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# Evaluating the Morphological and Functional Properties of Nano Graphene based on Synthetic Graphite Obtained by Modified Hummers Method

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## ABSTRACT

In this study, graphene oxide was synthesized by modified Hummer's method. The structural and elemental composition of the samples were observed by high resolution scanning electron microscopy and energy dispersive x-ray analysis (SEM and EDX). The nature of the functional groups and the electrical characterization were analyzed by Fourier transforms infrared (FTIR) spectroscopy and four-point probing system. The micrograph of the thin film on gold substrate revealed a porous spongy structure, a flate-like surface in layers. EDX pattern showed peaks corresponding to carbon C (86.34%), Molybdenum (10.37%), Potassium (2.38%) and Titanium (0.91%) in K shell. The FTIR indicated that oxidized graphite has an aromatic, carbonyl, carboxyl, epoxy and hydroxyl groups. The strong band at 2363.50 $cm^{-1}$ and 1559.32 $cm^{-1}$can be attributed to the stretching vibrational mode in carboxyl, carbonyl groups and un-oxidized graphitic domains. This result has revealed that the sample is electrically conductive due to the delocalized pi-electrons at the basal plane and the high electronic conductivity at room temperature. It is suitable for integration in designing photovoltaic cells by improving the efficiency, production cost, and life time.

**Keywords:** Electrical properties, Hummer's method, 2D Graphene, Carboxyl, Epoxyl, Hydroxyl Groups, PVCs

## INTRODUCTION

The two-dimensional (2D) allotrope of carbon has hexagonal pattern. The electronic and thermal conductivity of graphene is very high [1]. The oxidized form known as graphene oxide (GO) has carboxylic acid, epoxy and hydroxyl groups. Carbon molecules can absorb light energy in the UV-visible range at different wavelength [2]. These special structures cause GO to be electrically conductive [3 − 5]. There are several electron and hole transport layer such as PCBM, fullerene $C_{60}$, $TiO_2$, ZnO and spiro-OMeTAD, $P_3HT$, $MoO_2$, and $NiO_x$ which are vital in solar cells and not all have been tested with graphene [9 − 10].

The quest for clean and sustainable energy worldwide has been on the increase due to higher energy demand, rapid population growth and industrial consumption [1,6]. Excessive use of fossil fuel creates serious environmental complications, global warming, and atmospheric pollution. There is growing need for alternative sources of energy from solar, hydro, wind and geothermal energy which are renewable [7]. United Nations advocate for clean and safe alternative energy system that is cost-effective. This is a major challenge in order to reduce dependence on petroleum production for energy uses. The huge flux of photons from the Sun on the earth is massive about 10,000 times higher than the current world's energy production and if

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properly harnessed solar power can solve the energy crisis[8].

Nowadays, the creation of transparent and flexible electronics for applications in smart windows, integrated circuit (IC) cards, displays, LEDs, solar cells, and electronic notebooks are heavily being investigated. 2D materials are need electrode in batteries, capacitors, and supercapacitors. It is suitable in the fabrication of graphene-electrode in electronic devices such as Field-effect transistors, memory devices, solid state molecular electronic equipment, and optical devices in the form of liquid crystal displays (LCD), touch screen (TS), light-emitting diodes (LEDs) and in different types of graphene-based organic and inorganic solar cells (SCs) [20]. In this study, GO samples were prepared by Hummer's method were characterized by Fourier transform infrared (FTIR), and UV-visible spectroscopy, high resolution scanning electron microscopy and energy dispersive x-ray analysis (SEM and EDX). The electrical activities of the samples were explored using a four point probing system to determine their conductive properties.

## MATERIALS AND METHODS

### Synthesis of Graphene Oxide

The GO sample was prepared by modified Hummers method by mixing 5g of graphite, conc. $H_2SO_4$ and $HNO_3$ in a 500ml beaker. After sonication, heating and dilution with 200 ml distilled water, the reaction was stopped by addition of 30 ml $H_2O_2$ (30%) to form was a golden yellow colour of GO. 0.5g of the synthesized GO was coated on gold substrate to produce GO thin films by thermal process.

Plate 1: Synthesized GO in solution and lump form after centrifugation at 3000 rpm

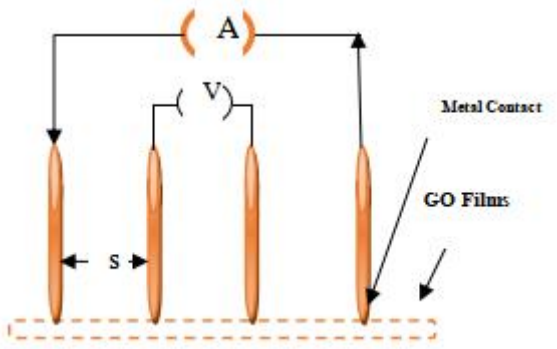


**Figure 1:** Electrical characterization of GO thin films using four point probe

### Characterization

The nature of the functional groups present in the samples were analyzed using FTIR spectroscopy (model: Nicolet i-S50) to scan the specimen from 650 to 4000 $cm^{-1}$ at raw material research labouratory, Abuja. The SEM/EDX analysis was observed on EVO LS 10-electron microscopy (AUST, Abuja) at the magnification of 10-100x with accelerating voltage of 20 kV, 10mA, photon no. 4654 and the energy dispersive X-ray (EDX) on an Amatek device for the elemental composition.

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### Electrical Properties

Electrical characterization of the thin film was performed using four point probe (4200 Keithley instruments) at room temperature. By assuming that the metal tip is infinitesimal and the samples are semi-infinite. Measurements of thickness by atomic force microscopy (AFM) showed that the thickness of graphite oxide after the reduction to few layer graphite decreases from about 10-20 nm to 5-6 nm (Neustroev et al., 2015); $[15-17]$ (figure 1).

The resistivity $\rho$ of the graphite thin films were determined by

$$\rho = R_S t$$

Where $R_s$ is the sheet resistance and t is the films thickness. The determination of the thickness by atomic force microscopy (AFM) is about $\sim$ 10-6 nm for few layer graphene (Neustroev et al., 2015).

$$R_s = \frac{\rho}{t} = \frac{\pi}{ln2}\left(\frac{V}{I}\right) = K\left(\frac{V}{I}\right) = \left(\frac{V}{I}\right) 4.532$$

Where V is the measured voltage, I is the current and K is the geometric factor which depends upon the configuration of the probes (K = 4.532).

## RESULTS AND DISCUSSION

### FESEM Surface and Structural Morphology

The surface morphology of GO thin film revealed a porous spongy structure with the graphene sheet not well connected together, a flate-like surface in layers. This shows that graphite has been exfoliated during the oxidation process. It can be attributed to the distorted graphene sheets when oxygen and other functional groups attached to the carbon atoms. GO sample looks clustered $[3-4]$ (Figure 2).

### Energy Dispersive x-ray (EDX) Analysis

The energy dispersive x-ray (EDX) analysis of GO thin films on gold substrate showed the peaks corresponds to carbon C (86.34%) for electrons in the K shell, Molybdenum Mo (10.37%) L shell, Potassium (2.38%) K shell and Titanium (0.91%) in K shell (table 1). The presence of phosphorous, sulphur, and potassium were due to $H_2SO_4$ and $KMnO_4$ used as oxidizing agent while silicon, magnesium and sodium were obtained from the glass substrate [14,17] (Figure 3).

### Fourier Transforms Infrared Spectroscopy

Fourier Transforms Infrared (FTIR) spectroscopy was used to identify the bonding interactions and functional groups present in GO. The percentage transmittance on the y axis showed how strongly light is being absorbed at each frequency while the IR wavelength in term of the wave number $cm^{-1}$ was indicated on the x-axis. The results have revealed that GO consist of an aromatic (C=C), carbonyl (C=O), carboxyl (COOH), epoxy (C-O-C) and hydroxyl (O-H) functional groups. The presence of oxygen containing functional groups confirm the oxidation of the synthetic graphite to GO. The strong band at 2363.50 $cm^{-1}$ can be attributed to the stretching vibrational modes of the C = O group in the carboxylic acid and carbonyl groups. The peak at 1559.32 $cm^{-1}$ can be due to the skeletal vibrations of un-oxidized graphitic domains. The band at 1019.88 $cm^{-1}$ is assigned to the epoxy groups, C – O and 3423.05 $cm^{-1}$ represents O-H stretching vibrations reported in literature [17] while 481.41 and 410.58 $cm^{-1}$ can be due to bonding configuration. This result shows that GO is highly conductive due to the delocalized $\pi$-electrons at the basal plane of GO $[3, 18]$ (figure 4).

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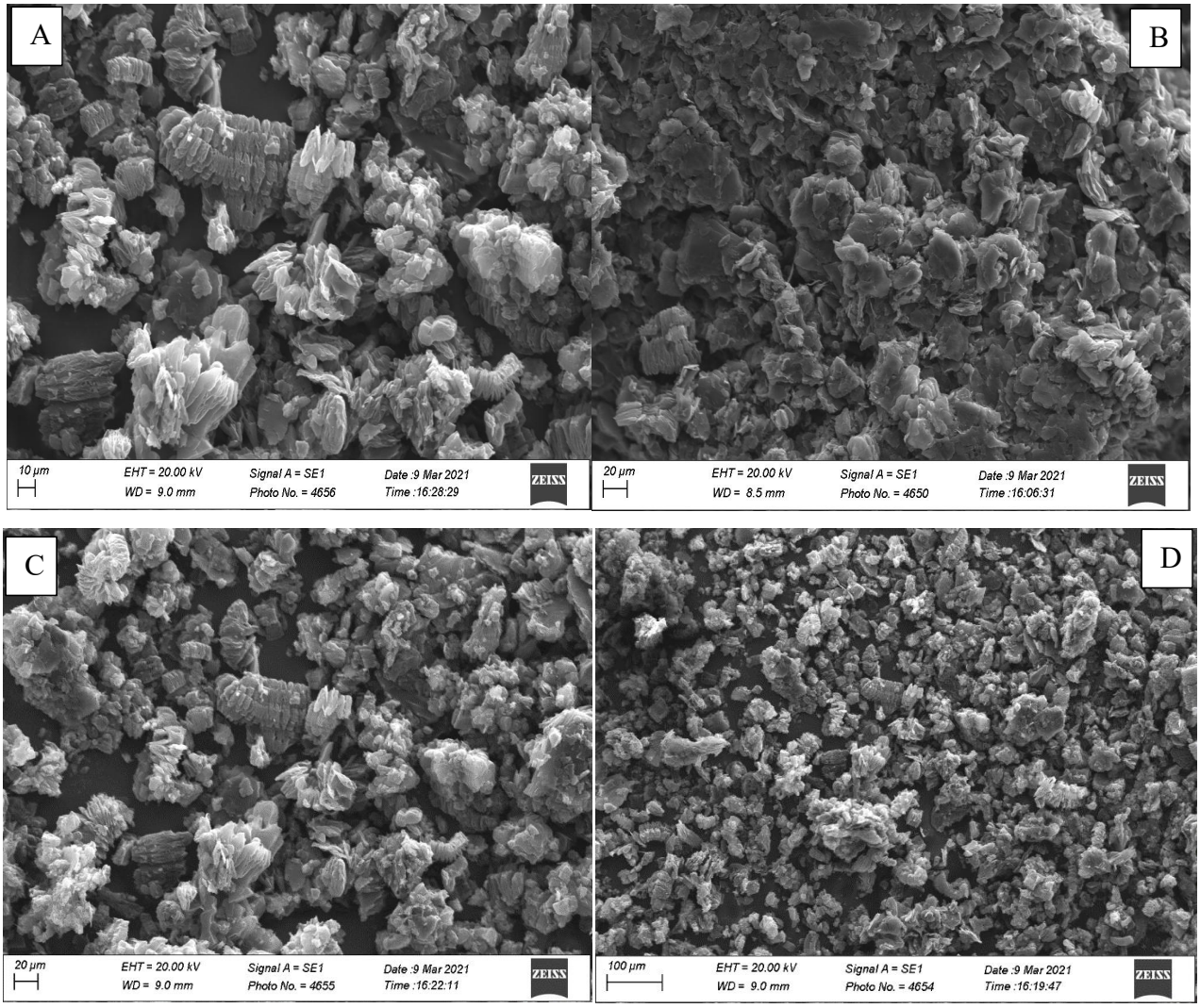


**Figure 2:** SEM micrograph of the GO working distance of (a) 10$\mu m$; 9.0 mm (b) 20$\mu m$; 8.5 mm (c) 20$\mu m$; 9.0 mm (d) 100$\mu m$; 9.0 mm.

**Table 1:** EDX analysis of GO

| Element | Weight % | Atomic % | Error % | Net Int. | K Ratio | Z | R | A | F |
|---|---|---|---|---|---|---|---|---|---|
| C K | 74.19 | 88.74 | 99.99 | 35.04 | 0.378 | 1.0495 | 0.9632 | 0.4855 | 1 |
| O K | 8.8 | 7.91 | 30.03 | 2.62 | 0.0093 | 1.005 | 0.9841 | 0.1051 | 1 |
| Na K | 0 | 0 | 99.99 | 0 | 0 | 0.9137 | 1.0092 | 0.4071 | 1.0031 |
| Mg K | 0 | 0 | 99.99 | 0 | 0 | 0.9295 | 1.0164 | 0.5805 | 1.0055 |
| Al K | 0.05 | 0.02 | 99.99 | 0.19 | 0.0003 | 0.8954 | 1.0232 | 0.7321 | 1.0097 |
| Si K | 0.26 | 0.13 | 70.12 | 1.27 | 0.002 | 0.9153 | 1.0295 | 0.8452 | 1.0155 |
| Mo L | 12.58 | 1.88 | 9.09 | 28.93 | 0.1077 | 0.7003 | 1.2299 | 1.2051 | 1.0146 |
| S K | 0 | 0 | 99.99 | 0.02 | 0 | 0.8968 | 1.0412 | 0.9703 | 1.0217 |
| K K | 1.53 | 0.56 | 35.86 | 4.98 | 0.0125 | 0.8496 | 1.0562 | 0.9454 | 1.0176 |
| Ca K | 0.52 | 0.18 | 60.74 | 1.46 | 0.0044 | 0.8653 | 1.0606 | 0.9624 | 1.0224 |
| Ti K | 0.5 | 0.15 | 61.52 | 1.21 | 0.004 | 0.7847 | 1.0683 | 0.991 | 1.0415 |
| Mn K | 1.57 | 0.41 | 58.68 | 2.53 | 0.0129 | 0.7605 | 1.0769 | 1.0078 | 1.0728 |

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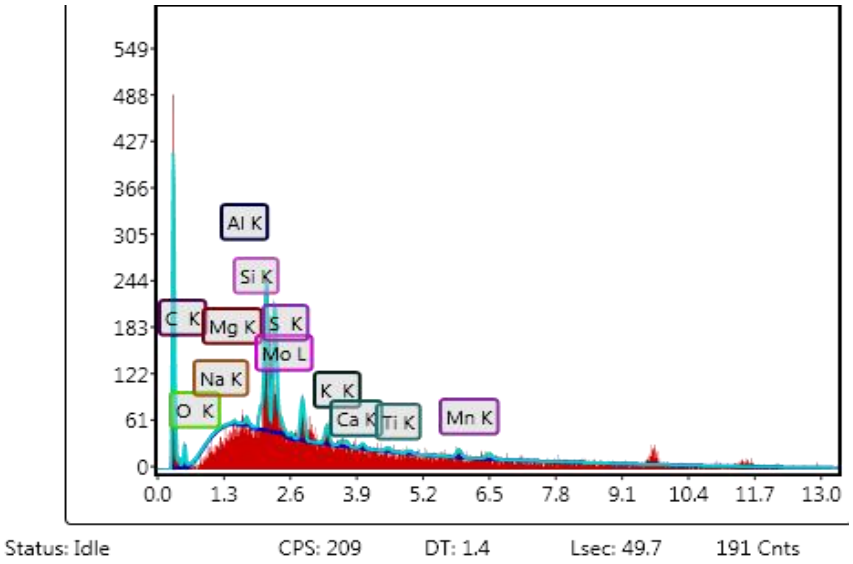


**Figure 3:** EDX micrograph of GO showing the elements and the elemental counts

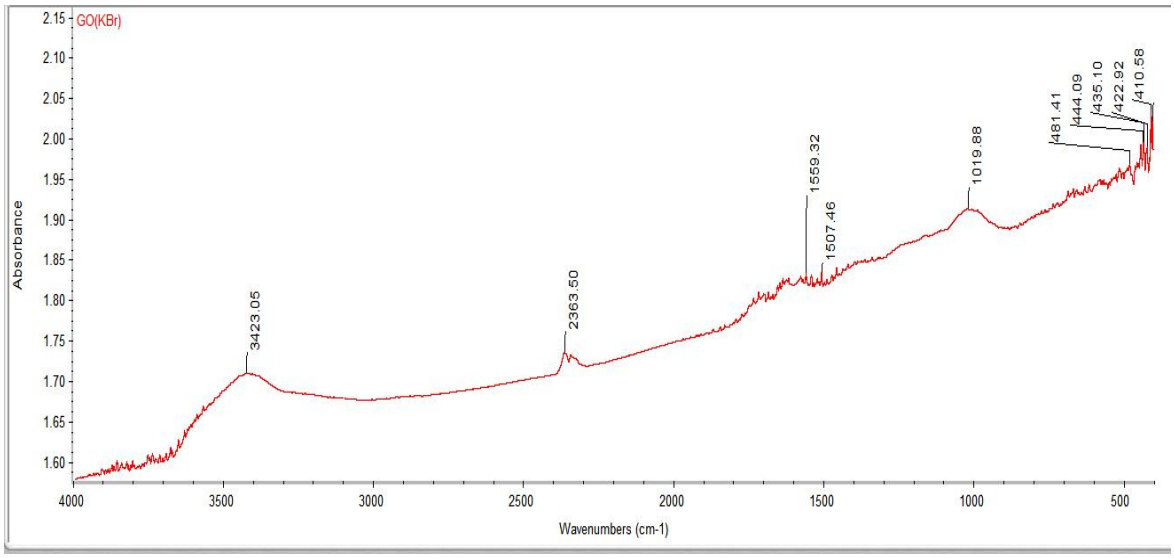


**Figure 4:** FTIR spectrum of showing the absorbance versus wave numbers GO powder

## Electrical Characterization

The results of the conductivity measurement for GO and RGO thin films are presented in table 2 and is compared with similar studies that are reported in the literature. The application of graphene films in transparent electrodes is governed by the $R_s$ values, absorption and transmission rate to light energy. Each of these criteria has its own unique requirements in improving the optoelectronic properties which can be fine-tuned to the desired value by changing the thickness, doping and composite formation. The smaller the value of $R_s$, the higher the transmission of light through it by enhancing the contrast ratio. Doping improves the stability and decreases the energy band gap if optimized as in a typical solar cell[17, 20 – 21 ].

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**Table 2:** The conductivity of the GO and RGO thin films

| Graphene films | Thickness (μm) | V(mV) | I(A) x $10^{-9}$ | $R_S$ ( Ω/sq ) x $10^6$ | ρ (Ωm) | $\frac{1}{\rho}$ S/m | References |
|---|---|---|---|---|---|---|---|
| GO | 0.022 | 28.0 | 5.47 | 23.20 | 0.5104 | 1.9592 | This study |
| RGO-S | 0.015 | 53.0 | 90.65 | 2.65 | 0.0398 | 25.1256 | This study |
| GO-NGF | 0.027 | 25.0 | 4.95 | 22.90 | 0.6183 | 1.6173 | [17] |
| RGO-NGF | | 50.0 | 45.69 | 4.95 | 0.1330 | 7.5188 | |
| RGO | 0.025 | - | - | 0.0179 | - | 23.30 | [19] |
| G optimized | - | - | - | 400.0 | - | - | [22] |
| G unoptimized | - | - | - | 30.0 | - | - | [22] |

## CONCLUSION

The surface morphology of GO revealed by SEM analysis showed a porous spongy structure with the graphene sheet not well connected together, a flate-like surface in layers. The EDX analysis revealed peaks due to presence of carbon C (86.34%), Molybdenum Mo (10.37%), Potassium (2.38%) and Titanium (0.91%). Fourier Transforms Infrared (FTIR) spectroscopy results indicated that GO consist of an aromatic (C=C), carbonyl (C=O), carboxyl (COOH), epoxy (C-O-C) and hydroxyl (O-H) functional groups. The strong bands can be attributed to the stretching and skeletal vibrational modes of the carboxylic acid, carbonyl groups, and un-oxidized graphitic domains respectively. This result has revealed that GO is highly conductive due to the delocalized pi-electrons at the basal plane as revealed by FTIR spectroscopy and high electronic conductivity at room temperature suitable for integration in designing photovoltaic cells. It will improve efficiency, reduce production cost, and enhance life time of the device.

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