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Analysis of Carbon Functional Groups through FTIR Patterns in The Preparation of Reduced Graphene Oxide (RGO)

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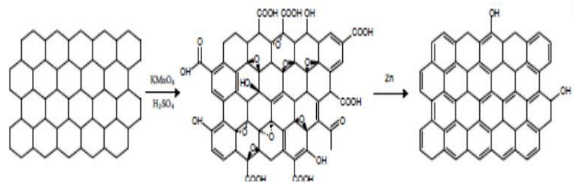
Abstract. The preparation of Reduced Graphene Oxide (RGO) through oil palm fiber biomass using a strong acid activation method with sulfuric acid (H_2SO_4) media, followed by application of microwave waves, is an interesting approach to obtain RGO. The study primarily focused on using the Fourier Transform Infrared (FTIR) spectroscopy technique to detect the formation of RGO based on changes in functional groups. The success of the formation of this RGO was confirmed by the presence of sharp and widening peak in the range of wave numbers 3254-3399 cm^{-1} which was OH-stretching. the longer the microwave wave is applied, the narrower the OH-stretching peak due to the removal of oxygen due to the application of microwave wave.

1 Introduction

Reduced graphene oxide (RGO) is an intriguing nanomaterial that has received a lot of interest recently because of its distinct characteristics and prospective uses in many different industries [1–3]. Graphene Oxide (GO), a derivative of graphene, a two-dimensional carbon material with a single sheet of carbon atoms organized in a hexagonal lattice, is the source of RGO [1]. The addition of oxygen-containing functional groups in GO results in a hydrophilic and dispersible material [4,5], making it more appropriate for some applications even though graphene has great electrical, mechanical, and thermal properties.

The process of reducing GO results in the removal or decrease of the majority of the oxygen functionalities and the restoration of graphene-like characteristics. In comparison to GO, this reduction technique gives RGO improved electrical conductivity and mechanical strength. Figure 1 shows the chemical bonding in (a) graphite, (b) GO, and (c) RGO [1]. RGO has consequently become a potential material for a variety of applications, such as electronics, energy storage, sensors, catalysis, and biological ones [6–9].



(a) (b) (c)

Figure 1. The chemical bonding in (a) graphite, (b) GO, and (c) RGO [1].

Researchers have concentrated on customizing RGO's features by managing the reduction process and adding particular functional groups in recent years, leading to the development of a variety of synthetic techniques. Using biomass from oil palm trees as a precursor in a strong acid activation process using sulfuric acid medium was one of the exciting ways to prepare RGO. This ground-breaking technique not only made use of a sustainable and renewable biomass source, but also provided the opportunity for RGO production on a wider scale, which could have favorable effects on the economy and the environment. Dayekh et al. [10] has been successful in producing RGO as a candidate for a photocatalyst material by synthesizing it in acidic environments up to pH=6. However, since the carbon source was derived from industrial products, carbon reduction was simpler. Moreover, Qazi et al. [11] had studied RGO as an excellent catalyst for the removal of dyes in real conditions. In this study, graphite powder was prepared by 10:1 volume ratio and 100 mL of hydrofluoric acid

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(HF) for the purification process. But according to earlier research, RGO hasn't received any additional care beyond being prepared in acidic media. Therefore, the impact of microwave treatment as a relatively straightforward RGO preparation method will be investigated in this work.

In this study, we report a thorough evaluation of the production of RGO from biomass derived from oil palm fiber utilizing the sulfuric acid activation method. To identify changes in functional groups and confirm the effective reduction of GO to RGO, we will concentrate primarily on the characterisation of RGO using FTIR spectroscopy. Through the use of plentiful biomass resources, the generated RGO has the potential to significantly advance numerous technological applications and could support sustainable development.

2 Experimental

Biomass from Aceh's oil palm plantations was used in this study which no longer used. Whereas the other materials that we used were sulfuric acid (H_2SO_4). First, palm fiber was cleansed with distilled water. The sample was then dried in an oven for 24 hours at 110°C. Grinding and sifting were used to purify dry samples into small particle sizes. Additionally, samples of palm oil fiber with small particle size underwent a 24-hour chemical activation process using 2 M sulfuric acid (H_2SO_4). The precipitate, which has now taken the shape of a gel, was filtered after which it was neutralized with distilled water to a pH=6 in order to eliminate H^+. Activated charcoal was prepared by carbonizing the neutralized gel at 600 °C for one hour. The activated charcoal was then reduced to a powder by being crushed in a mortar.

The activated carbon was subsequently heated with microwave radiation at the various time, i.e., 0, 10, and 20 minutes. Here, we prepared 1 gram carbon in 100 ml H_2O. The aim of using microwave waves was to successfully generate RGO by reducing the formed graphene oxide. The interactions and alterations in functional groups from carbon bonds were then studied using FTIR characterisation to assess the efficacy of this synthesis.

3 Results and Discussion

The preparation of RGO from carbon derived from palm fiber has been successfully synthesized. This was shown by Figure 2.

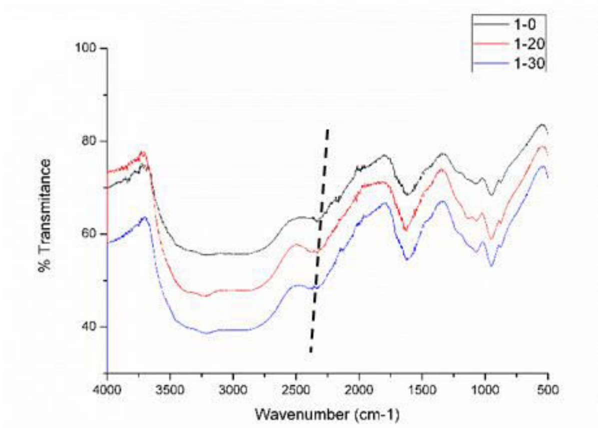

Figure 2. The FTIR pattern of RGO samples in various time (0, 10, and 20 minutes).

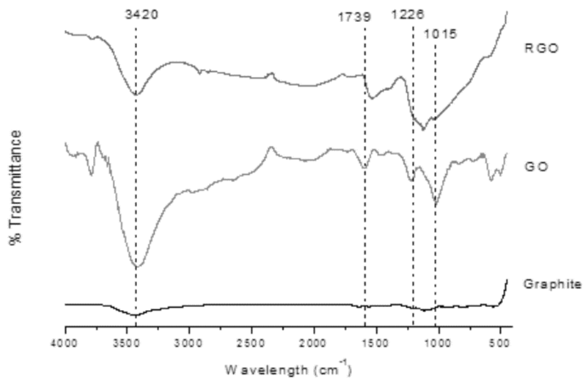

Figure 3. The FTIR pattern of Graphite, GO, and RGO from previous study [1].

Table 1. Functional group assignments of Fourier Transform Infrared (FTIR) Spectroscopy from RGO samples.

Functional group Assignment	**Sample 1-0**	**Sample 1-20**	**Sample 1-30**	**Refs** [1,3, 5,12,13] **(cm^{-1})**
O-H bonding	3254	3267	3399	3420
C=O stretching	1622	1624	1624	1739
C-O-C Stretching	1065	1069	1179	1226
C-O-C Stretching	951	949	958	1015

A similar transmission peak pattern is depicted in Figure 2 as well. When examined more closely, the resulting FTIR peaks are thinner the longer the microwave heating period is. The shrinking of the peaks around the wavenumber of 3500–3000 cm^{-1}, which is one of the prerequisites for the formation of RGO, is demonstrated by our data (Figure 2 and Table 1), which are in line with earlier studies (Figure 3) [14–17]. This transmission peak's shrinking suggests that there is less oxygen in GO.

When the acid-treated biomass is subjected to oxidation, it leads to the formation of Graphene Oxide (GO). GO is an oxygen-functionalized form of graphene with various oxygen-containing groups, including epoxides, hydroxyls, and carboxyls. The obtained GO is then subjected to microwave waves. The application of microwave waves is expected to reduce GO and produce Reduced Graphene Oxide (RGO). During this reduction process, oxygen-containing functional groups are removed or reduced, converting GO to RGO. After the reduction step, the FTIR spectroscopy is employed to analyze the functional groups present in the obtained RGO. One of the key features of interest is the OH-stretching peak, which is characteristic of the hydroxyl groups (-OH) present in both GO and RGO.

The successful formation of RGO is indicated by changes in the OH-stretching peak in the FTIR spectrum. Specifically, the narrowing of the OH-stretching peak in the FTIR pattern shows a reduction in hydroxyl groups in the RGO structure compared to GO. This reduction of oxygen-containing functional groups confirms the successful conversion of GO to RGO.

In contrast, the strong and broad peak for GO at 3399 cm^{-1} wavelength reveals the presence of an O-H bond (hydroxyl group). Additionally, stretching C-O-C (epoxy group) is seen at wavelengths of 1179 and 958 cm^{-1}, whereas stretching -C=O (group -COOH) is visible at 1624 cm^{-1}. It is confirmed that molecular oxygen (O) is more occupied at the margins and basal planes of GO than RGO in the presence of all these carboxylic, hydroxyl, epoxide, and carbonyl groups than RGO.

When the sample is heated in microwave for a longer period of time, the peak changes more toward a higher wave number, as seen by the dotted line in Figure 2. The closer the peak value is to the RGO peak value in the reference (Table 1), the higher it is. Sample 1-20 with 20 minutes heated in microwave show the best characterization. This finding supports the idea that oxygen may be removed by microwave heating, which also makes the RGO functional groups sharper and more similar to the values in the reference.

4 Conclusion

Synthesis of RGO from oil palm fiber biomass has been successfully carried out using the acid and microwave heating method. The results of microwave heating proved to be able to reduce the GO peaks to become narrower and close to the RGO peak values according to the reference, with the highest RGO peak value at 3399 cm^{-1} in samples 1-20 which was caused by the reduction of oxygen during microwave heating.

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