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# Reduction of reduced graphene oxide from synthetic graphite produced from oil palm trunk waste

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## ABSTRACT

Graphene has received great attention in various fields, including energy storage, electronics, gas sorption, separation, sensing, and catalysis fields due to its exceptional thermal, electrical, magnetic, optical, and mechanical capabilities as well as its substantial specific surface area. However, this super great graphene is derived from precursor materials, primarily graphite. Synthetic graphite produced from oil palm trunk (OPT) waste has been reported to have excellent chemical properties that are comparable to those of commercial graphite. Through the synthetic graphite from oil palm trunk (OPT) waste, graphene oxide (GO) can be obtained via the modified Hummers' method. The as-produced GO can be reduced to form reduced graphene oxide (RGO) through various processes such as thermal and chemical reduction. In this study, GO that was obtained from synthetic graphite via the modified Hummers' method undergoes chemical reduction to produce RGO. Sodium borohydride ($NaBH_4$) is used as the reducing agent in the chemical reduction. The RGO produced by the chemical reduction is expected to show all the characteristics comparable to those of RGO produced from commercial graphite. X-ray diffraction (XRD) analysis reveals a peak at 26° ($2\theta$) for all graphene oxide samples. RAMAN spectroscopy confirms the graphitic nature of the produced RGO, with observed D, G, and 2D peaks. Additionally, Fourier-transform infrared (FTIR) spectroscopy indicates the presence of functional groups such as amine, phenol, alkene, and alcohol in the RGO produced from synthetic graphite, demonstrating its similarity to RGO derived from commercial graphite.

**Keywords:** *Graphene, Reduced graphene oxide, Waste material, Oil palm trunk*

## 1. INTRODUCTION

Graphene, a single sheet or a few layers of $sp^2 – sp^2$-hybridized graphitic carbon has attracted a lot of attention in both technological and scientific fields because of its extraordinary chemical and physical characteristics. As a conducting semimetal, graphene has sparked a lot of interest in electronic research and applications. However, in utilizing graphene in electronic devices, mass synthesis of graphene with adjustable size and low cost remains a major problem. Different graphene manufacturing techniques result in materials with different characteristics. According to [1], since the initial achievement in graphene production using the "scotch tape" method, various techniques for graphene synthesis have been developed. Among these, graphene oxide (GO) and its reduced form, reduced graphene oxide (RGO), have been widely used as precursor materials in a significant portion of graphene-related research. Graphene oxide is a chemical compound composed of carbon (C), oxygen (O), and hydrogen (H). It was first produced in 1958 via Hummers' technique where graphite, potassium permanganate ($KMnO_4$), sodium nitrate ($Na_2NO_3$), and sulfuric acid ($H_2SO_4$) are used in a chemical process. Graphene oxide is a one-molecule-thick or few-layer-variant of the material graphite oxide (GO). The GO is not conductive, but it may be reduced to generate conductive RGO by chemical reactions, heat treatment, or a variety of other techniques as shown in Figure 1 [2].

GO can regain its graphene-like characteristics through additional reductive exfoliation treatments, which transform GO to RGO [3]. RGO offers an excellent balance between graphene and graphene oxide, as it retains graphene-like features such as high conductivity while being easier to prepare in desirable quantities from cost-effective GO using various methods.

Previous studies have reported that synthetic graphite derived from oil palm trunk (OPT) waste exhibits impressive chemical properties comparable to commercially available graphite in terms of its graphitic nature [4]. Researchers successfully synthesized synthetic graphite at a temperature of 800°C with a heating rate of 20°C/min [5]. The synthetic graphite produced was further exfoliated using a modified Hummers' method to synthesize graphene oxide (GO). Experimental characterization

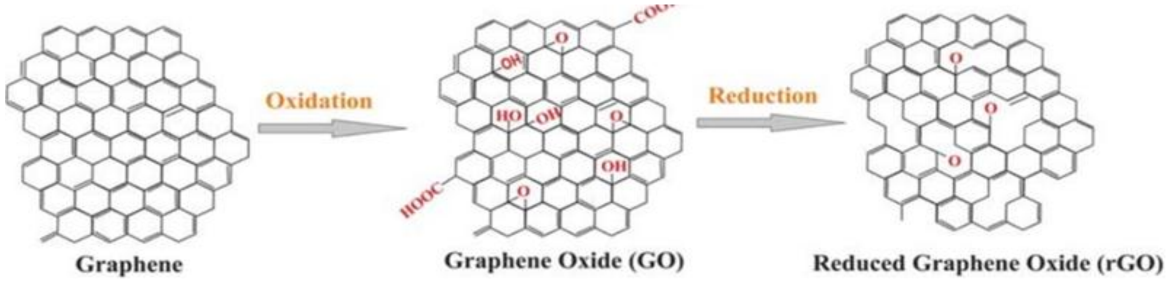

**Figure 1**. The chemical structures of graphene, graphene oxide (GO), reduced graphene oxide (RGO), and the conversion of graphene into GO and RGO via oxidation/reduction reaction [3]

confirmed that the obtained GO retained the expected properties of graphene oxide. Consequently, this GO can be further reduced to form reduced graphene oxide (RGO). The graphene-like properties of RGO make it a top-notch material for electronic applications [6, 7].

## 2. METHODOLOGY

### 2.1. Materials

The chemicals used in the exfoliation process to produce the GO are Nitrogen gas ($N_2$), sulphuric acid ($H_2SO_4$), potassium persulfate ($K_2S_2O_8$), phosphorus pentoxide ($P_4O_{10}$), potassium permanganate ($KMnO_4$), hydrogen peroxide ($H_2O_2$) and hydrochloric acid (HCl). All of the chemicals were strictly used without further purification. For RGO, Sodium Borohydride ($NaBH_4$) was used as the reducing agent, also without further purification.

### 2.2. Modification of Hummers Method

Synthetic graphite produced from oil palm trunk waste was further exfoliated using Modification of Hummers Method. The pre-oxidation of synthetic graphite involved the use of chemicals such as phosphorus pentoxide and potassium persulfate and sulphuric acid. The solution was stirred for almost 4 hours and left overnight before being filtered and proceeding with the oxidation process.

The oxidation takes place the next day after the filtration process. Pre-oxidized graphite undergoes further oxidation in an ice bath at 0°C to form graphene oxide. For pre-oxidize graphite, 120 mℓ of strong sulphuric acid and 15 g of potassium permanganate are added to a 1000 mℓ Schott bottle. The mixture is stirred at 800 rpm in an ice bath. The oxidation agents are added gradually and in moderate doses to prevent overheating. The temperature should remain below 20°C. If no reaction is observed based on temperature and heat generation, the mixture is stirred at a decreasing temperature of 35°C for two hours. After two hours, the mixture is diluted with 250 mℓ of DI water while maintaining the temperature at 35°C. The mixture is then left for two hours. After two hours, the mixture is diluted with 700 mℓ of DI water and 20 mℓ of $H_2O_2$ at a concentration of 30%. The temperature rises to 70°C, and the colour changes to bright yellow, indicating that the graphite oxide has been completely oxidised. The mixture is then filtered and washed with a solution of 1 ℓ of 1:9 Hydrochloric acid (HCI) to remove metal ions. The paste is subsequently collected on a filter membrane and weighed. The paste is dispersed in DI water by ultrasonication for 30 minutes, followed by centrifugation at 6000 rpm for 1 hour every cycle to neutralise the solution through a separation procedure. The centrifuge operation must be repeated at least six times. The acid is neutralised by adding DI water and stirring until the colour of the liquid is balanced. Centrifugation continues until no further separation is observed. The pH of the mixture is determined using pH paper to be 7. Once the desired pH is achieved, the graphene oxide solution is sonicated. To further exfoliate the graphene oxide, a 20-minute centrifugation at 4000 rpm is performed.

The GO that has been synthesized from synthetic graphite from the oil palm trunk (OPT) waste is taken to undergo chemical reduction with sodium borohydride (NaBH4) acting as the reducing agent [8].

The procedure for producing RGO began with a 100 mM sodium borohydride (NaBH4) and the addition of deionized water. The GO solution was added to the suspension after 10 minutes of magnetic stirring at 300 rpm. The resultant mixture was then treated with a reducing agent for 2 hours by magnetic stirring at 200 rpm. After 2 hours, an inhomogeneous brown suspension was formed. The RGO solution was then filtered using a vacuum filtering process. To remove metal ions, the mixture was then filtered and washed with a 1:10 HCl solution (1 litre), followed by 1 litre of deionised water to remove the acid. The resultant solid was air-dried and diluted to produce an RGO dispersion [9]. The sample produced was then characterized using XRD, RAMAN and FTIR.

## 3. RESULTS AND DISCUSSION

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

XRD analysis was performed using the Xpert Highscore Plus software and the resulting diffraction pattern is shown in Figure 2. The diffraction pattern was recorded at a scan range of 0 degrees to 50 degrees. From the diffraction pattern, the reduced graphene oxide (RGO) was found at 26° in 2θ with an intensity of 1052.4 a.u, confirming the presence of reduced graphene oxide in the sample. In Figure 3, a peak is also observed at 26° with an intensity of 704.64 a.u. The intensity of the RGO can be compared with that of GO to prove the reduction of GO. This comparison demonstrates that the reduction of GO is consistent with commercially available reduced graphene oxide [10].

Figures 4 and 5 show that the intensity of reduced RGO is lower than that of GO. The intensity of GO reaches 900 a.u, while RGO reaches an intensity of 750 a.u. This decrease in intensity indicates a successful reduction of GO.

### 3.2. Analysis of RAMAN spectroscopy

To support the earlier XRD analysis, RAMAN spectroscopy was performed. The presence of D, G, and 2D peaks in the Raman spectrum confirms the graphitic characteristics of the material.

Raman spectroscopy is a widely used non-destructive technique for analyzing the structural details of carbon-based materials. The G and D peaks, along with their overtones are the primary characteristics of the Raman spectra of graphitic carbon-based materials. The RAMAN spectrum of RGO derived from commercial graphite, shown in Figure 6, shows peculiar peaks: G band at 1611 $cm^{-1}$ and D band at 1340 $cm^{-1}$. Figure 7, which presents the Raman spectrum of RGO obtained from synthetic graphite, shows peculiar peaks designated as G band at 1599 $cm^{-1}$ and D band at 1352 $cm^{-1}$. While the G mode is created by the first-order dispersion of the $E_{2g}$ mode, the D mode is activated by the existence of defects and its strength is a measure for short-range disorder. To support the XRD analysis performed earlier, the reduction of GO is confirmed through the value of intensity (a.u) between the GO. $E_{2g}$ mode, the D mode is activated by the existence of defects and its strength is a measure for short-range disorder. To support the XRD that had been done earlier, the reduction of GO is proved through the value of intensity (a.u) between the GO.

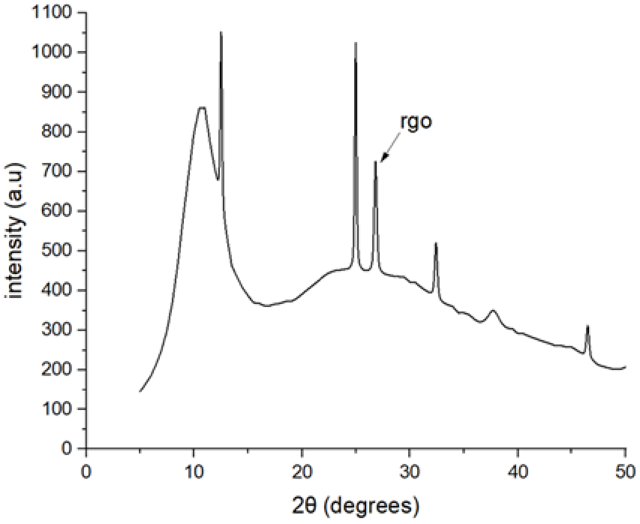

**Figure 2**. XRD analysis of reduced graphene oxide (RGO) from commercial graphite

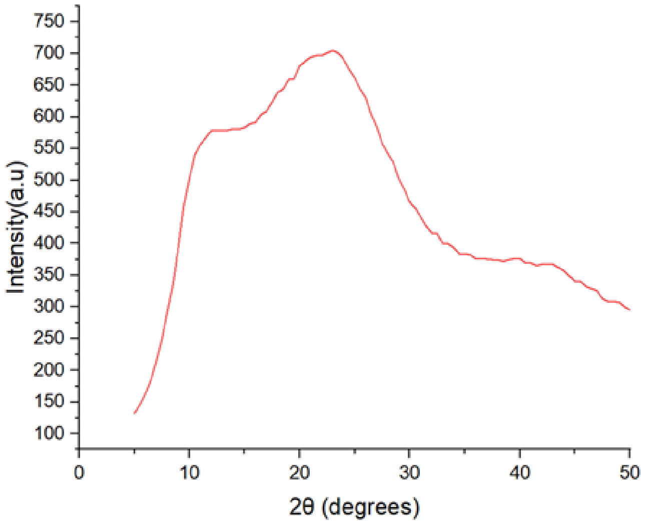

**Figure 3**. XRD analysis of reduced graphene oxide (RGO) from synthetic graphite

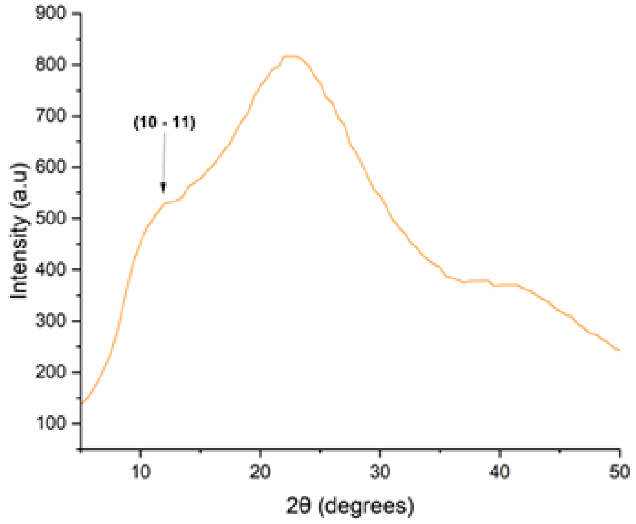

**Figure 4**. Graphene oxide (GO) from the synthetic graphite diffraction pattern

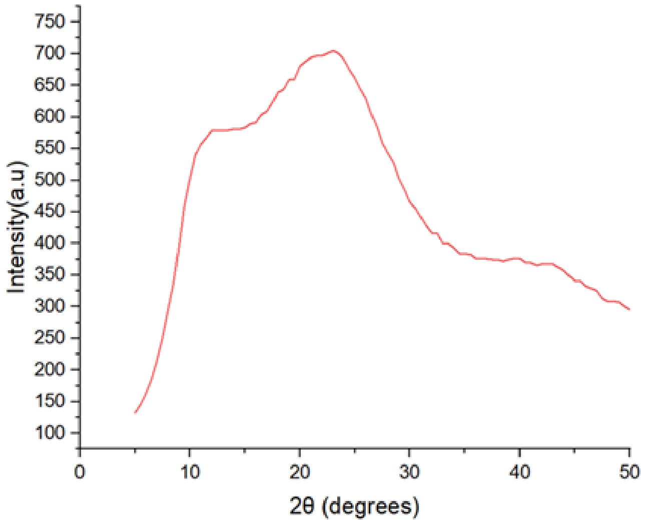

**Figure 5**. Reduced graphene oxide (RGO) from the synthetic graphite diffraction pattern

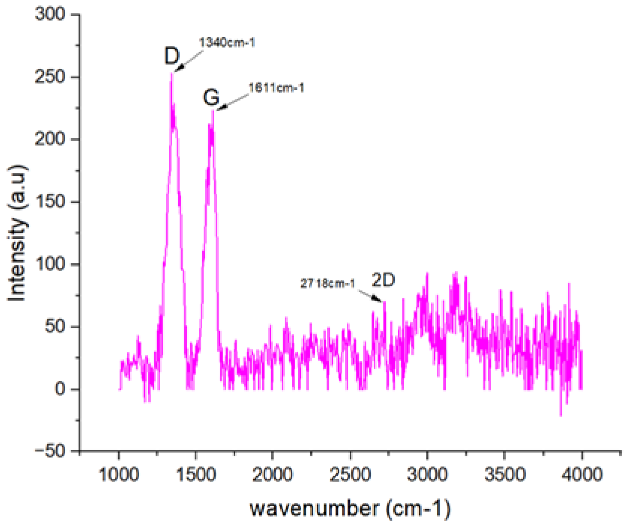

**Figure 6**. RAMAN analysis of RGO from commercial graphite

According to Figure 8, RGO shows a lower intensity value of 50 a.u, while the GO shows an intensity value of 100 a.u. This confirms the successful reduction of GO, as the result aligns with the XRD result [11].

### 3.3. Analysis of FTIR

One of the well-known analytical methods for determining if a substance is organic or inorganic and confirming the presence of a chemical composition in a sample is FTIR. In addition, this approach determines the existence of a certain functional group in the sample that was used.

**Table 1.** Typical vibrational frequency of functional groups in RGO prepared from synthetic graphite

| Bond | Functional Group | Wavelength ($cm^{-1}$) |
|---|---|---|
| C-N streching | Amine | 1118.00 |
| C-H bending | Alkene | 1385.00 |
| C=O stretching | Amide | 1631.50 |
| C=H stretching | Alkene | 2926.00 |
| N-H stretching | Primary Amine | 3430.19 |

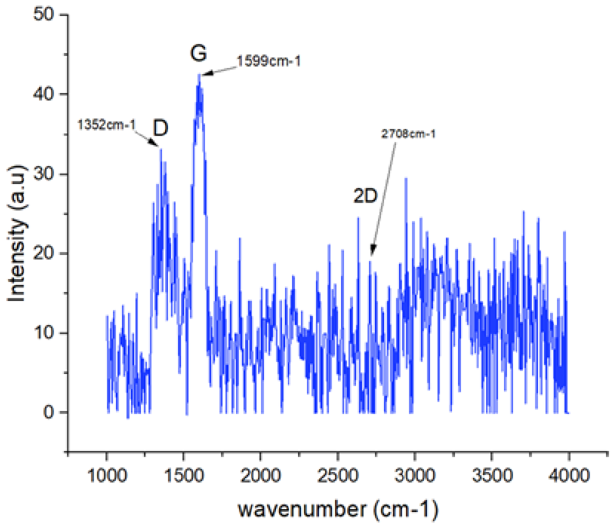

**Figure 7**. RAMAN analysis of RGO from synthetic graphite

Figures 9 and 10 show the FTIR analysis for RGO produced from synthetic and commercial graphite. The vibrational frequencies and functional groups of the synthetic graphite are summarized in Table 1 and Table 2 for each graphite. All of the peaks in the RGO prepared from synthetic graphite will be used as reference peaks and will be compared with peaks of GO prepared from synthetic graphite to confirm the success of the reduction of GO. As shown Figures 11 and 12, RGO exhibits fewer peaks than GO. Specifically, GO exhibits a CΞN stretching at 2302 $cm^{-1}$, whereas RGO does not. This absence of the C≡N stretching peak indicates the successful reduction of GO [12].

**Table 2.** Typical vibrational frequency of functional groups in RGO prepared from commercial graphite

| Bond | Functional Group | Wavelength ($cm^{-1}$) |
|---|---|---|
| C-N streching | Amine | 1099.20 |
| O-H bending | Phenol | 1386.00 |
| C=C stretching | Alkene | 1632.69 |
| O-H stretching | Alcohol | 2927.00 |
| N-H stretching | Primary Amine | 3414.05 |

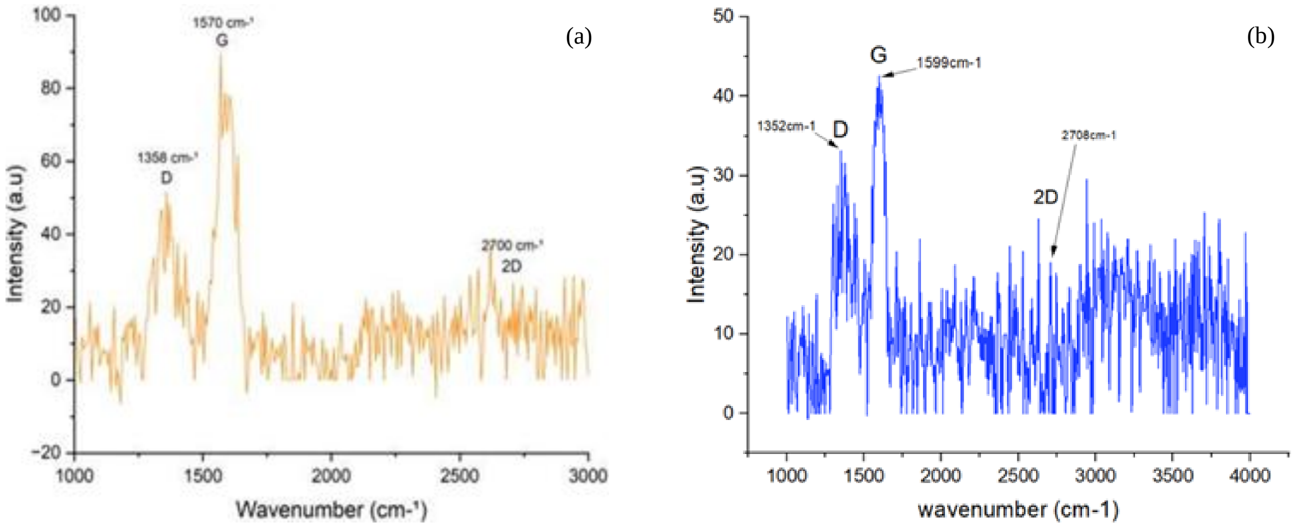

**Figure 8**. RAMAN analysis of (a) GO and (b) RGO

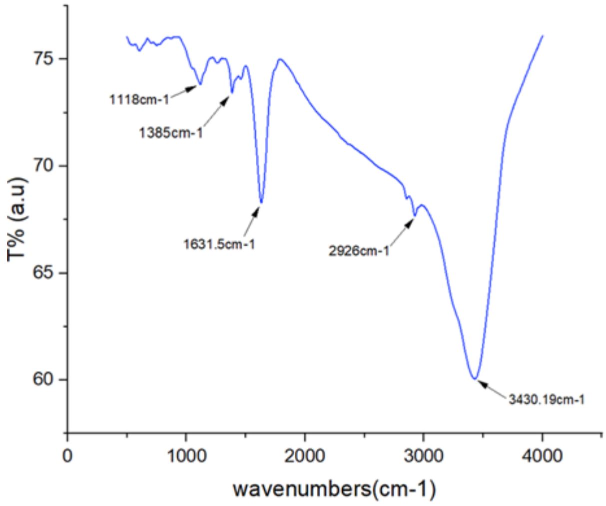

**Figure 9**. FTIR analysis of RGO prepared from synthetic graphite

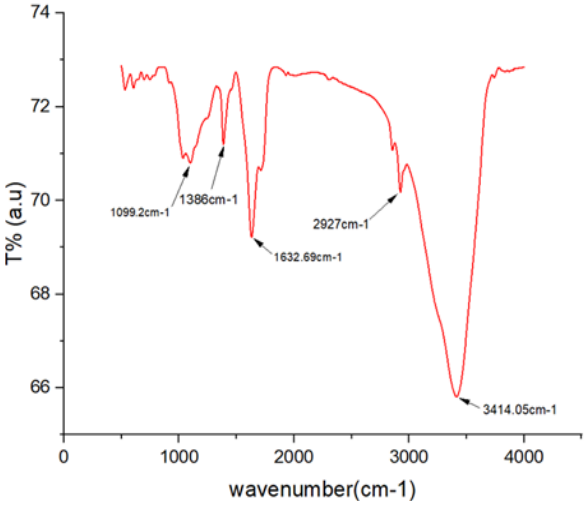

**Figure 10**. FTIR analysis of RGO prepared from commercial graphite

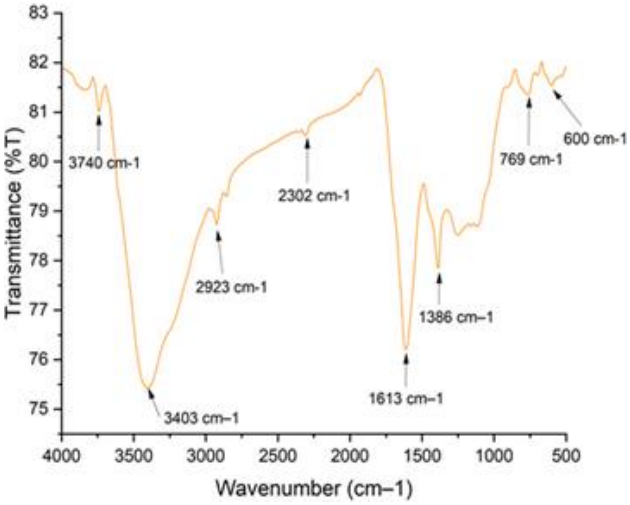

**Figure 11**. FTIR analysis of GO

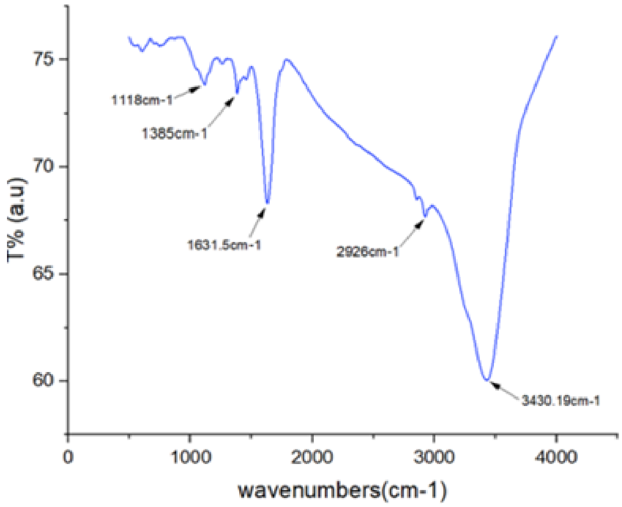

**Figure 12**. FTIR analysis of RGO

## 4. CONCLUSION

In this research project, the main objective was to produce RGO from GO derived from Oil Palm Trunk (OPT) waste. The reduction of GO that was prepared from synthetic and commercial graphite using sodium borohydride ($NaBH_4$) as a reducing agent was successfully achieved. The reduction process was confirmed through various characterization techniques, including X-ray diffraction (XRD), Raman spectroscopy, Fourier-transform infrared (FTIR) analysis, and scanning electron microscopy (SEM). The results were consistent with expectations. XRD analysis demonstrated the successful reduction of GO to RGO by revealing a peak at 26° and a lower intensity (a.u.) in RGO compared to GO. Raman spectroscopy further supported this finding, showing a decrease in intensity (a.u.) for RGO, indicating structural changes due to the reduction process. FTIR analysis confirmed a significant reduction in oxygen-containing functional groups in RGO compared to GO, further validating the success of the reduction process. Based on these results, it can be concluded that RGO was successfully synthesized from synthetic graphite derived from oil palm trunk waste.

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