to produce graphene oxide [3]. Along with the amount of chemicals used, the density of the produced synthetic graphite was prone to change.

### 2.2. Characterizations

The phase analysis of the samples was characterised by X-ray powder diffraction (XRD) method. The diffractogram of each sample was analysed using X'pert HighScore Plus. From this software, the diffraction pattern of the sample can be matched to the diffraction pattern of reference [4]. Crystallite size can also be determined using this software [5]. Apart from XRD, each sample was also characterised by Raman spectroscopy to observe the graphitic nature of the synthetic graphite produced.

## 3. Results

### 3.1. Analysis of synthetic graphite from oil palm trunk waste

#### *3.1.1. Analysis of X-Ray diffraction*

The diffraction pattern was characterised by XRD in the $2\theta$ range of 10–90°. Figure 1 shows the XRD diffraction pattern at the heating temperature of 800°C at 3 various heating rates.

The reference code that matches the graphite is 00-041-1487 [6]. The diffraction pattern in Fig. 1 at the heating rate of 20°/min matches the reference code 00-041-1487 and is also comparable with the commercial graphite.

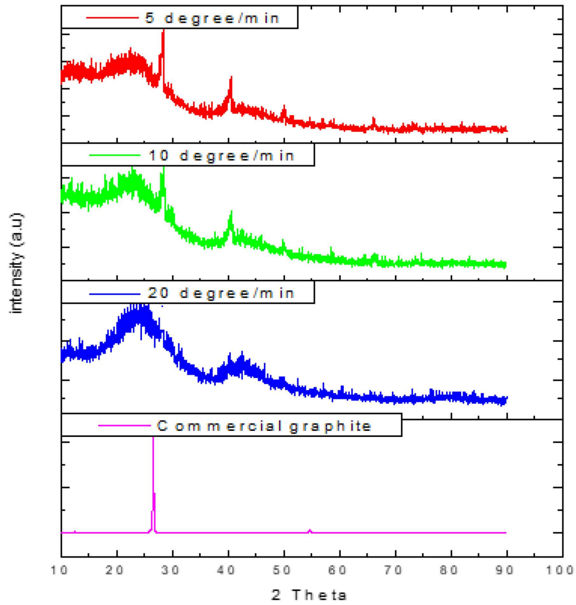


Fig. 1. Sample heated at 800°C at various heating rates.

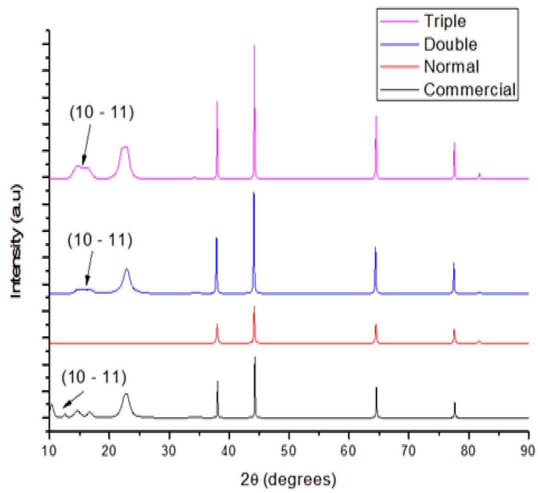


Fig. 2. XRD analysis of graphene oxide.

### 3.2. Analysis of graphene oxide from modification of Hummers' method

#### *3.2.1. Analysis of X-Ray diffraction (XRD)*

XRD analysis of graphene oxide was analysed using X'Pert HighScore Plus software and the diffraction pattern is shown in Fig. 2. The diffractogram was analysed at a scan rate from 10 to 90°. From the diffraction pattern, the graphene oxide peak was found to be in the $2\theta$ range from 10 to 11°. This confirmed the presence of graphene oxide in the sample.

The peak of graphene oxide present in the diffraction pattern indicates a successful exfoliation process via modifying the Hummers' method [7]. Hence, it is strongly stated that graphene oxide was produced during the chemical exfoliation process.

#### *3.2.2. Analysis of atomic force microscope (AFM)*

AFM is a high-resolution imaging technique, in which a small probe with a sharp tip is scanned back and forth in a controlled manner across a sample to measure surface topography up to atomic resolution [8]. The AFM can detect a single layer of graphene oxide together with its thickness.

Figure 3 shows the four images analysed at 5$\mu$m. It can be seen that a single layer of graphene oxide was found, which confirms the presence of

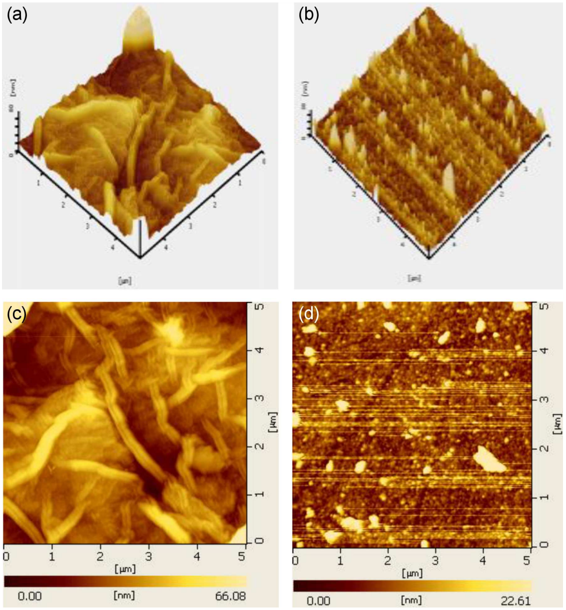

Fig. 3. The images of AFM analysis of graphene oxide for triple parameter.

graphene oxide. This fact confirms the success of the exfoliation process via modification of Hummers' method [9].

## 4. Discussion

Graphitisation is defined as the occurrence of limited movement and rearrangement of carbon atoms undergoing a reconstructive transformation during the heat treatment process. Suitable soaking times applied also support the graphitisation process. This is because the atoms in the carbon phase have enough time to rearrange during graphitisation to form a turbostratic structure. Synthetic graphite is considered to be successfully formed when the reference code in XRD analysis matches the reference code 00-041-1487, indicating the formation of the graphitic phase.

## 5. Conclusions

This study was successfully carried out to synthesize synthetic graphite from oil palm trunk waste at lower heating temperature with the controlled condition and optimized processing parameters. The obtained graphene oxide was further characterised using X-ray diffraction (XRD) and atomic force microscope (AFM). From XRD, a graphene oxide peak was observed from 10 to 11° in the $2\theta$ range for the sample of graphene oxide with triple amount of chemical being used. The analysis of FTIR showed that the functional groups present in the graphene oxide produced from the triple parameters were comparable to the functional groups present in the graphene oxide produced from commercial graphite. The AFM study showed that a single layer of graphene oxide was also present in the graphene oxide sample produced from triple parameters.

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## References

[1] N.A. Karim, C.M.R. Ghazali, M.M. Ramli, A.V. Sandu, E. Chirila, M.M.A Abdullah, *IOP Conf. Ser. Mater. Sci. Eng.* **572**, 012060 (2019).

[2] N.A. Karim, C.M.R. Ghazali, M.M. Ramli, D.S.C. Halin, I. Nainggolan, *AIP Conf. Proc.* **1885**, 020227 (2017).

[3] N.A. Karim, M.M. Ramli, C.M.R. Ghazali, C.A.I. Nadia, G. Denesh, *IOP Conf. Ser. Mater. Sci. Eng.* **932**, 012125 (2020).

[4] S. Radhakrishnan, Y. Devarajan, A. Mahalingam, B. Nagappan, *J. Oil Palm Res.* **29**, 380 (2017).

[5] S.S. Masura, N.I. Tahir, O.A. Rasid, U.S. Ramli, A. Othman, M.Y.A. Masani, G.K.A. Parveez, A. Kusairi, *J. Oil Palm Res.* **29**, 469 (2017).

[6] Kurniasari, A. Maulana, A.Y. Nugraheni, D.N. Jayanti, S. Mustofa, M.A. Baqiya, Darminto, *IOP Conf. Ser. Mater. Sci. Eng.* **196**, 012021 (2017).

[7] H. Okubo, C. Tadokoro, Y. Hirata, S. Sasaki, *Tribol. Online* **12**, 229 (2017).

[8] N.A. Karim, C.M.R. Ghazali, M.M. Ramli, D.S.C. Halin, I. Nainggolan, *AIP Conf. Proc.* **1885**, 020227 (2017).

[9] N.A. Karim, M.M. Ramli, C.M.R. Ghazali, M.N. Mohtar, *Mater. Today: Proc.* **16**, 2088 (2019).