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Production of Low Temperature Synthetic Graphite

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ABSTRACT

Synthetic graphite is a material consisting of graphitic carbon which has been obtained by graphitizing a non-graphitic carbon. The growth in demand, particularly in customizing properties for certain usage has brought about research on viable alternative, low-cost, and environmentally pleasant synthetic graphite production. Biomass wastes are amongst appealing carbon precursors which have been broadly checked out as replacement carbon for graphite production. This research aimed to synthesize synthetic graphite from oil palm trunks at low temperatures (500 °C, 400 °C and 300 °C) under controlled conditions to determine the physical properties and properties of the graphite obtained. After the heat treatment process, the obtained samples were then characterized by using XRD, SEM and RAMAN characterizations. Based on SEM and RAMAN characterization, it can be seen that graphite that undergoes a 500 °C pyrolysis process shows the best results compare to graphite that undergoes a pyrolysis process at the temperatures of 300 °C and 400 °C. The graphite flakes and the peaks obtained for 500 °C graphite are obviously present. For XRD characterization, the best samples at 500 °C were chosen to be characterized. From the results, the sample shows slight behavior imitating the commercialized graphite. Hence, from the characterizations of the samples, it can be concluded that the best synthetic graphite produced was from the oil palm trunks heated at 500 ° C

Keywords: Hummer's method, Synthetic graphite, Carbon

1. INTRODUCTION

Carbon which is also known as charcoal is the fourth most plentiful element in the world and the second largest element in our bodies. In fact, all organic substances in the entire universe consist of carbon in some form or element. The formation of carbon allotropes differs in the arrangement of the carbon atoms and is becoming prominent in scientific research especially in electric and electron devices, sensor applications and energy storage devices [1]. There are a few carbon allotropes such as diamond, graphite, graphene and fullerenes. The different arrangements of atoms in allotropes of carbon can be seen in Figure 1.

Graphite can be classified into synthetic graphite and natural graphite. Natural graphite can be considered as a mineral structure of graphitic carbon where it differs significantly in

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crystallinity. It usually consists of additional minerals and impurities which makes it a good conductor of electricity as well as heat. Three main types of natural graphite include high crystalline graphite, amorphous graphite and flakes graphite where each class has different physical and chemical properties [2].

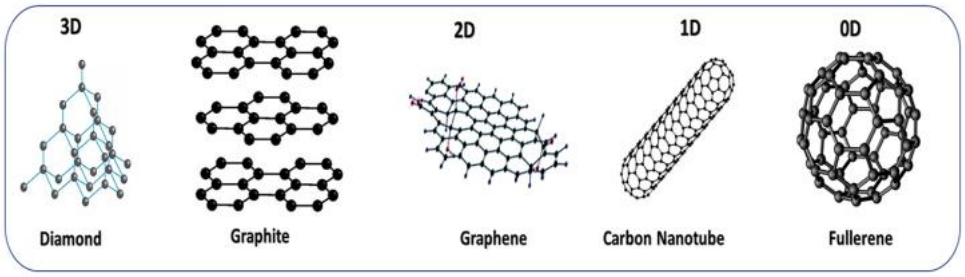


Figure 1. Different arrangements of atoms in allotropic forms of carbon [3]

Synthetic graphite is a substance that can be obtained from coke, petroleum, or natural and synthetic organic materials which is produced by the high temperature processing of an amorphous carbon material [4]. In the previous research, a temperature of up to 2500 K is required to produce the synthetic graphite which gives a high energy cost [5]. The main concern in producing synthetic graphite is to make sure that high carbon content materials were used as the main sources. In the presence, numbers of approaches have been utilized in order to synthesize the best synthetic graphite including chemical vapor decomposition, Joule heating, thermal heating and microwave heating [6]. In Malaysia, oil palm trunks can be considered as an abundant source with the possibility to act as an organic material for synthetic graphite production. Hence, in this research, we attempted to produce the synthetic graphite at a low heating temperature by using an oil palm trunk.

2. METHODOLOGY

2.1 Synthesis of Synthetic Graphite from Oil Palm Trunk Waste

The oil palm trunk waste that was collected from the oil palm plantation was first cut into big slices and let air dried under ambient conditions for several days to remove excessive moisture. Then the slice of the trunk was measured and cut into a small dimension to be produced as the oil palm trunk chips. The oil palm trunk chips were then undergoing the heating process in a controlled heating condition which is known as the pyrolysis method in a tube furnace as shown in Table 3.1 with the presence of nitrogen gas. The range of the heating temperature at 500 °C, 400 °C and 300 °C with 20°, 10° and 5°/min of heating rate was applied. After the pyrolysis process, the oil palm trunk chips were turned into charcoal and it is then ground manually to be produced into a fine powder form in order to further characterize the obtained graphite. The overall process to obtain the synthetic graphite from the oil palm trunk waste can be seen in Figure 2.

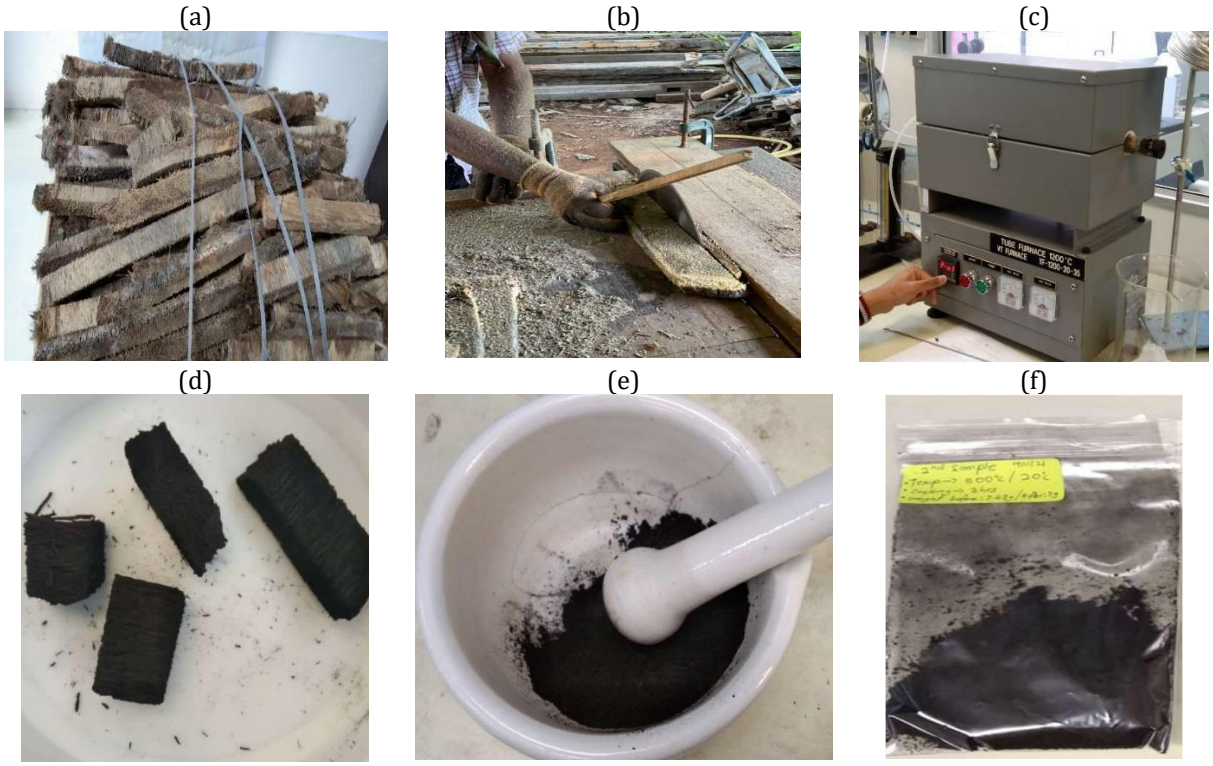


Figure 2. The overall process for synthetic graphite production includes (a) air-drying of the chopped oil palm trunk waste (b) chopping of the oil palm trunk waste into small chips (c) pyrolysis process at different temperatures and heating rates (d) production of charcoal (e) manual grinding of the obtained charcoal and (f) final product of the graphite in powder form

2.2 Characterization of Synthetic Graphite Obtained

The fine powder that was obtained from the synthetic graphite was first measured in a Densimeter. This measurement was done in order to compare the density of commercial graphite with the synthetic graphite produced. Next, samples characterizations with three types of analysis such as X-Ray Diffraction Analysis (XRD) RAMAN Analysis. and Scanning Electron Microscope Analysis (SEM) were done. XRD Analysis and RAMAN Analysis are important to identify the graphitic nature of the synthetic graphite while SEM Analysis is done to analyze the morphology of the samples.

To prepare the samples for RAMAN and SEM characterizations, the powder of the synthetic graphite produced was first diluted with DI water at the concentration of 1 mg/mL. Next, the prepared solution was deposited on a Silicon wafer at a temperature of 100 °C. For XRD Analysis, the best sample that imitated graphite behavior was analyzed in powder form without any modification from the obtained synthetic graphite

3. RESULTS AND DISCUSSION

3.1 Density Measurement of Synthetic Graphite

Density measurement was recorded once the synthetic graphite is produced from oil palm trunk waste. The measurement was conducted for all nine samples heated at temperatures of 300°C, 400°C and 500°C with various heating rates. Then, it was compared with the density of commercial synthetic graphite. TABLE 1 shows the different measurements of oil palm trunk graphite with commercial graphite. From the observation, it shows that the density of commercial graphite reported by [7] is much higher at 2.26 g/cm^{-3} than synthetic graphite produced from the oil palm trunk. This may be due to the synthetic materials used to produce the graphite. Different materials will exhibit different pores and particle sizes which relatively reduce the porosity of the material hence contributing to different densities measured.

TABLE 1. The density of the synthetic graphite obtained from the oil palm trunk.

Heating Temperature (°C)	Heating Rate (°/min)	Density (g/cm^{-3})
300	20	1.029
300	10	1.120
300	5	1.059
400	20	1.246
400	10	1.299
400	5	1.142
500	20	1.226
500	10	1.197
500	5	1.125

3.2 Characterizations of Synthetic Graphite

The production of low temperature synthetic graphite was done at three different temperatures with different heating rates via the pyrolysis method. The range of samples heated at different temperatures with various heating rates was applied to investigate which parameter gives the most suitable result. Furthermore, all of the analysis and results of characterization were recorded and explained briefly in the figures below.

3.2.1 Scanning Electron Microscopes (SEM)

Scanning electron microscope (SEM) was conducted to analyze the morphology, shapes and sizes of the materials prepared [8]. Figure 3 to Figure 5 shows all the graphite powder that had been prepared for this study. The magnification for SEM analysis of the prepared graphite was set to 1.5k times for each sample. Figure 3 shows the SEM analysis at temperatures of 500 °C, 400 °C and 300 °C with a 5°/minute heating rate. From the images, it can be seen that clearer flakes formation is shown at 500 °C (Figure 3(a)) compare to 400 and 300 °C (Figure 3(b) and (c)) which shows the unclear formation of flaky graphite. At a 10°/minute heating rate, the

flakes formations are more visible starting from the graphite produced at 500 °C, 400 °C and lastly at the temperature of 300 °C as shown in Figure 4(a), (b) and (c) simultaneously. As the heating rate increased to 20°/minute the best graphite flakes were obtained. From Figure 5 the best formation of the graphite flakes can be seen. Referring to the images, 500 °C (Figure 5(a)) temperature shows the best flakes formation. This may be due to the tissue of vascular bundle releases and the structure of the oil palm trunk starting to exfoliate in this state [9].

From overall SEM analysis of all three temperatures, such condition occurred due to the raw structure of the oil palm trunk that exists in the current temperature, a clumpy structure is formed which is also due to lots of vascular bundles occurring on the oil palm trunk surface and a complete exfoliation do not occur at these heating temperatures [9]. An incomplete rearrangement of carbon atoms during the graphitization process causes an incomplete formation of graphite flakes from these samples [10]. Hence, based on SEM analysis, it can be concluded that the best pyrolysis method at low temperatures was recorded at 500 °C with a 20°/minute heating rate.

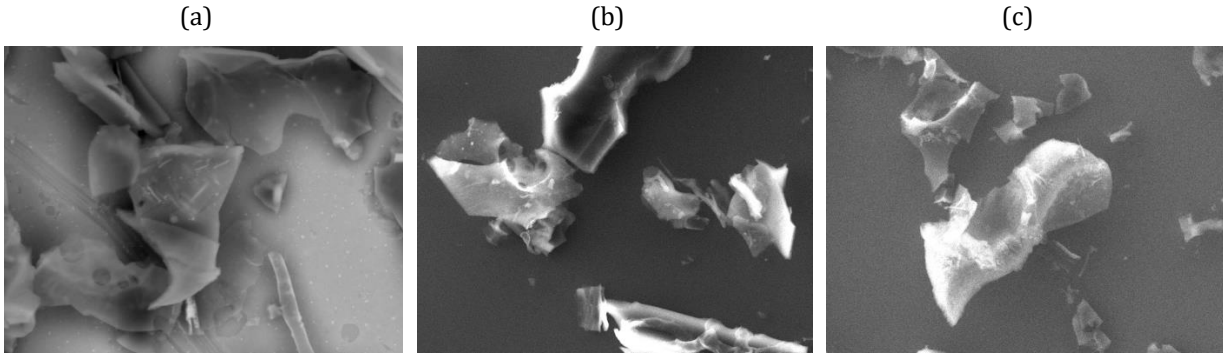


Figure 3. SEM images for (a) 500 °C, (b) 400 °C and (c) 300 °C graphite with 5°/min heating rate

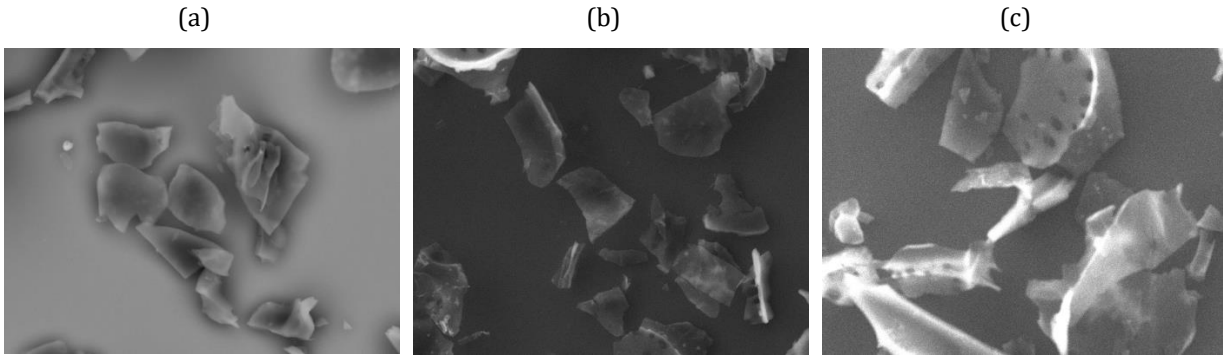


Figure 4. SEM images for (a) 500 °C, (b) 400 °C and (c) 300 °C graphite with 10°/min heating rate

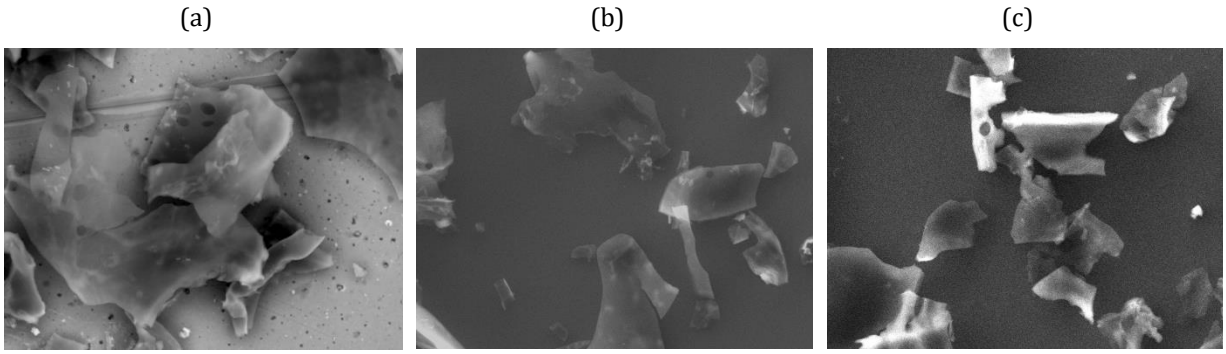


Figure 5. SEM images for (a) 500 °C, (b) 400 °C and (c) 300 °C graphite with 20°/min heating rate

3.2.2 RAMAN Analysis

RAMAN spectroscopy is done to analyze the graphitic nature of the graphite produced with the presence of D, G and 2D peaks where the peaks should be in a certain range of wavenumber so that it can be declared as graphitic in nature. RAMAN spectra are carried out to support the analysis of XRD. Based on the literature, the range of D peak should be between 1200-1500cm^{-1} wavenumber. Other than that, the range of the G peak must be between 1500-1800cm^{-1} while the 2D peak should be in the range of 2700 cm-1 wavenumber which is the main peak in RAMAN [11]. Figure 6 shows the sample of RAMAN analysis with the temperature of 500°C, 400 °C and 300 °C with 20°, 10°, and 5°/min heating rates in the presence of D and G peaks. According to Figure 6 (a), (b) and (c) only the graphite prepared at 500 °C shows the presence of D and G peaks. The sample heated at 5°/min (Figure 6(a)) shows a 1235 cm^{-1} wavenumber at the D peak and a 1513 cm^{-1} wavenumber at the G peak. However, the range of 2D peak cannot be seen at the stated wavenumber. At the same time, the sample of 10°/min heating rate Figure 6(b)) shows the D and G peaks at the wavenumber of 1241 cm^{-1} and 1502 cm^{-1} also without the presence of a 2D peak at 2700cm^{-1}. At a 20°/min heating rate (Figure 6(c)), the D and G peaks can be seen at 1260 cm^{-1} and 1509 cm^{-1}. The 2D peak did not exist in any three samples. From this analysis, we can conclude that there is no exact synthetic graphite was produced from the oil palm trunk waste. This might be due to the noise that occurred by a few independent phenomena in Raman spectra. Thus, the exact wavenumber of D and G peaks cannot be seen clearly and this indicates that there is no synthetic graphite was produced. However, comparing the three temperatures, the temperature of 500 °C shows the best RAMAN results. Hence, it can be concluded that 500 °C is the best lowest temperature for graphite production.

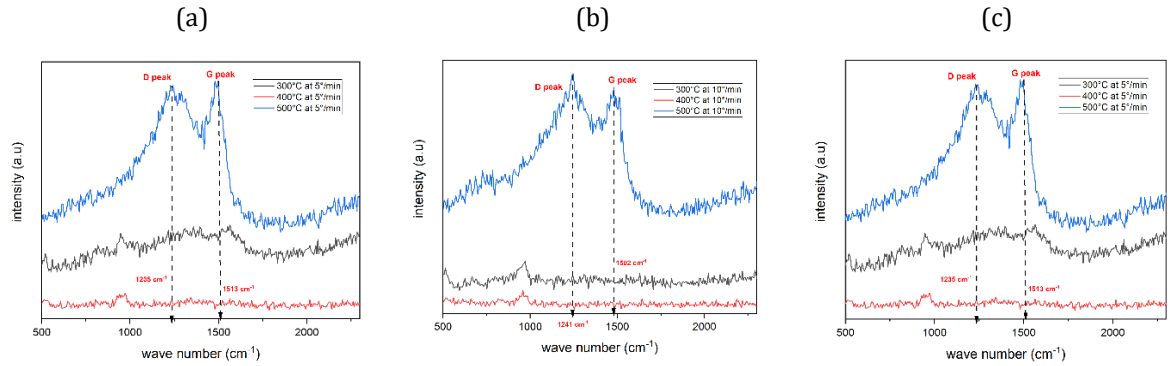
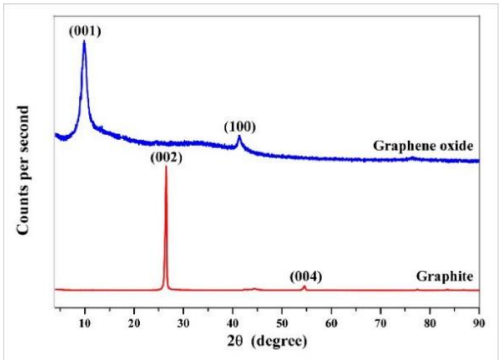


Figure 6. RAMAN analysis for (a) heating rate of 5°/min, (b) heating rate of 10°/min and (c) heating rate of 20°/min

3.2.3 X-Ray Diffraction (XRD) Analysis

To analyze the structure of the crystal, phase identification and crystallinity degree, x-ray diffraction (XRD) is known to be the most beneficial and preferable software with sufficient data in order to analyze the diffraction pattern [12]. For XRD characterization, the best result from previous characterizations which was the graphite produced at 500 ℃ was used. From Figure 7, the range of the diffraction pattern has occurred between 2 theta= 10°-90° for characterization. The comparison between the commercial graphite in Figure 7(a) and oil palm trunk synthetic graphite in Figure 7(b) was conducted to analyze the differences in the diffraction pattern. From the commercial graphite, the peak of the graphitic phase of graphite is shown at 26.5° in 2Θ and 55° in 2Θ, which represent the formation of the crystalline phase of graphite at (0,0,2) as stated in literature [13]. Besides, the sample of 500℃ temperature with various heating rates from oil palm trunk waste also showed its graphitic phase with slightly different peaks such as the sample with 5°/min of heating rate is 28°at 2Θ. The heating rate of 10°/min shows the peak in 2Θ at 27.5° which is the nearest to the commercial peak. Finally, the heating rate of 20°/min shows the peak in 2Θ at 28.5°. Thus, the closest graphitic peak that can be concluded is the heating rate of 10°/min as shown by the blue line in Figure 7(b) which has the possibility to form synthetic graphite compared to commercial graphite. The reason behind the different ranges of peaks occurring is due to insufficient graphitic structure formation during the process of graphitization, where the carbon atoms provide limited arrangement when the temperature does not allow complete time to undergo the process for the atoms to rearrange into the graphitic phase [14].

(a)



(b)

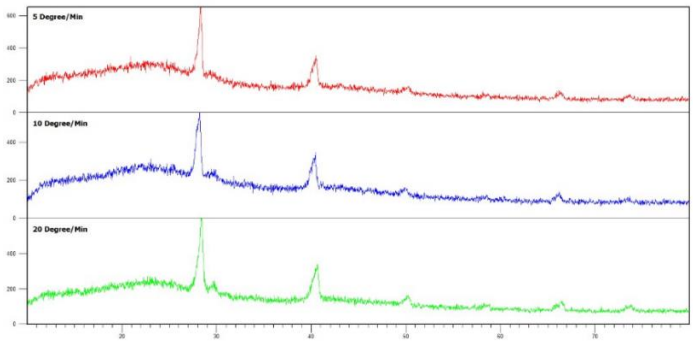


Figure 7. XRD analysis of (a) commercial graphite and (b) 500 ℃ synthetic graphite with 5°, 10° and 20°/minute heating rate

4. CONCLUSION

This research was conducted to produce the synthetic graphite at a lower temperature which is below 1000 °C by using the oil palm trunk waste with a lower heating rate via the pyrolysis process. Hence, three different parameters of heating temperature had been applied at 300 °C, 400 °C and 500 °C with a heating rate of 5°/min, 10°/min and 20°/min for each temperature. The graphitization process was carried out in a tube furnace with the presence of nitrogen gas and the absence of oxygen. The results of the samples are then compared with commercial graphite. In conclusion, the result successfully shows that the heating temperature of 500 °C with a heating rate of 20°/min gave the best results closest to the commercialized graphite according to the characterizations made, proving that the synthetic graphite was successfully synthesized and fulfilled the requirements.

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