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Characterization of Graphite Oxide synthesis from Manufactured Graphite, EFB, and OPS Bio-Char

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Abstract

This work presents the characteristic of synthesizing graphite oxide from various sources included manufactured graphite flakes (MG), Empty Fruit Bunch (EFB), and Oil Palm Shell (OPS) Biochar to produce M-GO, EFB-GO, and OPS-GO, respectively. The earlier Modified Hummers method was upgraded in this study, where the synthesis was done without Sodium Nitrate (NaNO3). The analyses were done by FESEM, EDX, XRD, and UV-VIS. The result from the analysis of GO shows a relevant relation as to be compared through all the analysis and supported by the previous study. The analysis also reveals that the oxidation of graphite has been moderately completed and the increase in stirring time during the synthesis can be done in order to improve the quality of GO.

Keywords: GO Synthesis; M-GO; EFB-GO; OPS-GO; Upgrade Hummer's Modified Method

Introduction

Graphene oxide (GO) consists of a few layers of graphite bonded to the oxygen functional group [1]. The presence of various oxygen groups results in good dispersibility of GO in water which then contributes to bio-application [2]. Recently, researchers were focused on developing a sustainable technique for the production of graphene-like materials from biomass waste by the carbonization process [3]. GO is soluble in water and other solvents [4], and it can even be reduced to graphene due to the fantastic properties it contains in the pristine graphene [5]. Although GO is not a good conductor, yet, tremendous studies have been done to improve its characteristics, especially in electrical properties. The uniqueness of its properties has attracted many interests to deep-dive this family of graphene. For example, a group from UNSW [6], led a new study that successfully discovered a unique behavior in GO. They found that the oxygen atoms are generally connected in a random manner. The oxygen atoms, on the other hand, develop more ordered structures by themselves at higher temperatures. This 'self-organization' process was discovered to greatly increase a variety of aspects of GO, including its electrical conductivity. The exploration has opened a new window to deeply investigate the possibility of biochar in synthesizing GO due to the highly saturated carbon element in the biochar. In this work, we study the characteristics of GO produced by implementing the biochar from empty fruit bunches and oil palm shells as starting materials. The work is fruitful and beneficial to the industry and researchers especially in biomass management towards the potential of the nanomaterials field.

Materials and Method

The samples of GO are synthesis from various sources of graphite including M-GO, EFB-GO, and OPS-GO. All samples are synthesized using the modified Hummer's method without including NaNO3 and the process is carried out at ambient temperature. Using this method, 81 ml of H2SO4 is mixed with 9 ml H3PO4 and being stirred for 10 minutes using a stirring hotplate at 5000 rpm. Then, 0.675 g graphite powder is slowly added and stirred for 10 minutes before gradually adding 3.96 g KMNO4. The mixture is stirred for 6 hours. Then, 2 ml of H2O2 has been dropped and stirred for 10 minutes in order to remove the excess KMNO4. Turn off the stirrer and let it cool down. Finally, 90 ml of DI water and 30 ml of HCL are mixed together with the solution and continue stirring for 10 minutes. The centrifuged samples are then dried using an oven for 5 minutes at 300℃ to get the powder form.

Results and Discussion

FESEM

The surface topography of the produced GO samples was examined at the submicron scale using FESEM analysis. The FESEM image obtained reveals that the undergo oxidation process has been merely completed for all the samples and the graphite oxide layers are also able to be seen. The morphology of GO shows a bit smooth, thin, and flat sheet with various wrinkled edges and contorted patches which is also reported by Abrah et. al [7]. The GO sheet is densely packed with oxygen-containing functional groups, which are mostly concentrated towards the sheet's edges and can be seen as the light parts [8]. Specifically, Figure 1 below shows the FESEM image for all the samples, and from this, it can be deduced that M-GO shows a very smooth and thin sheet. EFB-GO and OPS-GO tend to have a similar morphology which is thicker than M-GO and observation of a few carbon sheets can be seen. However, EFB-GO has shown wide open pores which contributes to the acid treatment process caused by highly synthesized sheets of GO [9].

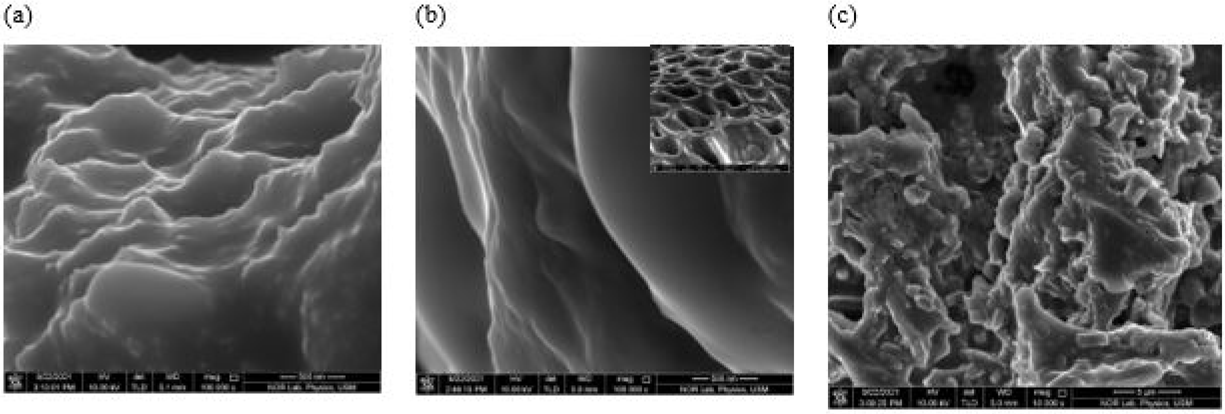

Figure 1: FESEM image of a) M-GO , b) EFB-GO and c) OPS-GO at 100k X magnification

EDX

The percentage value of elements in the sample was determined using EDX. The appeared percentage value for carbon elements of M-GO, EFB-GO and OPS-GO is 45.35 %, 47.51 %, 47.83 % respectively. Meanwhile, the percentage value for oxygen recorded is 54.65 %, 52.49 %, and 52.17 % respectively. From the result in Figure 2, all the samples of GO show a similar trend regarding the percentage ratio of carbon to oxygen. It has more oxygen than carbon, indicating that oxidation is more occurs at the sheet's edges.

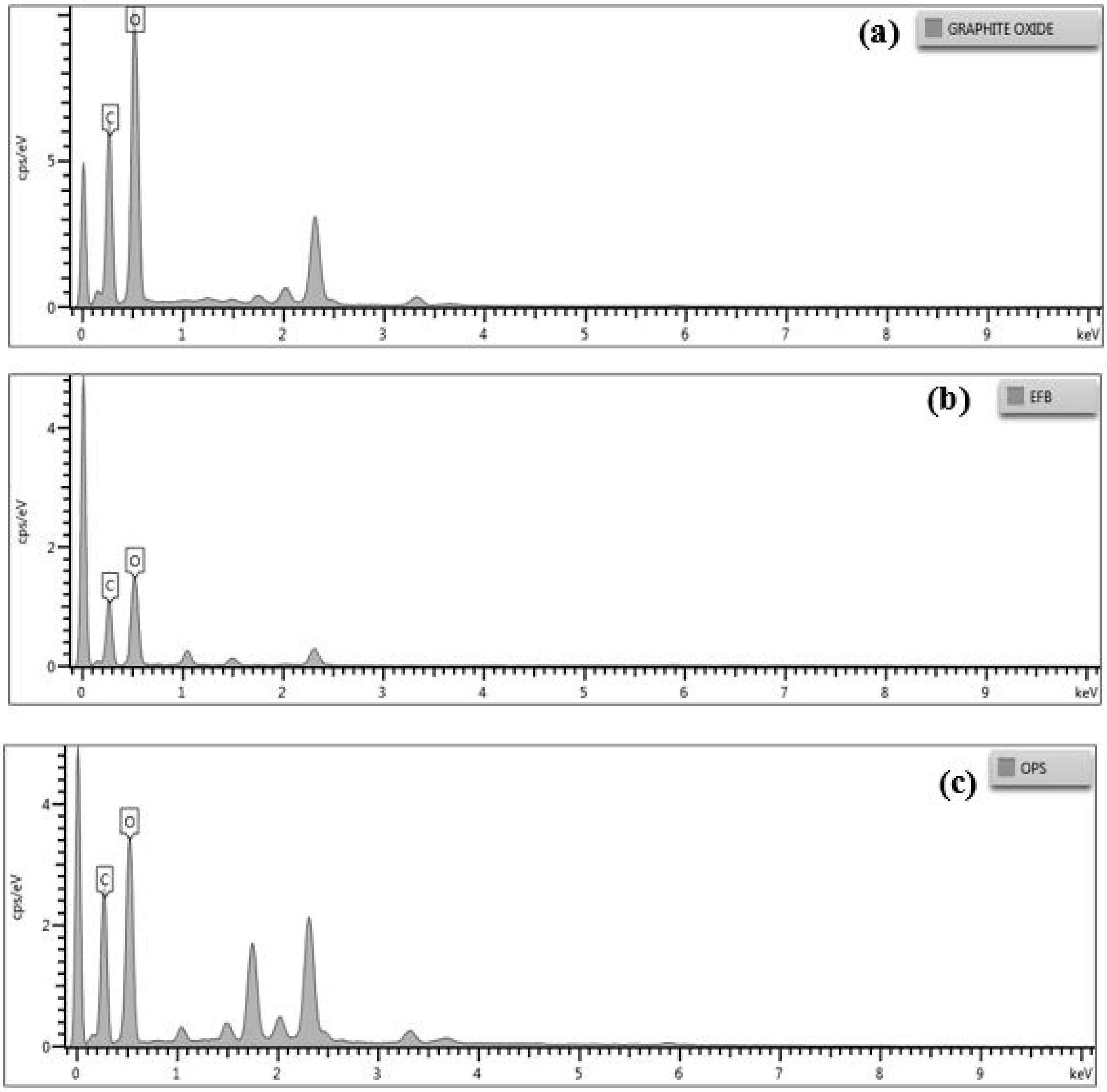

Figure 2: EDX spectra for 3 different samples which are a) M-GO , b) EFB-GO and c) OPS-GO indicate carbon to oxygen ratio

XRD analysis

To identify the material in the samples and estimate its crystallite size, and XRD scan was employed as a reference. XRD was also utilized to investigate the degree of graphite oxidation Figure 3 shows the peak intensity of all the GO samples. It can be seen that the peak appears between the range of 22.0-26.0 where M-GO and OPS-GO show a broad peak while EFB-GO shows a sharp peak. As reported by Yau et. al [8], the stirring time (reaction time) during the synthesis of GO will contribute to the peak intensity changes. The resulting ideal peak for GO (completed oxidation) was reported by another reporter at 10.5°-11.0° [10-12], The slightly shifted value of theta from the experiment range means the sample contains more graphitic structure because of the intensity peak for graphite is between the presented range.

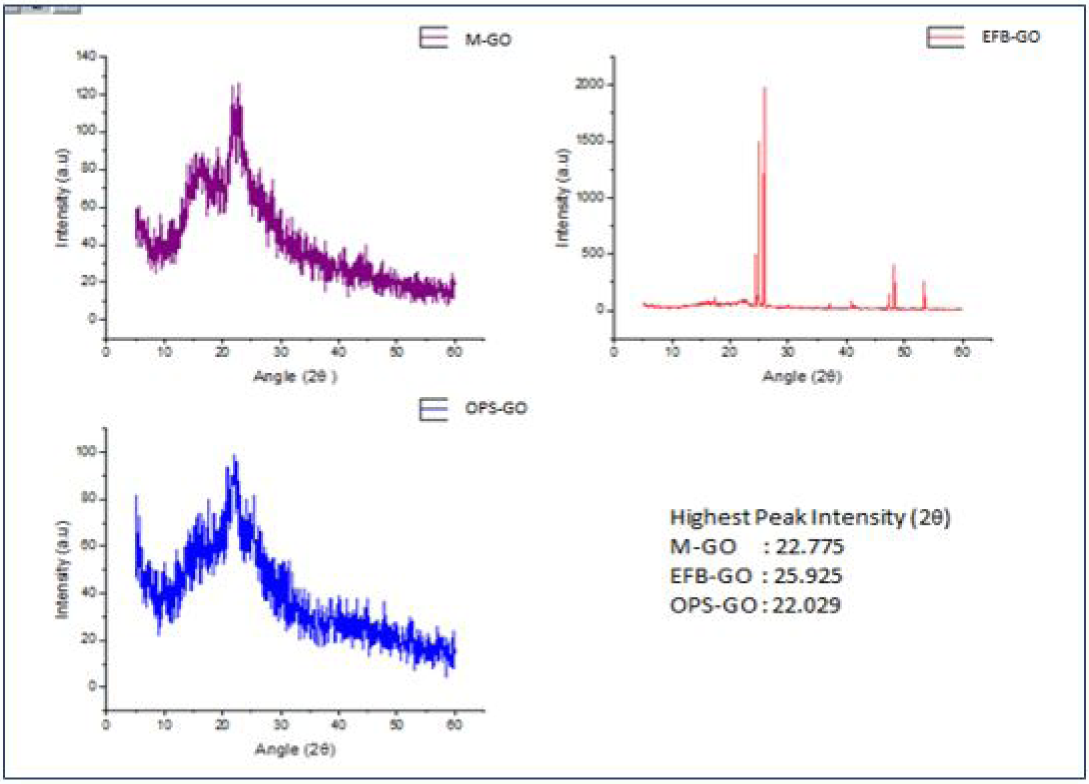

Figure 3: XRD spectra shows the peak intensity of M-GO, EFB-GO and OPS-GO

UV-VIS spectroscopy

Figure 3 shows the UV-vis spectra of aqueous GO dispersions. The presented spectra result appeared the same as reported by Sharma et. al [13]. To identify GO, two types of distinguishing characteristics were noticed in these spectra. The first is a shoulder at 331 nm, 308 nm, and 285 nm, which corresponds to the peak of n-π * plasmon which is owing to the existence of epoxide and carbonyl (C=O) bonds. Meanwhile -the* plasmon peak of aromatic C-C bonds in differently sized aromatic sp2 clusters arises at 218 nm, which is another distinguishing trait [14]. This feature's strength and position differ between samples. M-GO and OPS-GO exhibit a dominant high and sharp peak at 218 nm with a broad shoulder at 308 nm and 285 nm, respectively. Apart from that, the EFB-GO shows nearly disappeared shoulder at 331 nm with a couple of intense peaks at 300 nm and 218 nm.

ATR-FTIR spectroscopy

The presence of the functional groups on the surface of synthesis GO was predicted by FTIR in reflectance mood. In a broad sense, the trend exhibited by IR spectra can lead to a variety of conclusions. As mentioned by previous work [15], GO has epoxide and hydroxyl functional groups on both sides of its basal plane with carboxyl functionalities at the edge sites. Figure 4 presents the IR spectrum for synthesis GO which shows a broad and high peak between wavenumber of 1300 cm^{-1}-800 cm^{-1} for all the samples. It is indicated hydrocarbon containing a C=C bond and believed to belong to the existence of alkene and epoxide. O-H stretching vibration can be observed in OPS-GO for the small broad peak between 3400-3700. C-H stretching was observed at a very tiny peak between 3000-3100. The peak that appeared between 1600 cm^{-1} and 1800 cm^{-1} arose from the C=O stretching of the carboxylic acid groups while the band at 1650 cm^{-1} was assigned to the aromatic C-C stretching. O-H deformation can be seen at peak corresponding to approximately 1400 cm^{-1} and C–OH stretching at 1284 cm^{-1}.

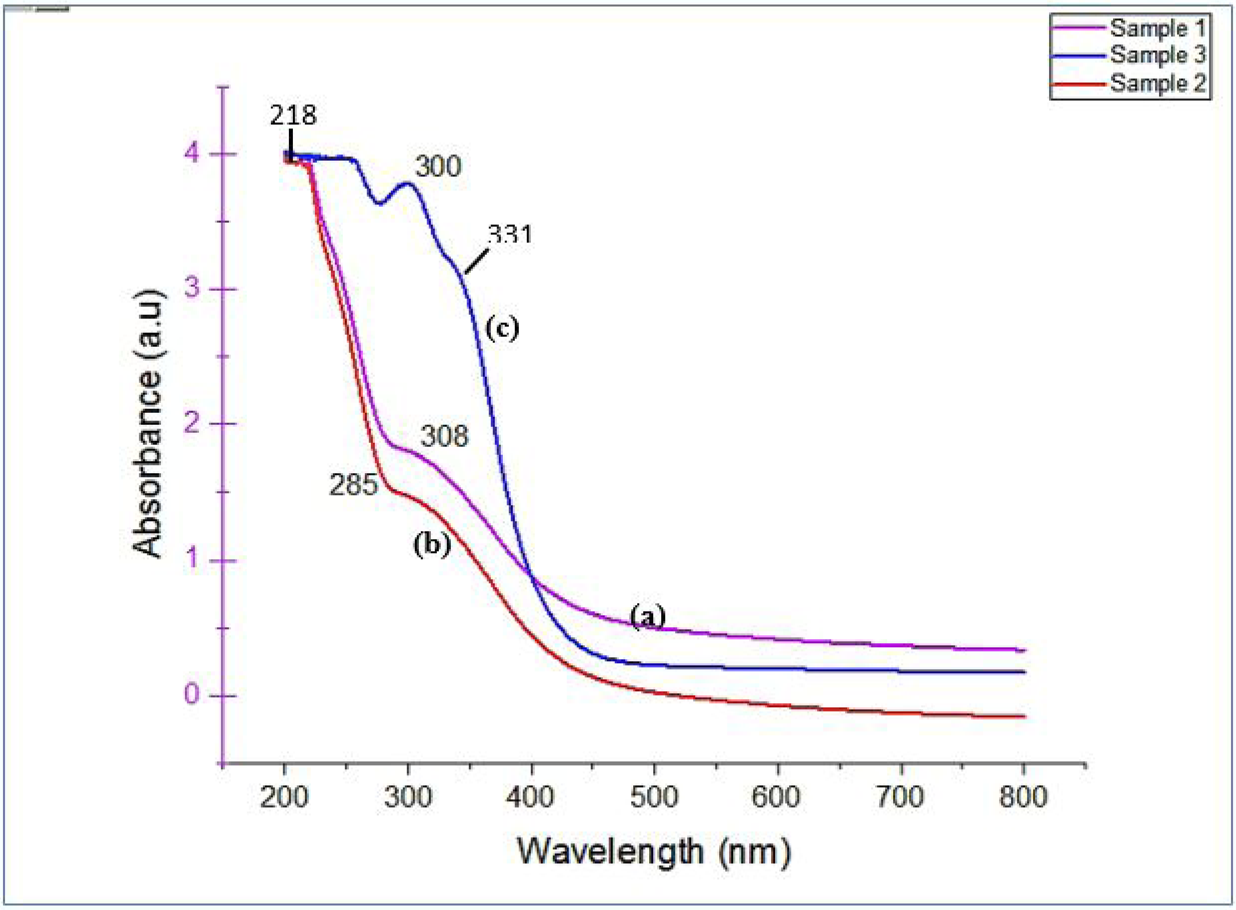

Figure 4: UV-Vis absorbance spectra for 3 different samples which are (a) sample 1 (M-GO), (b) Sample 2 (EFB-GO) and, (c) Sample 3 (OPS-GO)

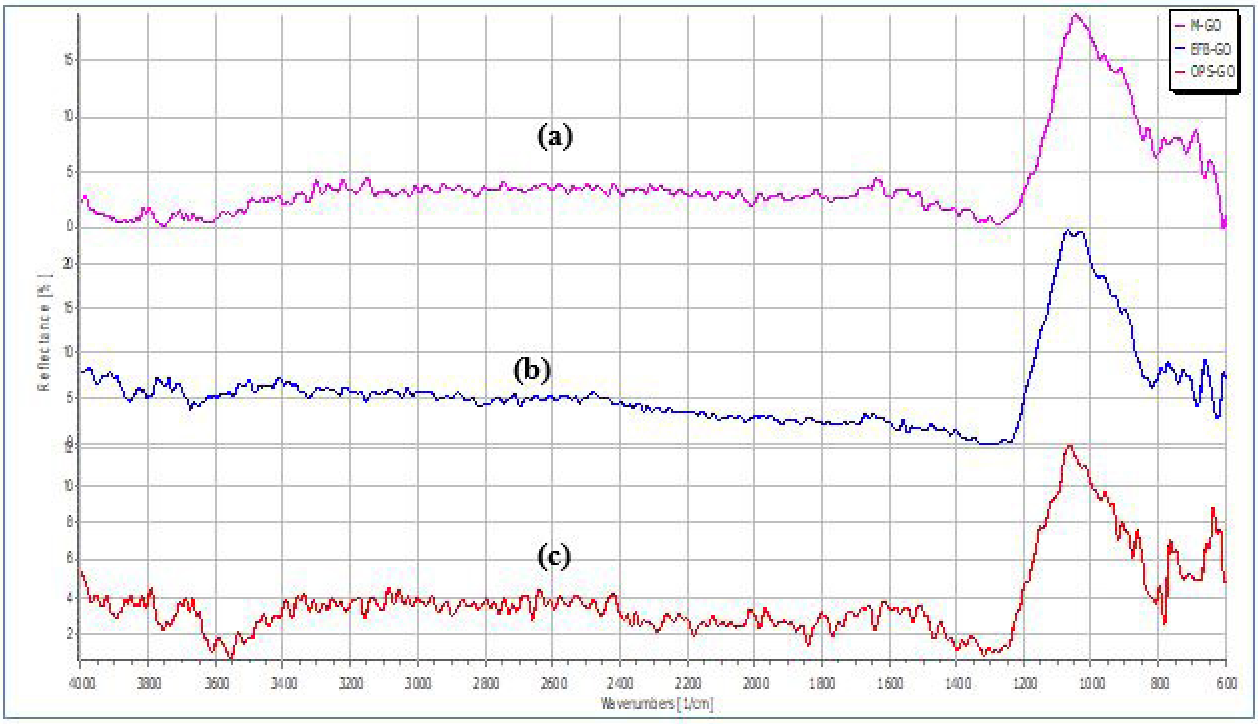

Figure 5: FTIR spectra of synthesis (a) M-GO, (b) EFB-GO and (c) OPS-GO in reflectance mode

Conclusion

The comparison of graphite oxide between M-GO, EFB-GO, and OPS-GO has been done and revealed that the biochar has undergone an oxidation process. From the results, the OPS-GO sample performed the highest oxidation as compared to M-GO and EFB-GO. The intensity peak of XRD appears at 24 °- 26 ° which indicated the GO is not fully oxidized due to the extensive volumes of carbon content in the biochar. The process was done by upgrading the Modified Hummer's method where it is simpler, affordable, environmentally friendly, and time-efficient.

Competing interests

The article confirms that there are no relevant financial or non-financial competing interests to report.

Acknowledgment

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