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To cite this article: N A Karim *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **572** 012060

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# Optimization of processing parameters for graphitization of oil palm trunk waste at lower heating temperature

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**Abstract.** Previously synthetic graphite was produced at higher temperature which is around 2,500 °C in complex processing method. The method bear the name of the scientist whose discover the synthetic graphite namely “Acheson Process”. However, via pyrolysis process, in controlled heating condition, and specific heating rate by utilizing oil palm trunk waste, synthetic graphite was managed to produce at much lower heating temperature. In this research, the heating temperature were varied in the range of 500 °C, 800 °C, and 1,000 °C. The heating rate applied also varied, from 5 °/min to 10 °/min and 20 °/min. After heating treatment, the samples were characterized using X-ray Diffraction (XRD) and analysed by X’Pert Highscore Plus software. Graphitic nature of the synthetic graphite produced was further supported by RAMAN analysis. The morphological study was carried out by using Scanning Electron Microscope (SEM). Based on the analysis, optimum processing parameters was optimized. It is at the temperature of 800 °C and at the heating rate of 20 °/min.

## 1. Introduction

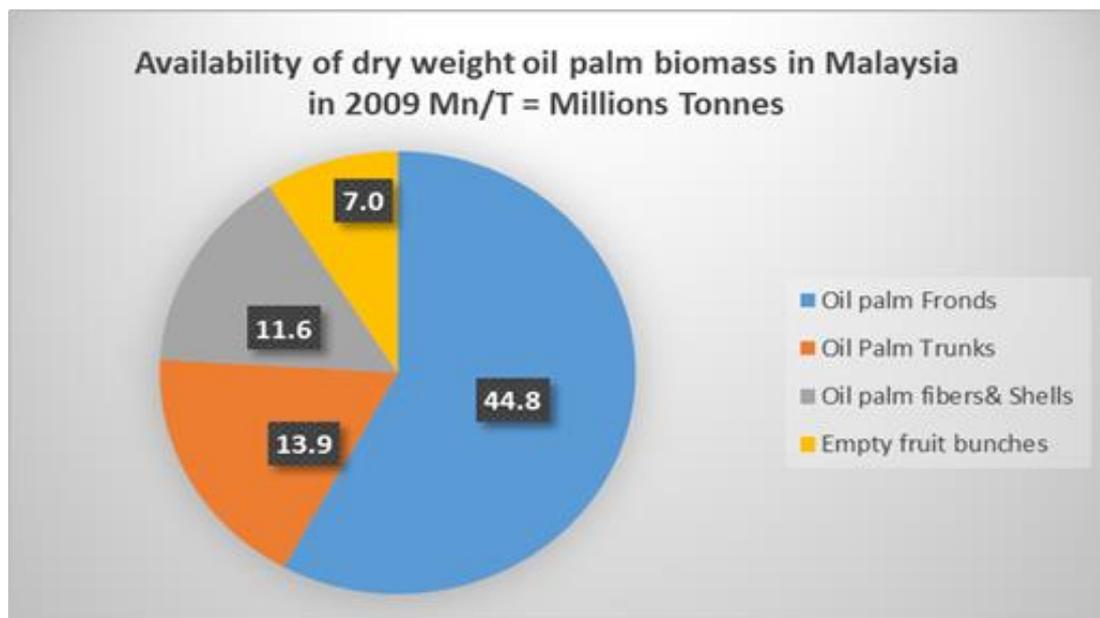
Graphite can be classified into natural graphite and synthetic graphite. Natural graphite is graphite that form by nature. In the other hand, synthetic graphite can be defined as the substance produced from high temperature processing of amorphous carbon materials. The carbon sources used as raw materials to produce synthetic graphite are various such as, petroleum, coal or natural or synthetic organic resources [1]. However, there also cases whereby graphite can even be produced directly from precipitation of graphitic carbon from pyrolysis of carbonaceous gas such as acetylene. The most important properties of graphite precursor is that they must contained carbon. Graphite is the most important type of allotrope or carbon and it can only be derived from other carbon containing substances[2]. Either it is synthetic graphite or natural graphite, it is the most important precursor for graphene material preparation by several chemical processes and exfoliation technique. Synthetic



graphite was discovered by accident by scientist name Edward Goodrich Acheson in 1800s. Acheson found that, an unintentional reaction product which is graphite crystals also formed when he was trying to produce carborundum in an electric furnace by the mixture of silica and amorphous carbon. High purity and crystalline of synthetic graphite can be obtained by refining process as well as elimination of silica. One of the furnace types still used to produce graphite, and the process method still bears Mr. Acheson's name: the Acheson furnace and the Acheson process [3].

The preparation of synthetic graphite requires temperatures above 2,500 °C which represents a high energy cost [4]. Various types of carbon allotropes exist which differ in term of atom arrangement and physical structures namely graphite, graphene, carbon nanotube and fullerenes. Graphene can be viewed as a honeycomb lattice, while graphite is a stack of graphene layers. Fullerene in the other hand is a rolled up cylinder of graphene layers.[5].

The utilization of oil palm trunk waste as the primary sources for synthetic graphite production begin when the issues of the disposal and handling of the oil palm trunk waste arise. Oil palm industries contributes 85.5 % of the total biomass production in Malaysia. The palm oil industry is one of Malaysia's major agricultural enterprises [6]. After the economical period of oil palm tree, which is after 25-30 years, the oil palm tree will be cut down for replantation. After replantation, several problem on the abundance of oil palm trunk waste arise. Oil palm trunk must be utilized significantly with respect to handling as to avoid the environmental issue. Among the generated biomass from oil palm industry, oil palm trunk (OPT) appears to be the second highest residues which amounted to be approximately 13.9 Mn/T as shown in Figure 1 below [8].



**Figure 1.** Availability of dry weight oil palm biomass in Malaysia in 2009 (Mn/ T¼Million Tonnes).

Since the oil palm industry contribute the highest biomass waste, the problem of tonnage of abundance oil palm trunk waste will give serious impact on environmental issue [9]. The oil palm trunk waste should be handle wisely in order to implement the waste to wealth concept. Since Malaysia Government launch the Government Economic Transformation Programme (ETP), the objective of ETP is to transform Malaysia into a high-income nation by year 2020. Via ETP, towards utilization and enhancement of oil palm waste, the attempt has been made as to utilize the oil palm trunk waste as potential carbon source for synthetic graphite production [10].

## 2. Experimental details

### 2.1. Graphitization of oil palm trunk waste

The preparation of the sample start with, collected of the oil palm trunk waste from the plantation and cut into chip in the dimensions of 5 cm x 2 cm x 2 cm becoming oil palm trunk chip. The drying process takes place in room temperature condition for several days. After drying process, the heating treatment was done in controlled heating condition with the present of nitrogen gas at three different heating rates which are, 5 °/min, 10 °/min 20 °/min at five different heating temperatures which are 500 °C, 800 °C, and 1,000 °C.

### 2.2. Characterizations

Phase analysis of the samples was characterized by X-Ray Powder Diffraction (XRD). XRD was used as analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The diffraction pattern from every sample was analyzed using X'pert Highscore Plus. From this software, the diffraction pattern from sample can be matched with diffraction pattern from reference. The crystallite size can also be determined from this software. The Scherrer equation describes the relation between the broadening of the reflection and the average crystallite size,  $\tau$  in Equation 1.

$$\tau = K\lambda / \beta_{\tau} \cos\Theta \quad (1)$$

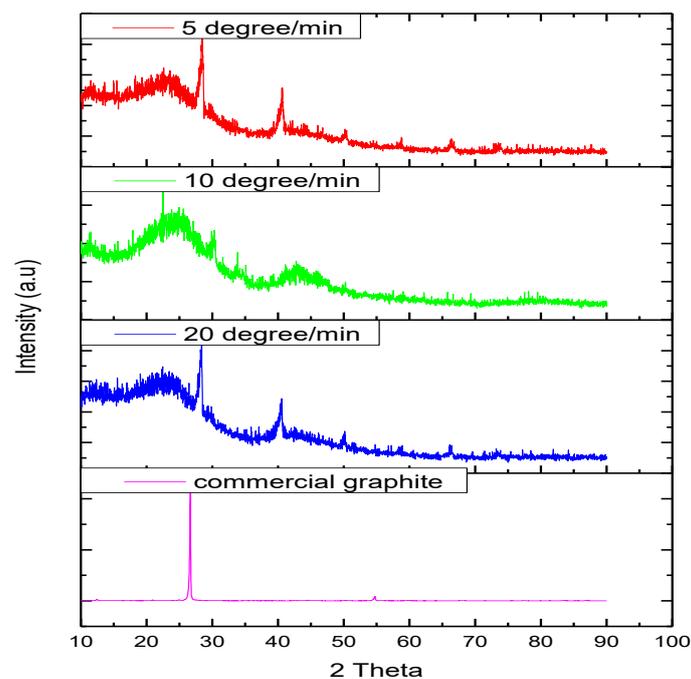
where  $\beta_{\tau}$  is Broadening of the reflection due to small crystallite sizes, with  $\beta_{\tau} = (B-b)$ ; B is FWHM of the reflection of the real sample and b is FWHM of the standard reflection (crystallite size is approximately 0.5 to 5  $\mu\text{m}$ ), K is approximately 0.9 (0.89 for spherical, 0.94 for cubic crystallite,  $\lambda$  is wavelength and  $\Theta$  is diffraction angle.

Apart from XRD, each of the sample also was characterized by RAMAN Spectroscopy to observe the graphitic nature of the synthetic graphite produced.

For morphological studies, all samples were characterized using Scanning Electron Microscope.

## 3. Results and discussions

### 3.1. Analysis of X-Ray Diffraction (XRD)



**Figure 2.** Sample heated at 500°C various heating rate.

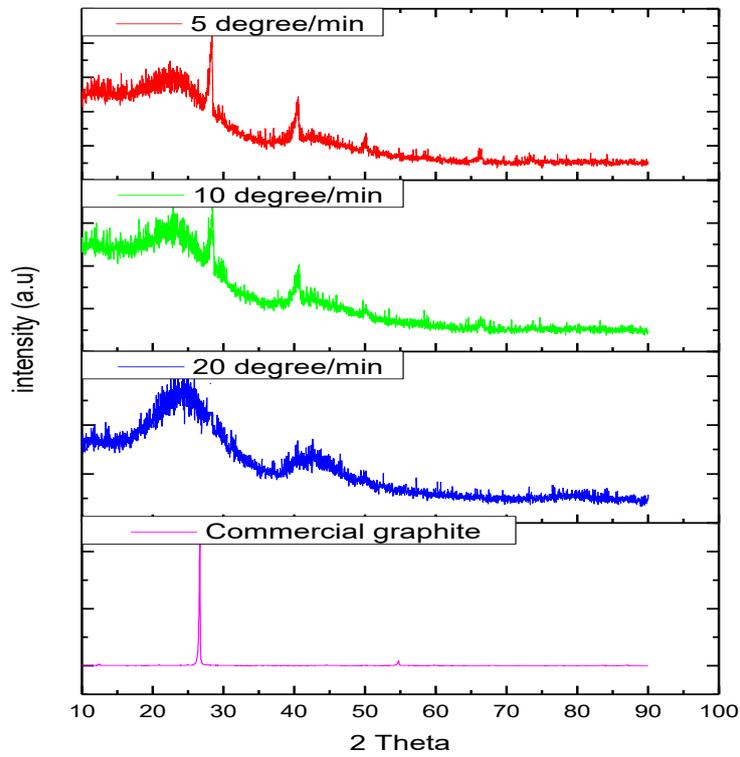


Figure 3. Sample heated at 800°C various heating rate.

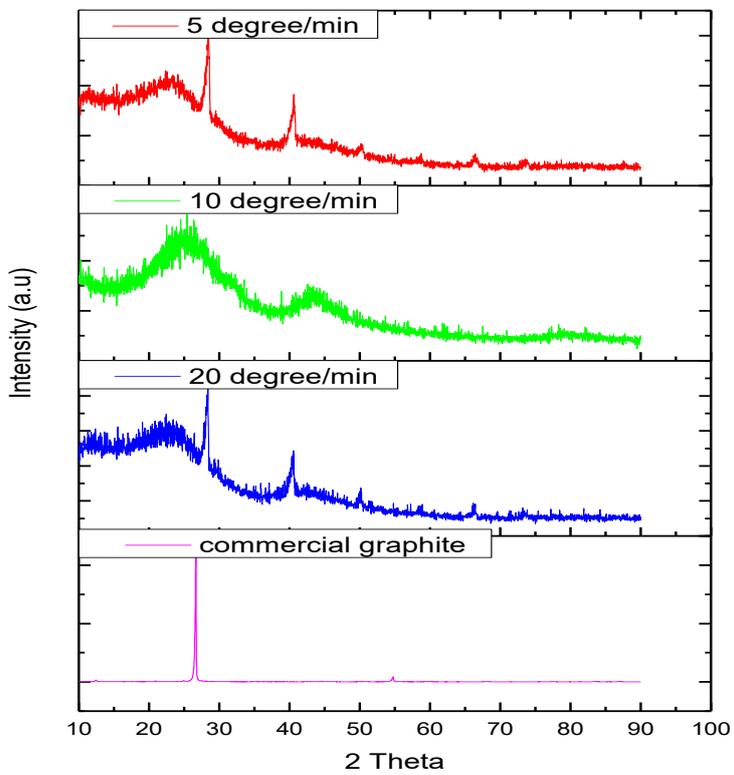
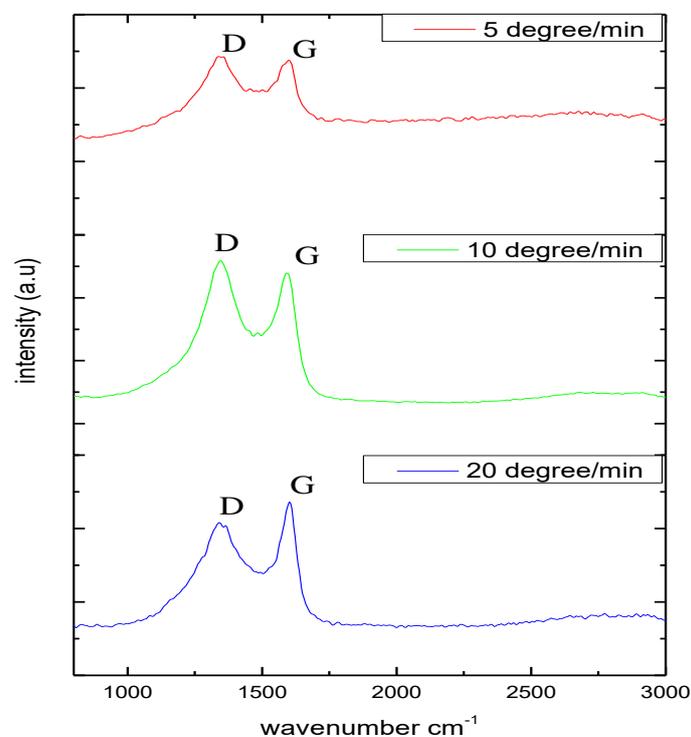


Figure 4. Sample heated at 1000°C various heating rate.

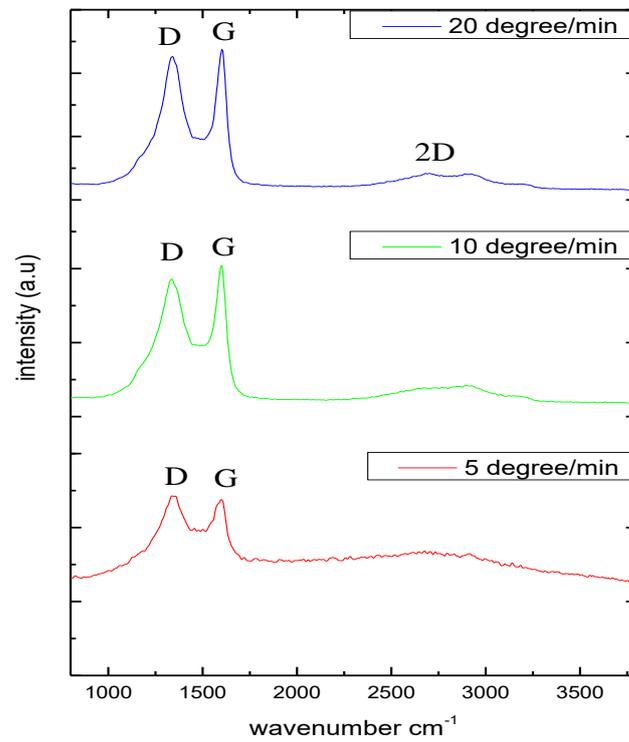
Diffraction pattern was characterized by XRD with the range of  $2\theta = 10^\circ - 90^\circ$ . Figure 2, Figure 3 and Figure 4 show the diffraction pattern of XRD at the heating temperature of  $500^\circ\text{C}$ ,  $800^\circ\text{C}$  and  $1000^\circ\text{C}$  at 3 various heating rate respectively. Diffraction pattern from Figure 2 and Figure 4, is not in agreement with the reference code of 00-041-1487. Besides, the diffraction pattern in Figure 2 and figure 4 also do not compatible with the commercial graphite. Reference code that matched with graphite is 00-041-1487 [11]. However, the diffraction pattern from Figure 3 at the heating rate of  $20^\circ/\text{min}$  is matched with the reference code 00-041-1487 and also comparable with the commercial graphite. Even though the peak is not intense as the commercial graphite, but it can be claimed that synthetic graphite are formed at current temperature and heating rate. The demote in intensity of the peak for sample heated at  $800^\circ\text{C}$  at heating rate  $20^\circ/\text{min}$  is due to the incomplete formation of graphite structures during graphitization process, because graphitization process is claimed to occur when limited movement and rearrangement of carbon atom undergo reconstructive transformation during the heat treatment process.

### 3.2 Analysis of RAMAN Spectroscopy

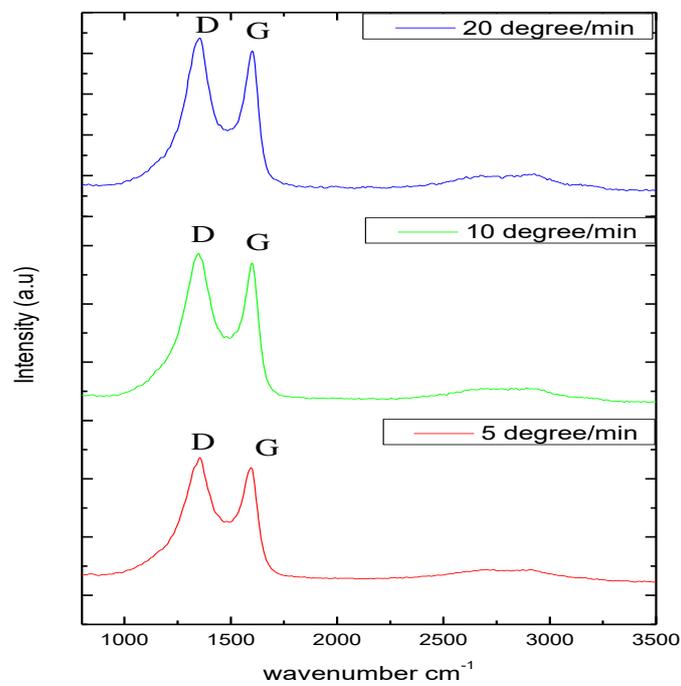
RAMAN Analysis was done in order to support the XRD analysis previously. RAMAN spectroscopy shows the graphitic nature of the graphite obtained in the presence of D, G and 2D peaks. The D, G and 2D peaks mention previously, must be in the stated range of wavenumber. Only then, the sample can be claimed as graphitic in nature. The D peaks must be in the range of  $1200\text{-}1500\text{ cm}^{-1}$  wavenumber. Meanwhile the G peaks should be in the range of  $1500\text{-}1800\text{ cm}^{-1}$  wavenumber. Finally, the most important peak in RAMAN is 2D, and 2D peaks must be presence at the range of  $2700\text{ cm}^{-1}$  wavenumber [12]. Figure 5, Figure 6 and Figure 7 show the RAMAN spectroscopy analysis for sample heated at the temperature of  $500^\circ\text{C}$ ,  $800^\circ\text{C}$  and  $1000^\circ\text{C}$  respectively at various heating rate. Figure 5 and figure 7 show the presence of G and D peaks.



**Figure 5.** RAMAN Spectroscopy sample heated at  $500^\circ\text{C}$  various heating rate.



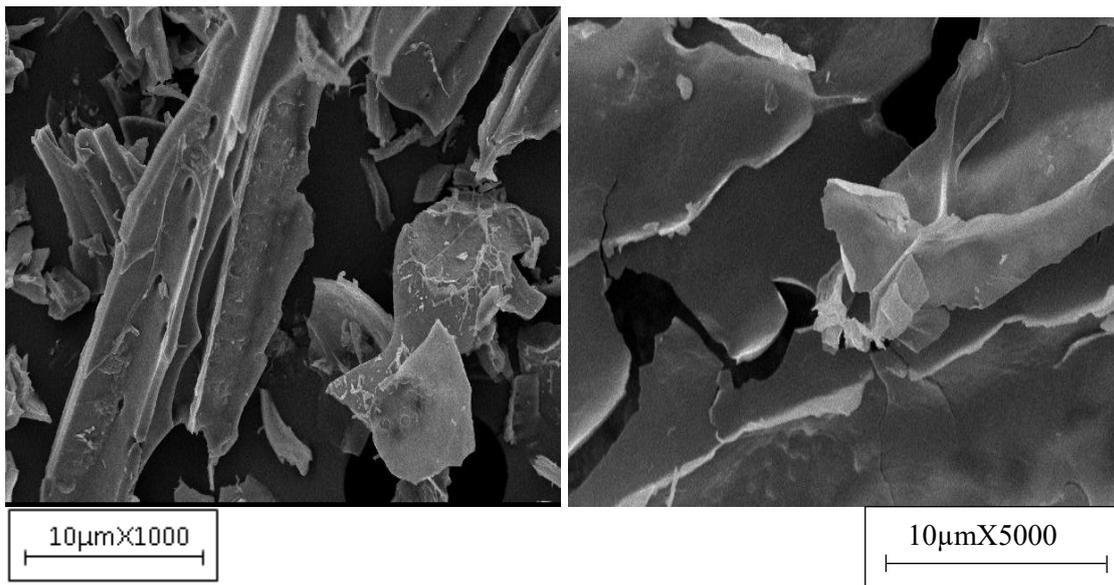
**Figure 6.** RAMAN Spectroscopy sample heated at 800°C various heating rate.



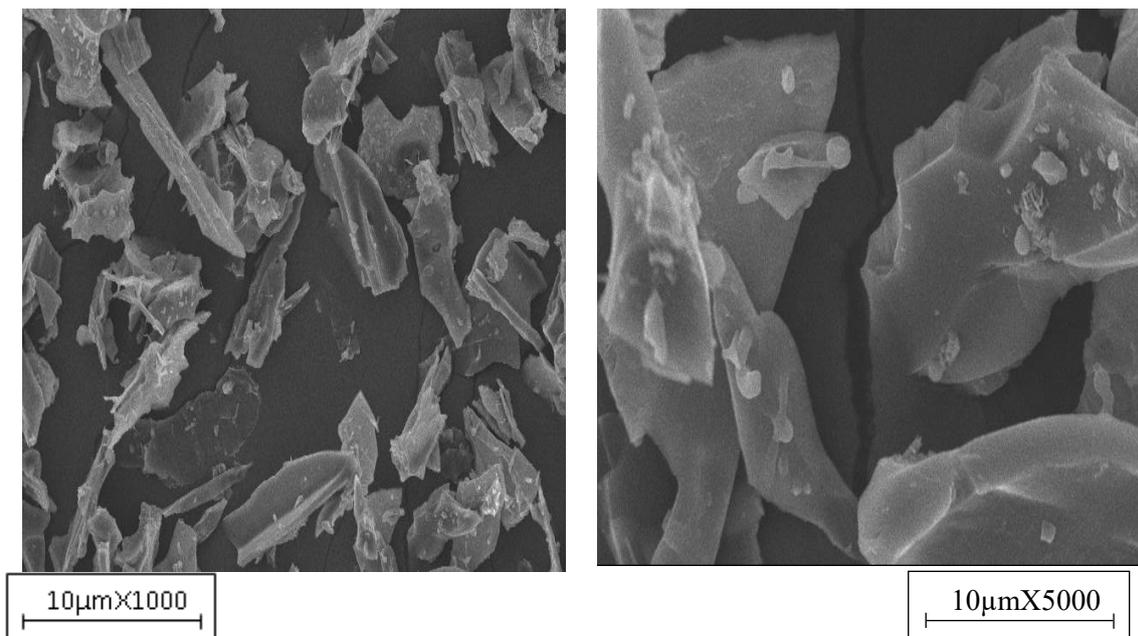
**Figure 7.** RAMAN Spectroscopy sample heated at 1000°C various heating rate.

Whereas, Figure 6 shows RAMAN Spectroscopy of sample heated at 20<sup>o</sup>/min heating rate at the temperature of 800 °C in the presence of G and D peaks with highest intensity. The G-peak was spotted at 1320cm<sup>-1</sup> which indicates the presence of graphitic nature in the produced sample. The presence D-peak which was observed at 1600cm<sup>-1</sup> because of the defect attached at the basal plane of graphite crystal lattice. The presence of 2D peaks was spotted at 2700cm<sup>-1</sup> wavenumber. Nevertheless, it confirmed the presence of graphitic nature of the produced sample and further support the XRD analysis done previously.

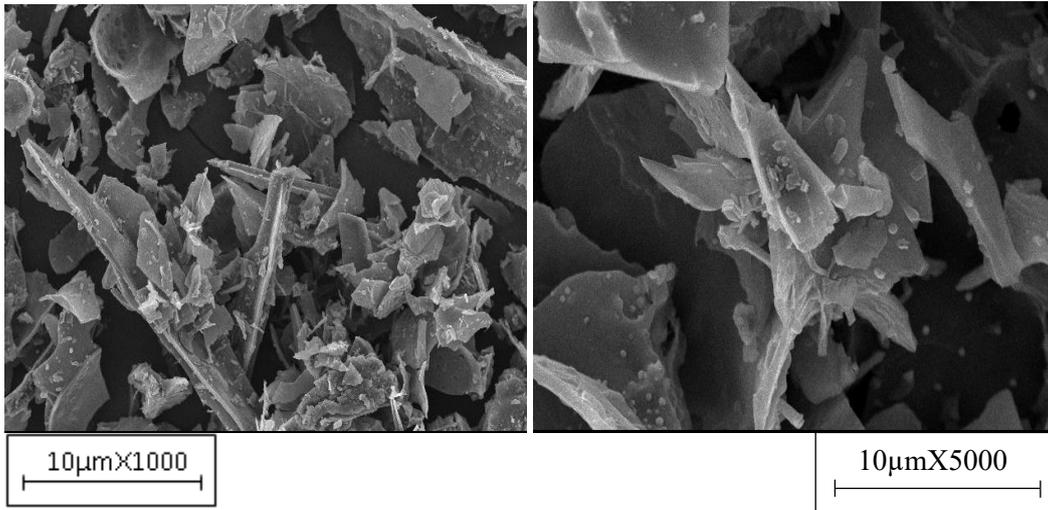
### 3.3 Analysis of Morphology



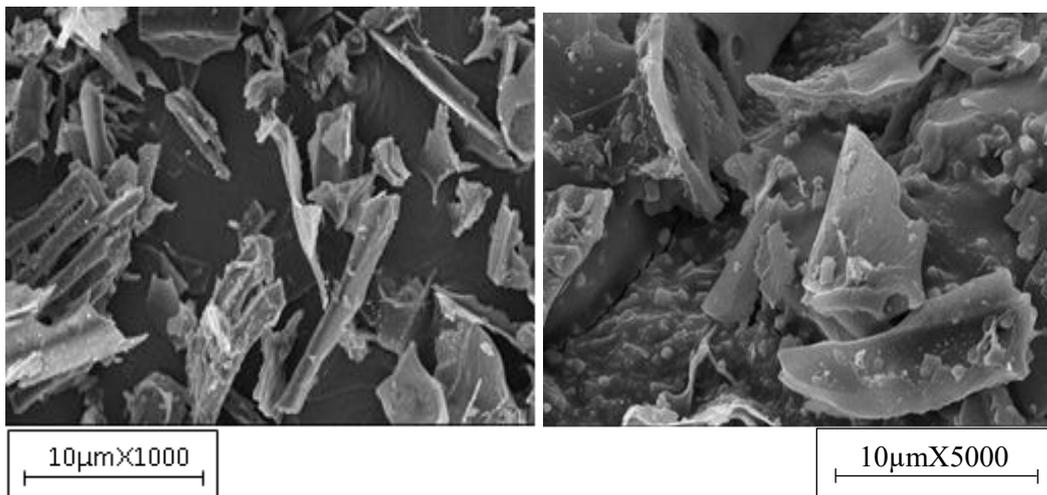
**Figure 8.** Sample heated at 5 °/min at the temperature of 500 °C.



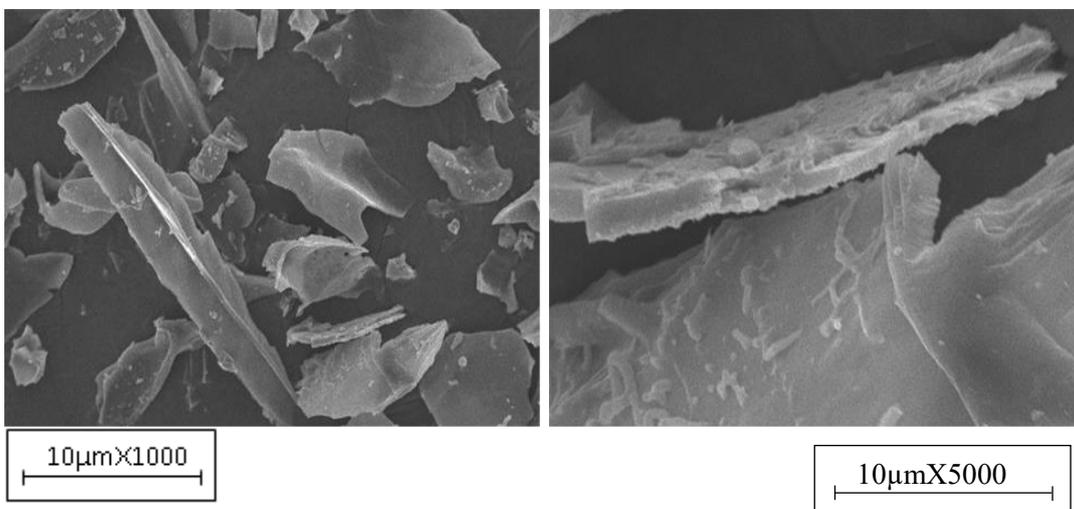
**Figure 9.** Sample heated at 10 °/min at the temperature of 500 °C.



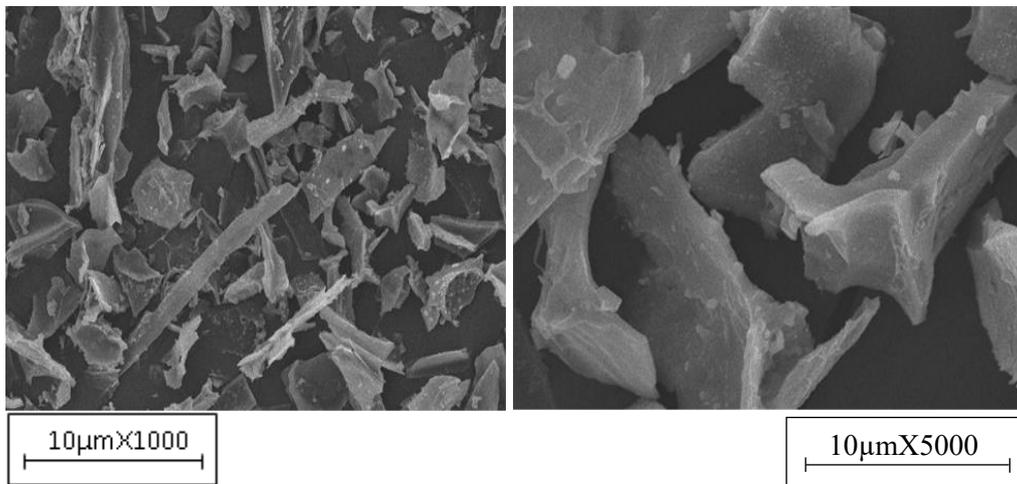
**Figure 10.** Sample heated at 20 °/min at the temperature of 500 °C.



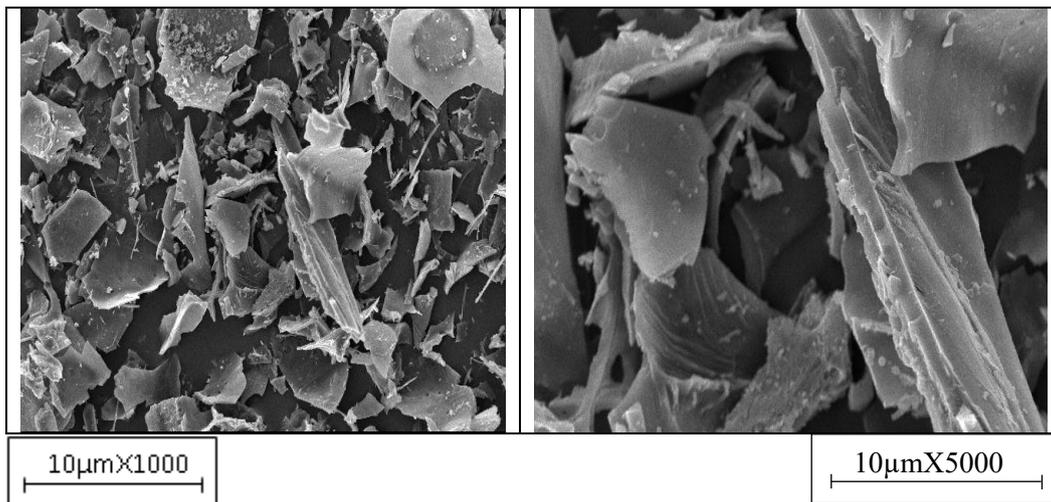
**Figure 11.** Sample heated at 5 °/min at the temperature of 800 °C.



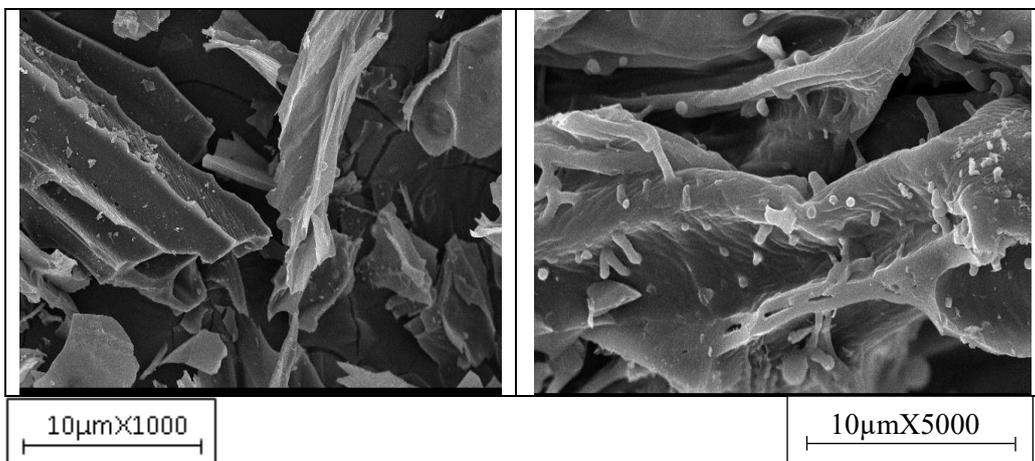
**Figure 12.** Sample heated at 10 °/min at the temperature of 800 °C.



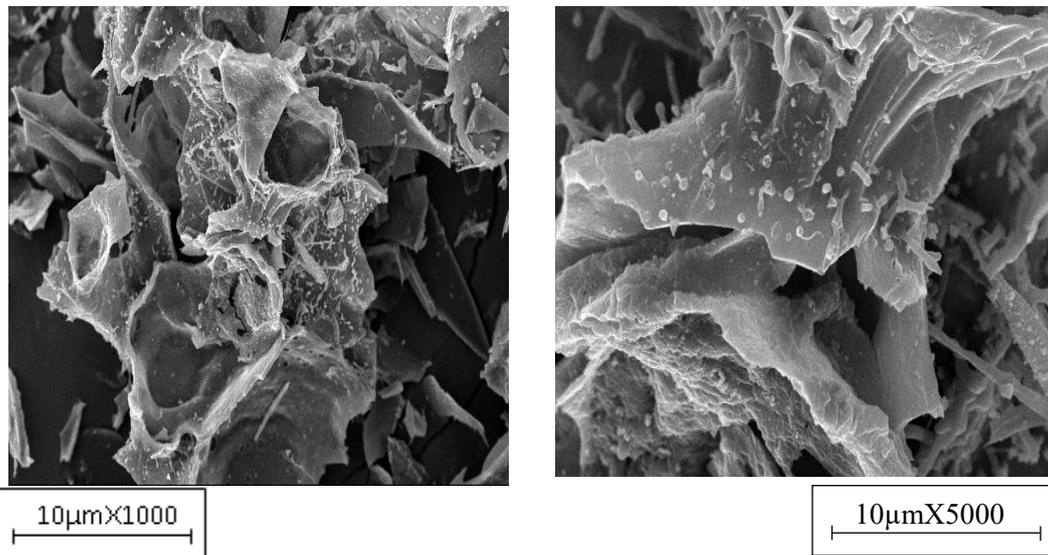
**Figure 13.** Sample heated at 20 °/min at the temperature of 800 °C.



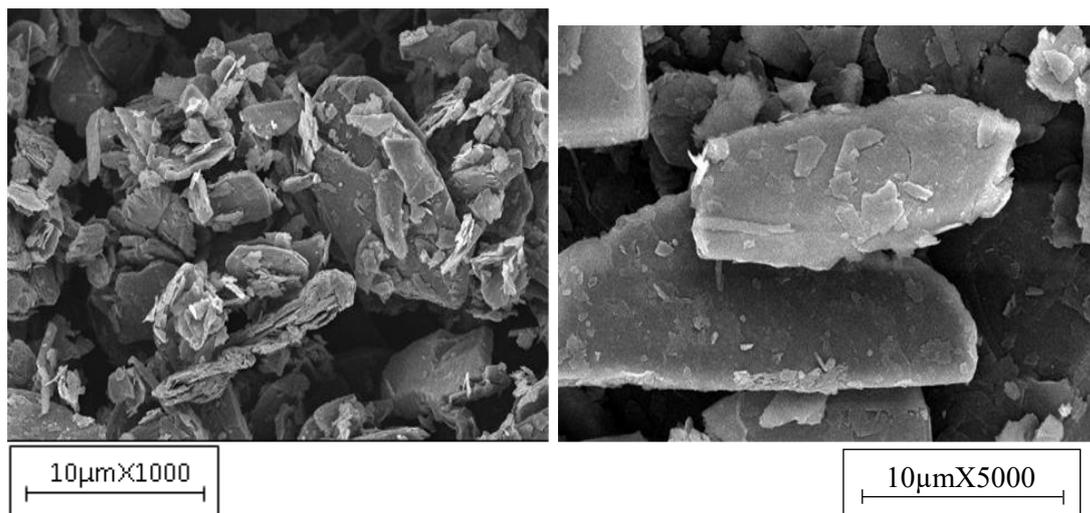
**Figure 14.** Sample heated at 5 °/min at the temperature of 1000 °C.



**Figure 15.** Sample heated at 10 °/min at the temperature of 1000 °C.



**Figure 16.** Sample heated at 20 °C/min at the temperature of 1000 °C.



**Figure 17.** Commercial graphite.

Figure 8, 9, 10 show the SEM images of graphite obtained at the heating temperature of 500 °C at 5°/min, 10°/min and 20°/min respectively. Whereas, figure 11, 12, 13 show the SEM images of graphite obtained at the heating temperature of 800 °C at 5°/min, 10°/min and 20°/min. While, Figure 14, 15, 16 show the SEM images of graphite obtained at the heating temperature of 1000 °C at 5°/min, 10°/min and 20°/min accordingly. Figure 17 shows graphite flakes in the commercial graphite scanned. Figure 13 shows the graphite flakes as it can be compared to Figure 17. From figures above, Figure 13 shows the formation of graphite flakes which comparable with commercial graphite. The graphite flakes were significantly formed and comparable with the graphite flakes from the commercial graphite for both magnification it is, at the 10µmX1000 and 10µmX5000. Complete rearrangement of carbon atom to form graphite turbostratic structure from graphitization process that occurred at the current temperature lead to formation graphite flakes. Vascular bundle tissue that embedded on the surface of the hollow structure of oil palm trunk was release after complete exfoliation upon.

#### 4. Conclusions

This study was successfully carried out to synthesize the synthetic graphite from oil palm trunk waste at lower heating temperature with controlled condition and optimized the processing parameters. The Synthetic graphite samples were characterized using XRD and supported by RAMAN spectroscopy analysis in order to confirm the graphite formation phase after annealing treatment. Based on the analysis by X'Pert Highscore Plus software and supported by RAMAN spectroscopy analysis, it can be concluded that, synthetic graphite was formed and the optimize processing temperature for synthetic graphite from oil palm trunk waste formation phase was formed at the heating temperature of around 800 °C at the heating rate of 20 °/min.

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#### Acknowledgments

This work was supported by Fundamental Research Grant (FRGS) (No.: 9003-00628) and Short Term Grant (STG) (No.: 9001-00496) funded by Ministry of Higher Education Malaysia.