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SYNTHESIS AND CHARACTERIZATION OF GRAPHENEOXIDE FROM *HYPHAENETHEBAICA* (DOUMPALM) SHELL

[1]Hassan L.G., [1]Liman, M.G.,[2] Ibrahim M., [1*]Yahaya, A, and [3]Babagana A.
[1]Department of Pure and Environmental Chemistry, UDUS, Sokoto Nigeria
[2]Department of Chemistry, Sokoto State University, Sokoto Nigeria
[3] Department of Chemistry, Borno State University, Maiduguri Nigeria
Corresponding Author's Email: auduyahaya776@gmail.com

Abstract
Graphene oxide (GO) is a significant carbon component of two dimensional nanomaterial with unique features and wide range of application owing to its physical, chemical and electronic properties. The primary aim of this work is synthesis and characterization of graphene oxide. The graphene oxide was prepared by Hummer's method using grapheme synthesized from Hyphaenethebaica shell (Doumpalm shell) as carbon based material. The characterization of GO was carried out using X-ray Diffraction Analysis(XRD),Fourier Transform Infrared (FTIR), Thermogravimetric analysis (TGA),Scanning Electron Microscopy (SEM) and Brunauer Emmett Teller (BET). The XRD images howed the formation of grapheme oxide from a strong peak at angle19.6º revealing its crystalline structure and indicates that the chemical reaction had an important role in the formation of the GO particle. This was confirmed by the FTIR, where the presence of various oxygen functional groups were widentified. The FTIR band showed that the GO had some functional oxygen group within its structure. The SEM image showed the morphological structure of GO with rough surface, while the BET data result proved that the graphene oxide has a large surface area (356.276 m^2/g), pore volume (2.15 m^3/g) and poresize (2.138 mm).
Keywords: *Graphene Oxide, Hyphaenethebaica, nanomaterial, synthesized, thermalstability.*

Introduction

Doum palm fruit which is commonly called *Goriba*in Hausa language in Northern Nigeria, display a very promising alternative source of biomass for the production of graphene oxidevia graphene. Doum plant (*Hyphaenethebaica*) is a perennial savannah palm tree which belongs to most extensively cultivated plant in the Areca ceae family (Orwa *et al*., 2009). The doum palm fruit contain a very hard ball like nut while the unripe fruits contain a very soft nut, the fruit mesocarp, which is known and consumed in Northern Nigeria.

In some African countries, they prepare the fruit either in pieces or in a powdered form, which is further dried and added to food as aromatizer. The powder made from them esocarp of the fruit is added to water and milk and left to stand for some hours to make an alcoholic drink, the stem is also used form making palm wine and the fruit mesocarp is eaten raw as medicine to control hypertension (Orwa*etal*., 2009).

The hyphaenetherbaica shell can be burned directly or utilize as a fuel for gas engines (Mohammed *et al.,* 2022) and a source of activated carbon which has a good adsorbent features that can be used for waste water treatment (Ogwuche, *et al*., 2015).

Hyphaenethebaica (Doumpalm) fruits are mostly found in the semi-arid part of Nigeria. Doum plant are used locally as a source of income, it was sold and distributed to most part of the country which are being used as fruit and medicine (Fatemaetal.,2022).The Doum palm leaves and fruits are very effective for their antioxidant, anticancer and anti-Inflammatory potencies (Hossam *et al.,* 2017). It is also used in solving many health problems such as fertility booster, analgesic, anti-pyretic, constipation, hypertension, bilharzias and also in creases the amount of hemoglobin in the body (Elmahdy *et al.,* 2022). The water extract of doum fruit can reduce hyperlipidemia in nephros is disease and lead to decrease the risk of glomerulosceleros is and atherosclerosis (Aboshora *et al.*,2014).The shell of doum plant is a

bio-waste which causes land pollution and contribute to economic waste. Using bio-waste to produce grapheme oxide is a great move to create economic value out of waste material (Ludwika *et al.*, 2020). The properties of graphite derivatives have been thoroughly revised in the scientific society for the purpose of developing various applications. Graphene is a single layer of carbon atoms organized in hexagonal rings of an aromatic electron structure. Graphene oxide (GO) is a graphene derivation distinctive by the presence of plentiful oxygen functional groups on the graphene surface. The presence of defect or disorder in the graphene structure leads to many unusual characteristics of graphene oxide (GO) (Ludwika *et al.*, 2020). Recently, agreat attention and concern in graphene related research have been increased due to its ability to remain exfoliated in water as a single atomic layer sheet, cast as films, which can be further reduced in a reciprocal manner to graphene. GO has been successfully tested in many applications such as electronics, conductive films, polymer composites and electrode material (Sohail, 2017).

Graphene oxide (GO) is a 2D carbon material derived from graphene by introducing covalent C-O bond. The bulk form of GO, typically named graphite oxide, has attracted repetitive attention since it was first prepared by Brodie in 1859 (Jirickova *et al*., 2022). With respect to the great capacity of the method and purity of the final product, reduction of graphene oxide is better than other techniques. Nevertheless, industrialization with low expense is one of the major difficult task in graphene field. In view of these facts, the mechanical exfoliation and reduction of graphene oxide are the two plausible methods for large production of grapheme from biomass. However, pyrolysis has been common techniques to synthesize graphene on metal surface from various carbon sources (Shams *et al., 2015).* The present research focused on the synthesis of graphene from hyphaene shell. The main target of utilizing these sources is to create greener trial and sustainable alternatives with low cost implications and larger production of graphene oxide. The synthesized graphene oxide was characterized by analytical techniques such as FTIR, TGA, XRD, SEM and BET.

Materials and method

Materials

*Hyphaenethebaica*shellswerepurchasedatlocalmarketcalled“TshohuwarKasuwa”inSokotostate. Thefruitwasidentifiedatthe Botany Unit, Department of Biological Sciences, Usmanu Danfodiyo University, Sokoto State, Nigeria. The fruit case was removed using a hack saw, the fruit was decorticated, washed and dried prior to grapheme productions. Chemicals were purchased from Sigma Aldrich, London (AR grade) and were used without further purification sodium hydroxide (NaOH), sulfuric acid (98% H_2SO_4), sodium nitrate ($NaNO_3$). Potassium per magnate ($KMnO_4$), hydrogen peroxide (30%H_2O_2), hydrochloric acid (5% HCl) and deionized water

Preparation of Graphene

The method described by Sahila and Prabhalitti (2020) was adopted which is thermal combustion under controlled condition at 550°C for 10 minutes. After goriba shell was collected, cleared and dried. Goriba shell was treated by thermal combustion at 550 ^{o}C, 10g of Goriba shell ash and 50ML of 2M sodium hydroxide (NaOH) was pour into a beaker and stir for 4hr at 70°C, using hot plate magnetic stirrer. After that, the solution was filtered using Whatman N0.41 ash less filter paper, the filtrate solution was discarded. The residue of carbonized material was mixed with 1g of D-tyrosine which dissolve in 50 cm^3 of trichloromethane, the mixture was treated with high speed centrifugation (1500 rpm, 15 min) after the procedure, graphene oxide was suspended in the solvent while other forms of carbon remain at the bottom.

Preparation of Graphene Oxide (GO)

Methods

Graphene oxide was prepared according to Hummer's method, 5g of graphene was added to concentrated H_2SO_4 ($100cm^3$) under stirring in an ice bath for 20 minutes, under vigorous agitation, 3g of $NaNO_3$ and 15g KMnO4 was added slowly to keep the temperature lower than 20 ^oC, then the mixture on a hot plate magnetic stirrer at 35 $^oC$for 30 minutes, then added $100cm^3$ of deionized water and raise the temperature to 90 ^oC. Again the mixture was diluted with $250cm^3$ warm deionized water and 30cm3 H2O2 till the brown solution turns to bright yellow. Finally, the obtained product (Graphene oxide) was dried at 60 ^oC for 12hr (Marcano *et al.*, 2010).

Results and Discussion

The Graphene Oxide (GO) synthesized from *H.thebaica* shell using modified hummers method was characterized by FTIR, XRD, TGA, BET and SEM.

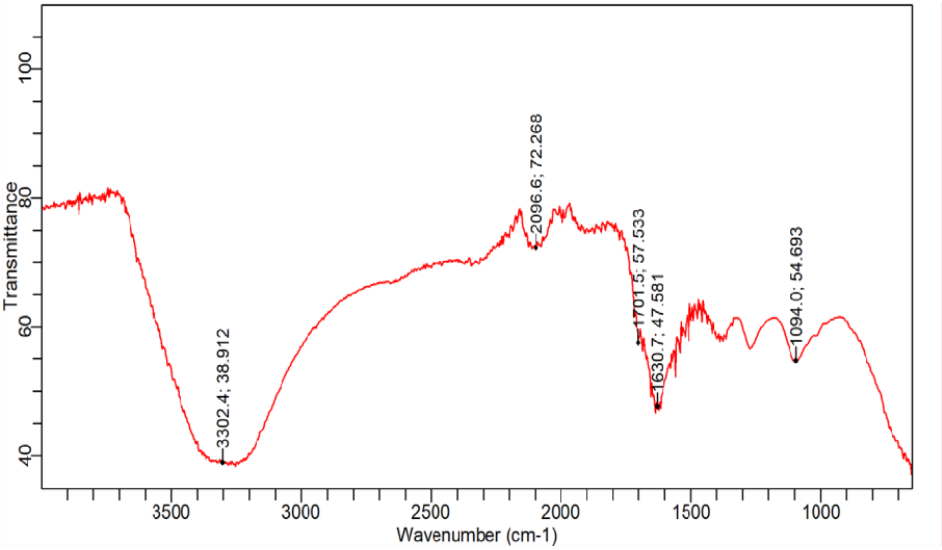

Figure 1: FT-IR of Graphene Oxide (GO)

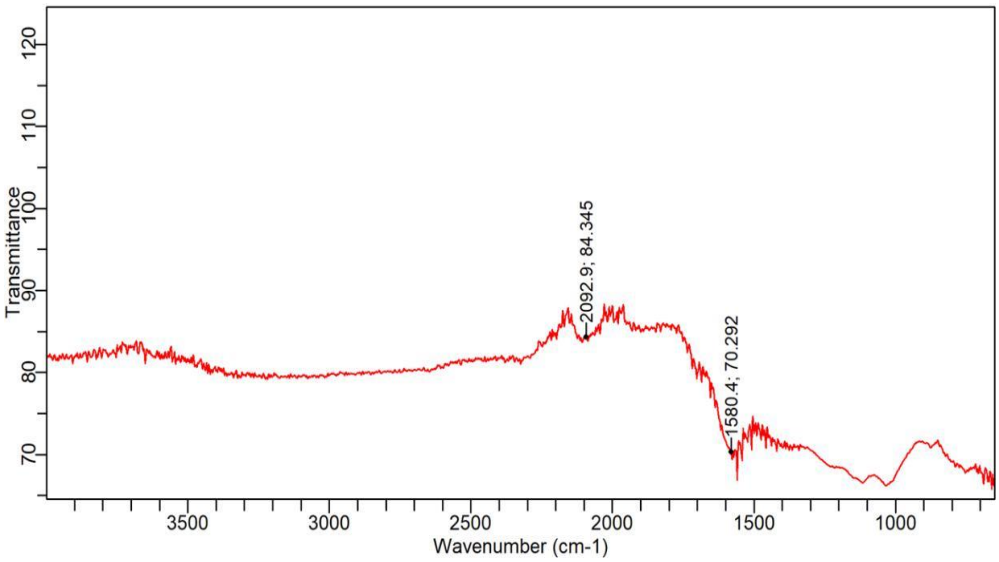

Figure 2: FT-IR of Graphene

FT-IR Results of Graphene

The F-IR spectrum of graphene presented in Table 4.1 showed intense peak at 1580 cm^{-1} and the peak around 1580.4 cm^{-1} was due to C=C double bond in graphene sheet. While the absorption around 1640.5 cm^{-1} was due to benzene ring and bending absorption around 3420 cm-1 due to water associated with potassium bromide (KBr) used for the preparation of Fourier Transform Infrared (FT-IR) sample. The result of this research correspondent with the findings of (Brarath*et al.*, 2016).

FT-IR Result of Graphene Oxide (GO)

The Fourier Transform Infrared (FT-IR) results presented in Figure (1) showed oxygen functionalities of grapheme oxide. The broad band around 3302 cm-1 attributed to C- OH stretching vibration of hydroxyl group. The peak of 1701 cm-1belongs to carbonyl group. The peak at 1630 was due to C=C double bond and the peak around 1525 cm-1 belong to benzene ring while the peak around 1094 cm-1 was due to C-O-C stretching in epoxy group. The result of the research was similar to the findings of the research conducted by Darius (2016).

XRD Analysis

X-ray diffraction pattern of the graphene oxide as shown in Figure (2) showed a strong defined peak at $2\theta = 19.6^{o}$ due to the intercalation of oxygen group functionalities. The oxygen space decreases the angle between transmitted beam and reflected beam. The broadening in the peak of XRD of graphene oxide arises due to small size of the crystal. If the graphene has a big crystalline structure, the peak in the XRD pattern will appear very sharp and intense.

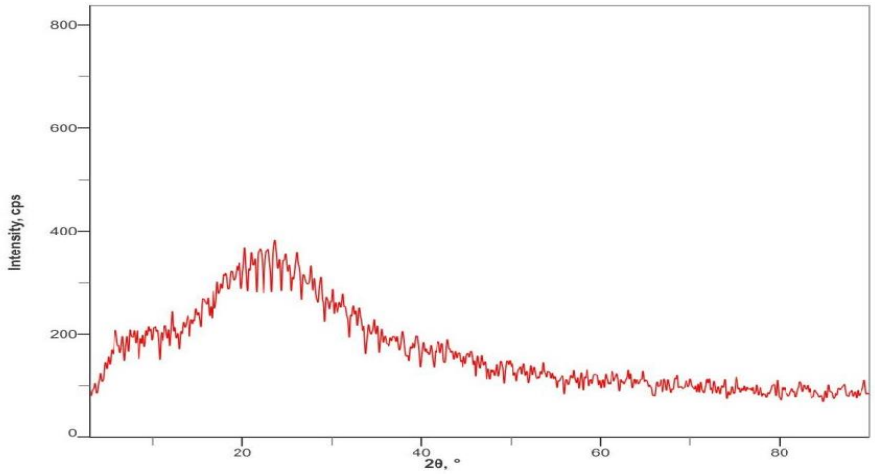

Figure 3: XRD Spectrum of Graphene oxide

Thermo-gravimetric Data Analysis (TGA) of Graphene Oxide

TGA analysis was conducted to test the thermal stability of GO as shown in Figure (3).
Three stages were observed in the mass loss curve of the GO sheet. Firstly, about 2 % massloss occurred at the temperature 100 ^{o}C as a result of H2O molecules in the GO layers, secondly the thermal decomposition of oxygen containing function groups indicate that about 16% degrade at a temperature of 350°C, and almost 94% mass loss occurred at 888°C due to the combustion of the carbon skeleton. The result of the analysis indicated that grapheme oxide has a good thermal stability. The result of graphene does not show any loss of mass at

low temperature because there are no oxygen functionalities in the layer of graphene. Graphene has higher T_{max}because it requires excessive amount of thermal energy to crackdown the sp^2 hybridized carbon atoms bonded by covalent bond in a hexagonal lattice framework. From these research, it is obvious that temperature can be considered as a key variable that can be used for identification, particularly for sensing the presence of oxygen functional group in graphene oxide. The outcome of the research is in accordance with literature (Farzaneh *et al*., 2021).

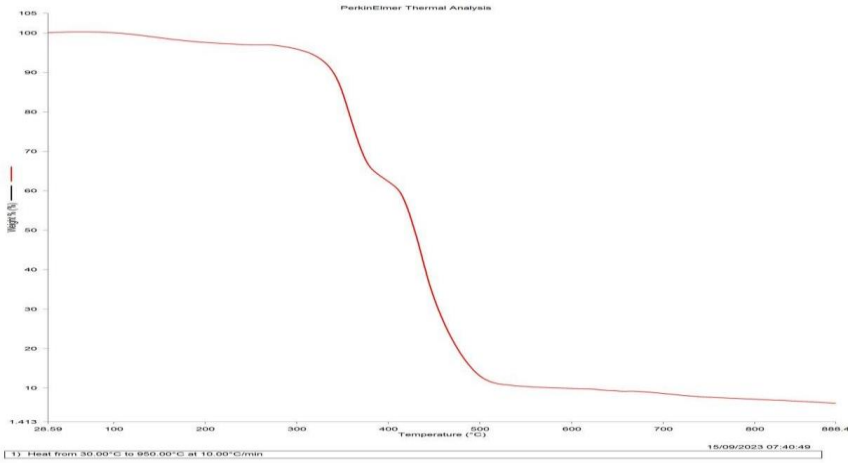

Figure 4: Thermo-gravimetric Data Analysis (TGA) of Graphene oxide

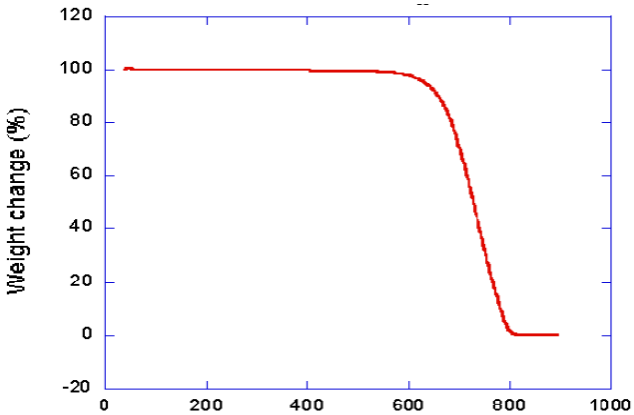

Figure 5: Thermo-gravimetric Curve (TGA) of Graphene

Brunauer Emmett Teller (BET) Result of Graphene Oxide Analysis

The measurement is typically based on nitrogen (N2) sorption at theory to measurement of gas adsorption. The result of BET of these research showed that the graphene oxide has a surface area$of356.276m^{2/}g$ as shown in Table 1.

Based on the result of BET analysis, graphene oxide is an excellent material for adsorption, since the degree of adsorption also depend upon the surface area of the solid. The greater the surface area, the larger the surface ~~is~~ available for adsorption. The surface area depends on the particle diameter of the material, the results of this research are also in agreement with literature (Zhihao *et al.*,2019).

Barette, Joyner, and Halenda (BJH) method was used to determine the pore volume and poresize of the graphene oxide in this research. Regarding the theories developed for pore size analysis, they are based on a reference adsorption isotherm. The results showed that the synthesized graphene oxide has an excellent pore size 2.138 nm which can be categorized under mesoporous range and pour volume of 2.15 m^3/g. The result is in line with research conducted by (Zhihao *etal.*, 2019).

Table1: Brunauer Emmett Teller (BET) result of grapheme oxide analysis

Slope	7.694
Intercept	2.080
Correlation coefficient	0.98929
C constant	4.698
Surface area	356.276m^2/g

Scanning Electron Microscope (SEM) of Graphene Oxide

The result of SEM analysis for graphene oxide shown in figure (3).The apparent enlargement of the image was at 1000 x magnification, the distance between the optical lens and the GO sample was 5mm.The result of analysis indicate that the whole surface morphology of the graphene oxide has rough surface and tiny pores. However, the BET results also proved that the grapheme oxide has larger surface area and mesospore size.

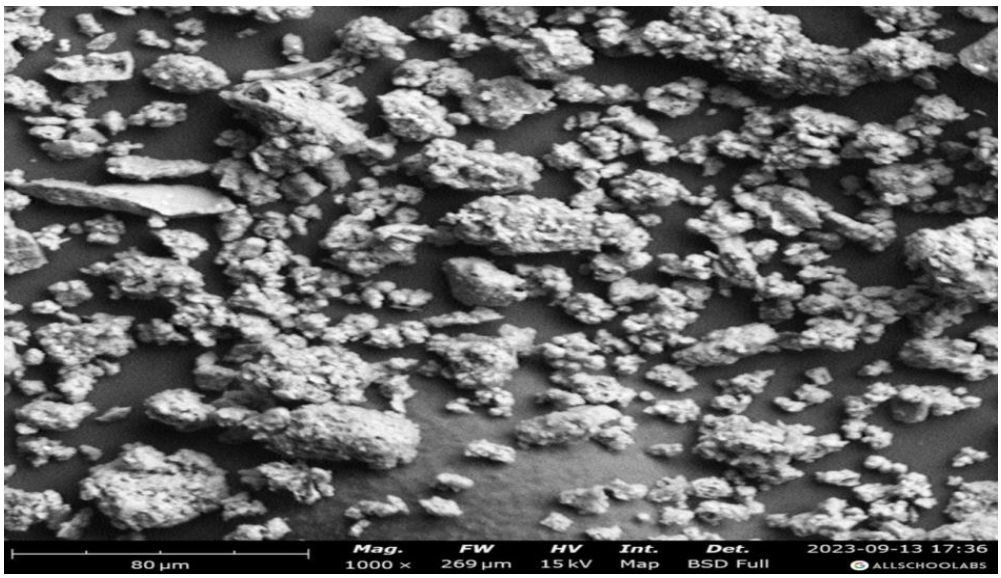

Figure 6: Graphene Oxide SEM Image

Conclusion

Basedontheresultsobtainedinthiswork,itispossibletosynthesizegraphemeoxide(GO)using*H.thebaica*shellasastartingmaterial.FTIRspectrumconfirmedthattheGOhadsome oxygen functional groups, XRD band confirmed the crystalline structure of grapheme oxide and TGA analysis proved that graphene oxide had an exceptional thermal stability and BET analysis also confirmed that the GO had an excellent large surface area and pore size. The morphological image in SEM shows that the GO had a rough surface for good adsorption

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