Low temperature synthetic graphite from oil palm trunk waste via pyrolysis process

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Abstract. Synthetic graphite was produced from oil palm trunk chip in lower heating temperature via pyrolysis process. The heating rate (10 °/min and 20 °/min) were varied whilst the heating temperature at 500 °C was fixed. All of the samples produced after heat treatment process were characterized by X-Ray Diffraction (XRD) and the diffraction pattern obtained analyzed using X’Pert Highscore Plus software to affirm the phase analysis. To ensure the graphitic nature of synthetic graphite produced, RAMAN analysis was conducted. Morphological study of the synthetic graphite produced involved scanning electron microscope (SEM) analysis. From the investigation, the results show, confirmation that synthetic graphite was successfully synthesized at the heat treatment of 500 °C (20 °/min heating rate) with fixed soaking hours. Synthetic graphite produced matched with XRD reference code of 00-041-1487. Analysis of RAMAN confirm the formation of D, G and 2D peaks at the respective wavenumber of 1250 cm\textsuperscript{-1}, 1625 cm\textsuperscript{-1} and 2700cm\textsuperscript{-1}.

1.Introduction
Different arrangement of carbon atom lead to the formation of carbon allotrope. Graphite, graphene, carbon nanotubes and fullerene are the most familiar and trending allotrope of carbon which gaining attention among researcher in the field of science and technology especially in electric and electronic device, chemical sensors and energy storage devices. During the present time, graphene since to receive huge attention in the research field [1]. However, the production of graphene comes from the chemical treatment and processes of graphite. Graphite can be categorized into two types, namely natural graphite and synthetic graphite. Natural graphite commonly explained as the result of the easing in sedimentary carbon compounds during metamorphism and occurs in metamorphic rocks. Whereas, synthetic graphite can be processed from high processing temperature of amorphous carbon materials [2].

Previous research reported that synthetic graphite requires high temperature processing parameters nearly 2500-3000°C in order to graphitize [3]. This indicate very high temperature, but the higher heating temperature are required to promote the mobility needed by carbon atoms to rearrange them into a graphite crystal lattice [4]. In short, synthetic graphite is generated from the high temperature processing of amorphous carbon materials. There are many types of materials used as precursor to produced synthetic graphite including, petroleum, coal, natural or synthetic organic materials [5].
Hence, in this study, the researcher is trying to draw attention on producing synthetic graphite at much more lower heating parameters by pyrolysis process.

The oil palm industry in Malaysia started when oil palm tree was introduced as a decorative plant. However, as time flies, oil palm tree becomes one of the most important crops in Malaysia. It was introduced into Malaysia in 1870 through the Singapore Botanic Gardens. Currently Malaysia is facing with the enlargement of vigorous oil palm plantation and palm oil mill. The issue on biomass overloaded began to rise as the industry expanding [12]. The leftover from the palm oil industry are the major contributors to biomass waste in Malaysia, and these biomass waste need extra attention in managing the waste wisely [6]. During replantation, oil palm fruit harvesting, and palm oil processing several biomass wastes are generated including oil palm trunk (OPT), oil palm frond (OPF), oil palm leaves (OPL), empty fruit bunch (EFB), palm kernel shell (PKS), Mesocarp fiber (MF) and palm pressed fiber (PPF) [7].

The oil palm tree only has its economic value for about 25-30 years only, and then it will be prepared for replantation. After replantation the oil palm trunk must be handled properly, if it were to left rotten in the plantation, it will muddle with the proliferation of new growing tree. Thus, the oil palm trunk waste has been utilize as a potential carbon source for synthetic graphite production, as to respond to the government policy to turn oil palm waste into value added products [8], [9]. Oil palm trunk is the second highest of oil palm waste generated in the oil palm biomass which is 13.9 Mn/T[10].

2. Methodology
The felled trunk waste was chopped and undergone chipping process become oil palm trunk chip [11]. Then, the chip oil palm trunk undergoes drying process at ambient condition for a few days. Heat treatment takes place after drying process in controlled condition with purge nitrogen gas at two different heating rate which are, 10o/min, 20o/min at 500 °C with 3 hours soaking. According to N.A. Karim the heating rate was chosen for both parameter [17]. Previous work stated that synthetic graphite was produced from oil palm trunk waste at 800°C with 20°/min of heating rate and 2 hours of soaking time. Each of the samples was characterized by X-Ray Powder Diffraction (XRD). Phase identification of the graphitic compound obtained was confirmed by XRD analysis. [16]. Analysis of the diffraction pattern for every sample was done by using X’pert Highscore Plus. The diffraction pattern from the sample will be matched with the diffraction pattern from the sample. 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.  

\[
\tau = \frac{K\lambda}{\beta\tau \cos\Theta}
\]  

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 \( \mu \)m), K is approximately 0.9 (0.89 for spherical, 0.94 for cubic crystallette, \( \lambda \) is wavelength and \( \Theta \) is diffraction angle [17].

To back up the XRD analysis done previously, the graphitic nature of the synthetic graphite obtained was observed by using RAMAN Spectroscopy analysis. One of the most familiar spectroscopic techniques which depends on inelastic scattering of monochromatic light regularly from laser origin, known as RAMAN Spectroscopy. The sample will draw up the photons of laser light and then release. RAMAN effect, defined as the regularity of the release photons is shifted or down in comparison with original monochromatic frequency. This shift supplies beneficial knowledge on vibrational, rotational and other low frequency transitions in molecules. Raman spectroscopy useful tool to conduct research on solid, liquid and gaseous samples [15].

The morphological studies of the sample were analyzed by using scanning electron microscope with a model of JEOL JSM-6460LA scanning electron microscope (SEM). The studies will include shape, size and texture and phase distribution of the physical objects.

3. Results and Discussion

3.1 Analysis of X-Ray Diffraction (XRD)
To analyze the diffraction pattern from XRD, X’Pert Highscore was found to be the most beneficial and advantageous software with adequate information on phase identification, crystal structure and
degree of crystalline. The characterization of the diffraction pattern occurred in the range of \(2\theta = 10^\circ - 90^\circ\) as shown in Figure 1. From diffraction pattern shown in Figure 1 commercial graphite shows sharp and intense graphitic phase formation peaks at 26° in 2\(\Theta\). This indicates crystalline phase formation of graphite and showing \(d \ (0, \ 0, \ 2)\). The reference code of graphite is 00-041-1487.

![Graphite Diffraction Pattern](image)

**Figure 1.** The comparison of diffraction pattern sample heated at 500°C at various heating rate

The sample heated at 10°/min shows the peak in 2\(\Theta\) at 28.16°. Meanwhile, the sample heated at temperature at 500 °C with 20°/min shows peak 2\(\Theta\) at 27.8° which is close to the reference peak. The clear and sharp of graphitic peak can be seen clearly for sample heated at 20°/min as indicated by green line in Figure 1. The potential of synthetic graphite to be formed is possible. Based on literature, commercial synthetic graphite peak can be seen at 26° in 2\(\Theta\) and 45° in 2\(\Theta\) for potassium peak. Sample heated at 20o/min shows shifted peak, even so, it is still in the range of 26° in 2\(\Theta\). This is due to the incomplete formation of graphite structures during graphitization process. However, the diffraction pattern for sample heated at 20°/min was matched with the reference code of graphite 00-041-1487 [14]. Hence, it is in agreement with [17] stated that graphite phase is formed. The process of graphitization involves the limited movement and rearrangement of carbon atoms which must undergo reconstructive transformation during the heat treatment process. However, the shifting of peak, maybe occur due to the temperature do not provide enough time for the mobility of carbon atom to rearrange into graphite crystal lattice.
3.2 Analysis of Raman Spectroscopy

![Raman Spectroscopy](image)

**Figure 2.** Raman Spectroscopy for sample heated at 20°/min at heating temperature of 500 °C

The appearance of D, G and 2D peak of Raman Spectroscopy for sample heated at 20°/min heating rate and the heating temperature of 500 °C shows can be perceived in figure 2. The explanation of graphitic nature, for D, G and 2D peaks best describes by, the range of 1200-1500 cm⁻¹, 1500 -1800 cm⁻¹ and 2700 cm⁻¹ wavenumber respectively [18]. The D-peak was observed at 1250 cm⁻¹ which notify the existence of graphitic nature in the produced sample. The existence G-peak which was spotted at 1625 cm⁻¹ because of the defect attached at the basal plane of graphite crystal lattice. Furthermore, the existence of 2D- peak marked at 2700 cm⁻¹ briefly stated that graphite phase is formed [18]. In spite of that, it is persistent said that, the existence of graphitic nature of the produced sample and further support the XRD analysis done formerly.

3.3 Morphological analysis by Scanning Electron Microscope (SEM)

![SEM Images](image)

**Figure 3.** Sample heated at the temperature of 500 °C with heating rate 10°/min
Figure 4. Sample heated at the temperature of 500 °C with heating rate 20°/min

Figure 5. Commercial graphite

Scanning Electron Microscope was used to study the samples morphology and structure at the magnification of 1000x and 5000x. The SEM images for sample heated at 10°/min, 20°/min at 500 °C and commercial graphite can be observed in Figure 3,4,5 respectively. Figure 3 shows the formation of graphite flakes begin at the heating rate of 10°/min. However, the formation of the graphite flakes in Figure 4 at the heating rate at 20°/min shows the comparable formation of graphite flakes as shown by the commercial graphite in Figure 5. The formation of graphite flakes and stacking layers of graphite more obvious when the magnification is higher [16].

4. Conclusion

Synthetic graphite was successfully produced at lower heating temperature via pyrolysis. The produced synthetic graphite was characterized using XRD and the diffraction pattern was successfully analysed by X’pert HighScore Plus software. From XRD, the synthetic graphite phase was formed at 27.8° of crystallinity in 20. RAMAN Spectroscopy done has confirmed the graphitic nature of the synthetic graphite obtained with the significant formation of the D, G and 2D peak at the spotted wavelength. Thus, with the reference on the analysis of X’Pert Highscore Plus software and backing up by RAMAN spectroscopy analysis, it is briefly confirmed that, graphite phase was formed at the heating parameter of around 500 °C of 20°/min rate.
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