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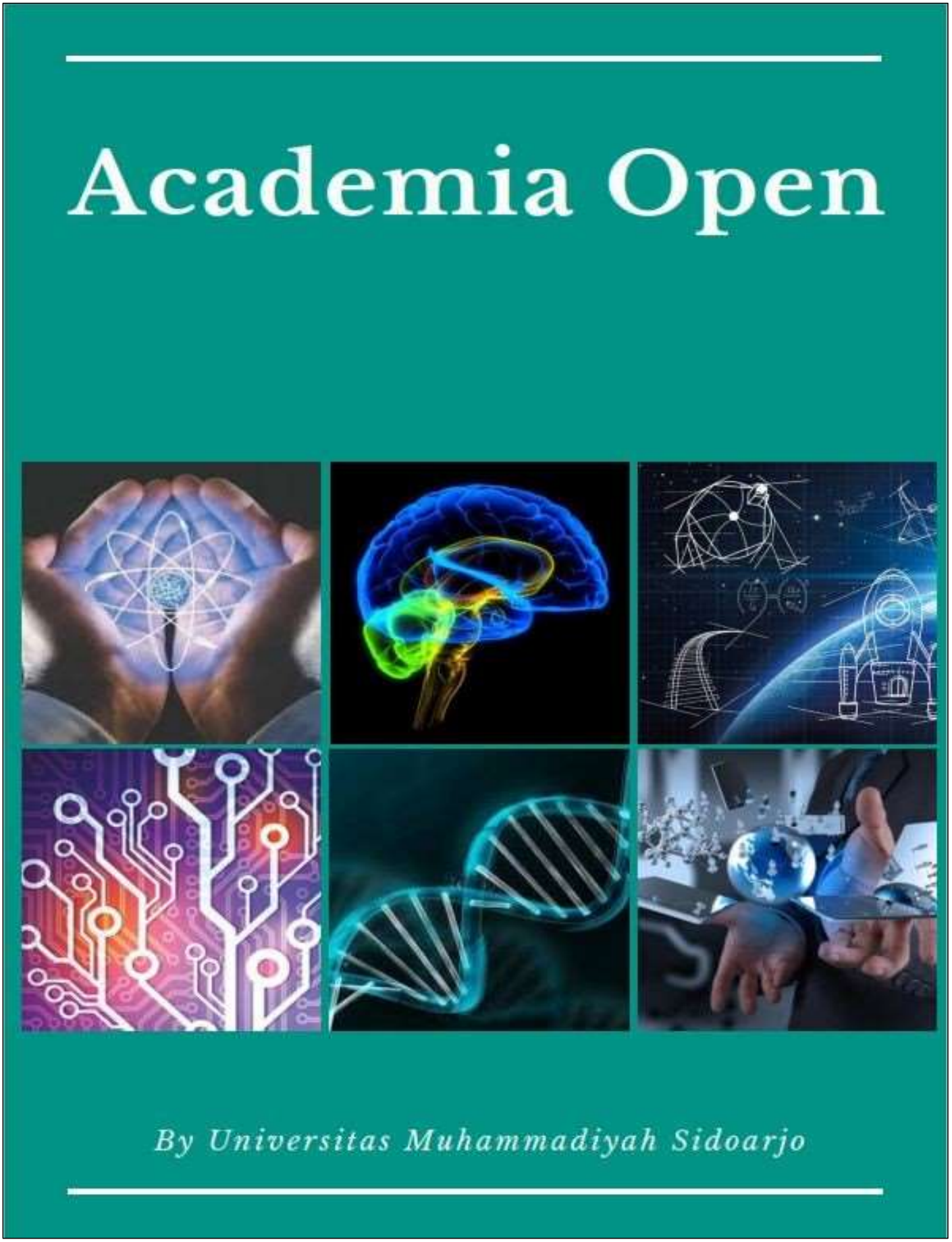

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Synthesis, Characterization, and Theoretical Analysis of Schiff Base Graphene Oxide Utilizing DFT Computing

Sintesis, Karakterisasi, dan Analisis Teoritis Grafena Oksida Basa Schiff Memanfaatkan Komputasi DFT

Ali Abra Naser, Ali.abra.naser@ec.edu.iq, (1)
Ministry of Education, General Directorate of Misan Education, Misan , Iraq

(1) Corresponding author

Abstract

The current study involved the synthesis of Schiff base graphene oxide (GOTM) and its characterization by various spectroscopy techniques, including EDX spectra, FESEM images, Raman shift, XRD diffraction, and FT-IR analysis. Furthermore, theoretical analysis (Gaussian application) was employed. Density functional theory was used to perform the calculations (DFT). The B3LYP method was used to integrate the basis sets "6-31G" (d, p) and "LanL2DZ." Total energy (E), total electrophilic index (ω), hardness (η), softness (S), energy gap (EH-L), highest occupied molecular orbital energy (EHOMO) and lowest unoccupied molecular orbital energy (ELUMO) are the determined quantum chemical characteristics associated with the efficiency decline. The azo group of -N=N-possesses a UV spectrum computed using DFT-TD in the region of 250-500 nm, according to the results of theoretical calculations using the density functional theory method (DFT).

Highlights:

1-Synthesis and characterization of Schiff base graphene oxide

2- Theoretical analysis of Schiff base graphene oxide by using DFT

Keywords:: Graphene Oxide; DFT; Schiff Base; GOT; GOTM

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Introduction

Because of its special attributes, graphene is known as the "material of the future," and is one of the most researched materials in the world [1, 2]. Recently, Graphene oxide (GO) has been employed as an appropriate matrix for a variety of metals, metal oxides, inorganic composites, and organic molecules because of its large surface area. [3]. Graphene oxide (GO) are considered a promising inert particle that can improve the properties of composite materials due to their large surface area, sufficient thermal stability, and stable mechanical properties [4]. To put it simply, graphene is a honeycomb structure made up of carbon atoms joined to three other carbon atoms via sp2 hybrid orbitals [5]. The carbon structure referred to as graphene oxide (GO) is layered, and oxygen-containing functional groups (=O, -OH, -O-, -COOH) are attached to the plane's edges and the layer's sides [6, 7]. The structure of GO can be single-layered or multilayered, identical to a typical 2D carbon material. A single-layered structure is called graphene oxide (GO), and a two-layered GO is made up of two layers of graphene oxide. The term "graphene oxide" refers to GO with two or more layers but less than 5 layers; "multilayered graphene oxide" corresponds to GO with 10 or more layers; and "graphene oxide" refers to a material with 11 or more layers [8]. Furthermore, the bandwidth of graphene oxide (GO) can be quantified, which is an important factor in determining the material's suitability for various applications [9, 10]. Schiff bases are nitrogen-containing aldehydes or ketone analogs in which the C=O group has been replaced with the C=N-R group. An aldehyde or ketone is usually added nucleophilically, then the primary amine is condensed to create it [11]. The study aims to describe the significance of DFT in Schiff-based graphene oxide diagnosis.

Methods

EXPERIMENTAL

Chemicals and Materials

Hydrogen peroxide, sodium nitrate, sulfuric acid, hydrochloric acid, potassium permanganate, ethanol, N,N-dimethylformamide (DMF), ethylene diamine, distilled water, p-vaniline, 4-Dimethylaminopyridine (DMAP), and N, N-Dicyclohexylcarbodiimide (DCC) were the chemicals that used in this investigation. These materials came from various companies.

“Fourier-transform infrared spectroscopy, (FT-IR) analysis”

An FTIR spectrometer (8400S-IR-FT SHIMADZU, Tokyo, Japan) was employed to conduct FTIR analysis with the KBr pellet protocol. Combined total of (100) scans of spectra were produced at a spatial resolution of (4 cm−1) between (400 and 4,000 cm−1).

“Field Emission Scanning Electron Microscopy, (FESEM)”

The samples were determined via the following parameters: an FEI NOVA nanosem450, Nano NOVA FE-SEM with a 20X–5000X magnification range, and an accelerating voltage of 15 kV.

“Raman spectroscopy”

Applying a portable Raman analyzer (Renishaw, UK) and an excitation light set to 532 nm, Raman results were obtained. The instrument's spectrometer can generate Raman spectra in the range of 0 to 5000 cm−1, with an average spectral resolution of 5 cm−1. Data on spectra were collected and analyzed using Origin program.

“X-ray diffraction, (XRD)”

A Philips-X, a Rigaku diffractometer, laser excitation, and Ni-filtered Cu K radiation (wavelength = 0.15406 nm) were used to create XRD patterns.

“Elemental analysis”

Bruker x Flash6i 10 energy dispersive spectroscopy (EDS) conducted between 0 and 20 kV.

Computational analysis

The density Functional Theory (DFT) serves as the underlying framework for understanding several chemical concepts used throughout various fields of chemistry using computational chemistry applications.

Synthesis of GO and GO ester functionalized (GOT)

In order to produce graphene oxide (GO), a modified version of Hummers' technique was employed. A 10 ml flask

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holding 50 ml of DMF was filled with 0.5 g of the generated GO following the addition of 1.52 g of p-vanillin to the reaction mixture and 30 minutes of ultra-sonication. The reaction mixture, 1.22 g of DMAP, and 2.06 g of DCC were subsequently mixed for 24 hours at room temperature. The DMAP is afterward neutralized by adding 5% HCl to the mixture. The final product (GOT) was obtained by filtering and repeatedly washing it in distilled water, resulting in an entirely black solid colour product. It was subsequently dried at 25°C for 24 hours. Figure 1 depicts the GOT compound synthesis [12, 13].

"Synthesis of Schiff base GO, (GOTM)"

After adding 30 ml of ethanol and the produced GOT (0.5 g) to a 100 ml round-bottomed flask, the liquid was sonicated for one hour to disperse it. 0.7 ml of ethylenediamine, or about 10 mmol, was then added. For four hours, the reaction mixture was agitated and refluxed. After filtering, the black-colored solid (GOTM) was formed. The material was then repeatedly washed with ethanol and water before being dried. Figure 2 depicts the GOTM compound synthesis [14].

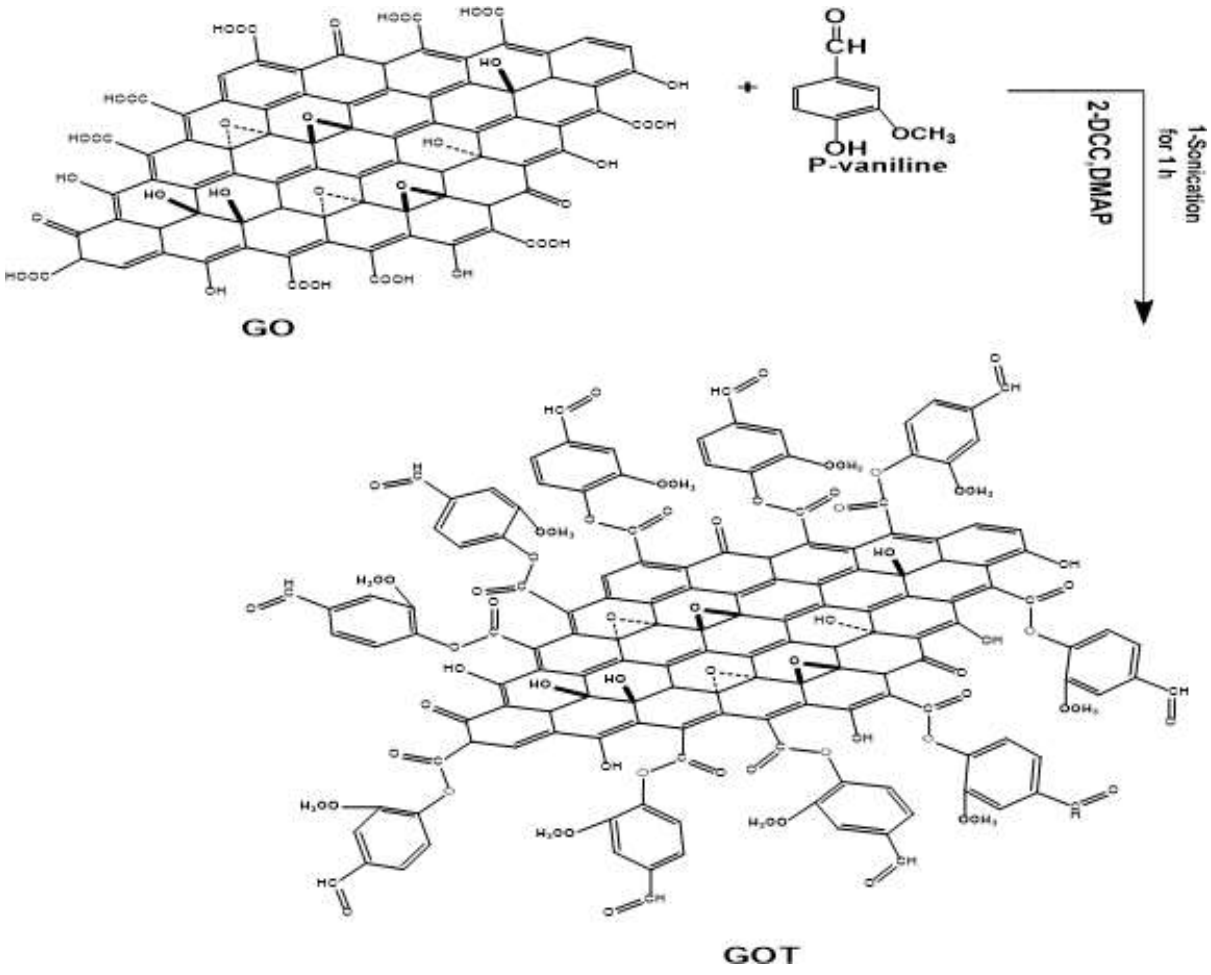

Figure 1. *Proposal of GOT.*

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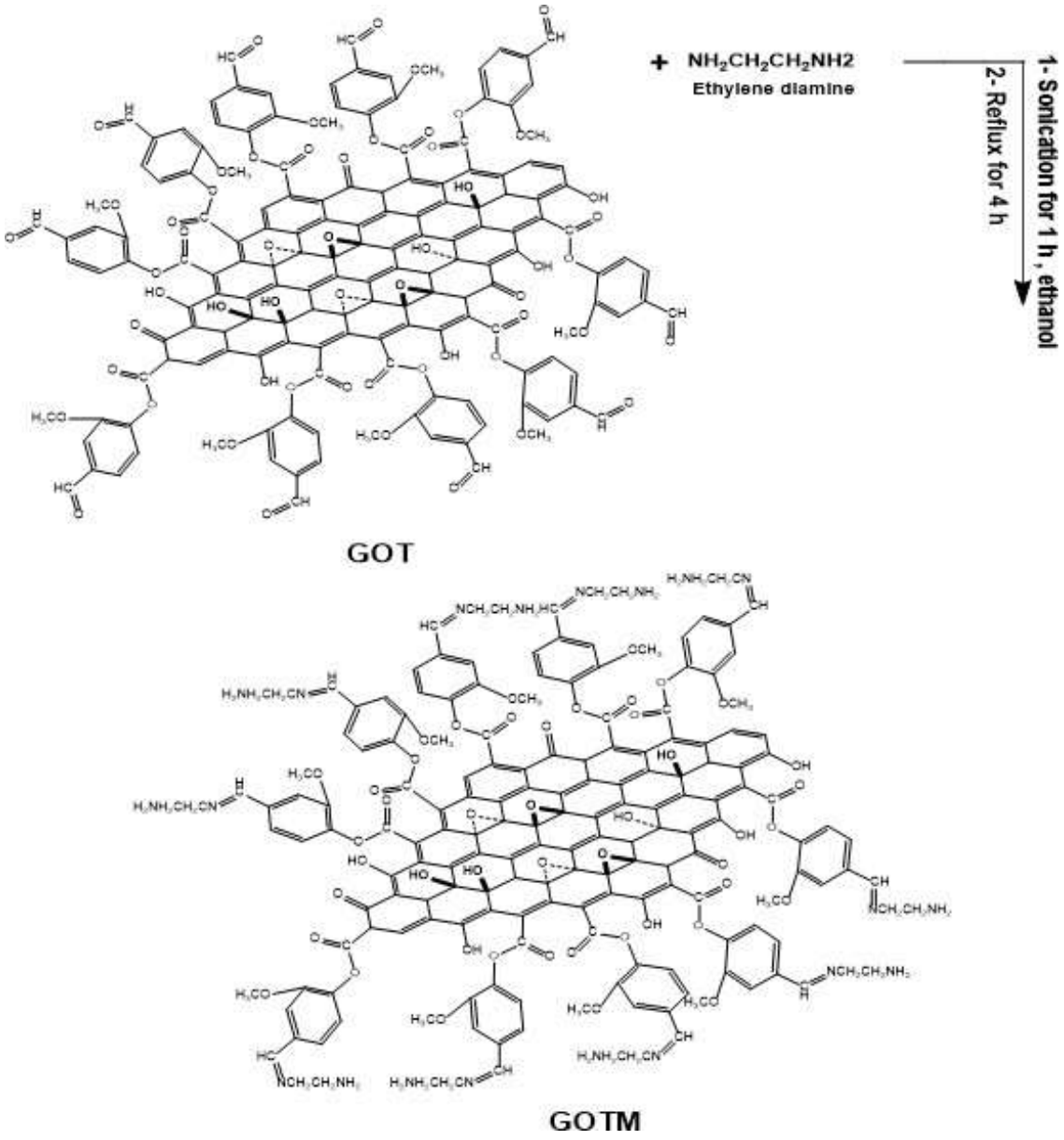

Figure 2. *Proposal of GOTM.*

Result and Discussion

Field emission scanning of electron microscopy, (FESEM)

To investigate the surface morphology of GO and GOTM, FESEM images were utilized. Due to GO's sp3-hybridized carbon backbone, the picture showed a characteristic crumpled, wrinkled, and sheet-like structure. The crumpling in the GO sheets enhanced after functionalization to form Schiff base on its surface. As seen in the FESEM image in Figure 3, the sheets were stacked on top of one another with extra protrusions, which may be related to the complex's attachment to the GO surface. The surface morphology difference indicated the source of the chemical modifications on the GO surface [7, 15].

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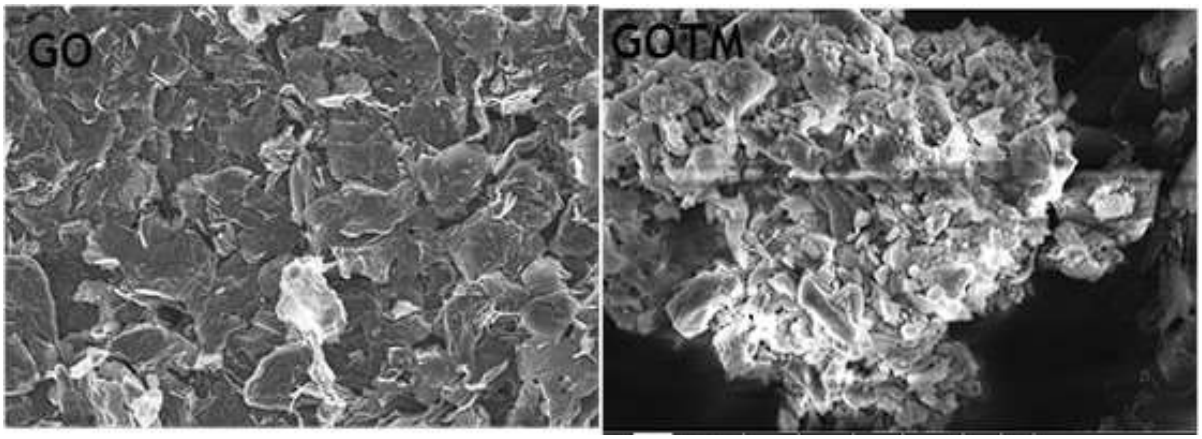

Figure 3. *FESEM images of GO and GOTM.*

EDX spectra

The elemental analysis of the final synthesized product is assessed using the EDX technique (Figure 4). It appears that the GO surface contains essential associated elements (C, O, and N) indicating that the EDX method is successful in creating the GOTM product. Interestingly, four novel, extremely weak peaks (Na, Al, Si, and Cl) that are present as impurities may be found on the prepared GOTM surface. The elemental analysis of this novel GOTM compound shows that 69%, 23%, and 6% of its elements are carbon, oxygen, and nitrogen, respectively (Table 1)[16].

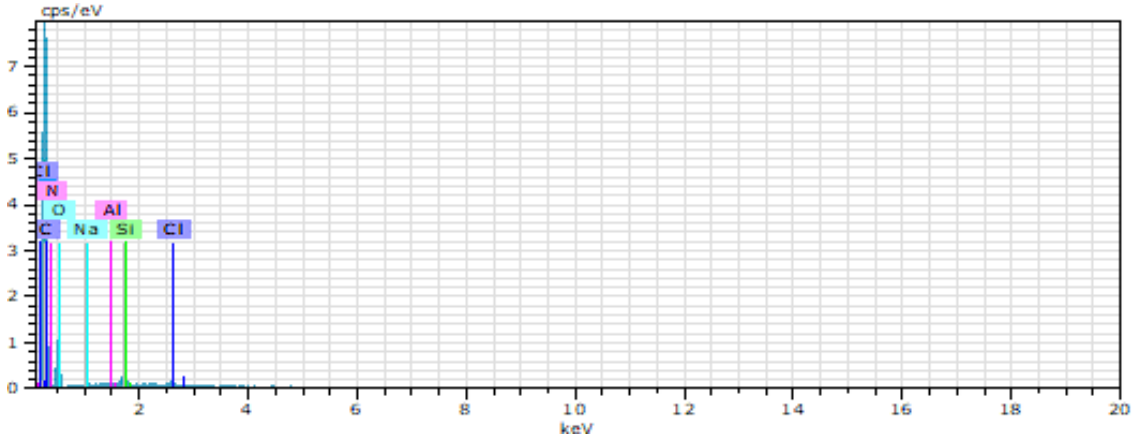

Figure 4. *EDX spectra of GOTM.*

Table 1: Elemental analysis of GOTM.

El	AN	Series	Unn.[wt.%]	Cnorm.[wt.%]	C Atom [at.%]	C Error (1 Sigma) [wt.%]
C	6	K-series	69.15	69.15	74.64	9.96
O	8	K-series	23.72	23.72	19.22	5.25
N	7	K-series	6.14	6.14	5.69	3.03
Si	14	K-series	0.37	0.37	0.17	0.06
Cl	17	K-series	0.36	0.36	0.13	0.06
Na	11	K-series	0.23	0.23	0.13	0.06
Al	13	K-series	0.02	0.02	0.01	0.03

Figure 5.

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FT-IR Spectra

The FT-IR spectra of GOTM and GO molecules are analyzed and compared in Figure 7. In the GO and GOTM's FT-IR spectra, the stretching of O-H on their surface is shown by the substantial band at 3352 cm-1[17]. In the exact same band, Amides A and B indicated that N-H overlapped with the OH group [18, 19]. The C-H group is clearly visible in the GOTM and GO spectra at the 2900 and 2829 cm-1 areas, respectively. On the other hand, the pronounced signal at 1728 cm-1 is caused by the carbonyl groups in GO and GOTM. The C=C and C=N groups, known as chromophores, are with the carbonyl group in the same band[20]. The GO and GOTM structures incorporate amides II and III[21]. These amides can be observed in the 1408–1300 cm-1 range. Additionally, C-O and C-C stretching are caused by the band in the 1051 cm-1 region. The following functional groups can be inferred to make up the skeleton of GO and GOTM: O-H, N-H, C-H, amide I, C=N, C=C, amides II, III, C-O, and C-C.

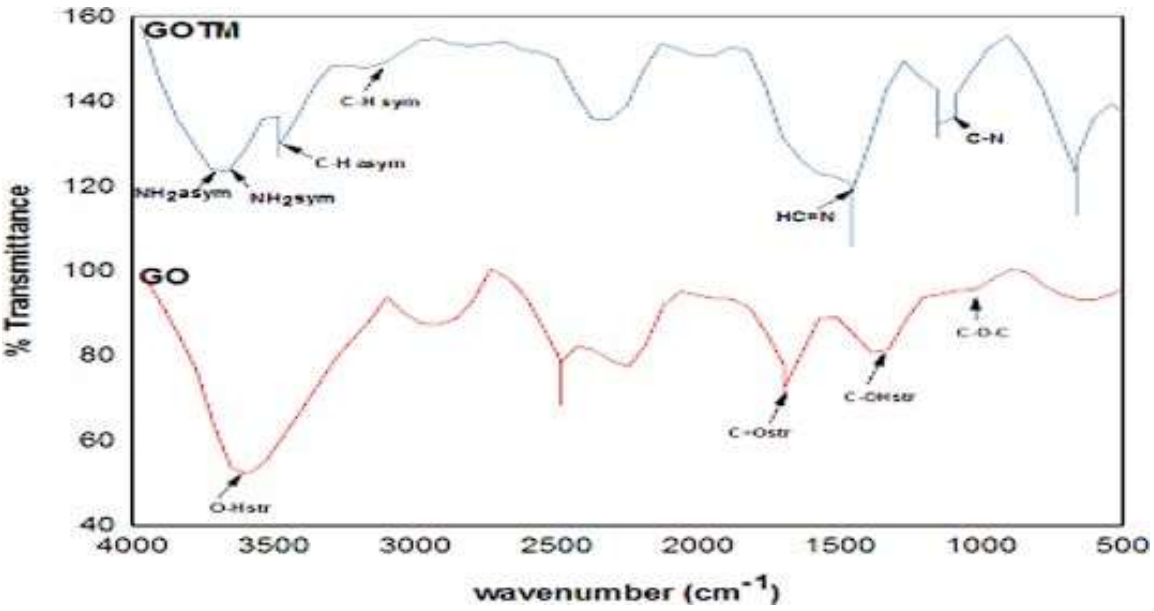

Figure 6. *FTIR spectra for GO and GOTM.*

Raman spectroscopy

One method that is frequently utilized to identify compounds like graphene oxide is Raman spectroscopy[22, 23]. According to our findings, the Raman spectra of these compounds displayed bands with respective centers at 1602 and 1359 cm−1. It is evident that the structure of these substances (GO and GOTM) incorporates more sp2 hybridized atoms. Regarding the abundance of sp2 carbon and the lack of sp3 carbon, the results of Raman scattering are substantially supported by the infrared spectroscopy results for (GO and GOTM) compounds. In Figure 5, Two bands, D and G, were detected in the graphene oxide (GO) Raman spectra at 1359 cm-1 and 1602 cm-1, respectively. The G band displays E2g type sp2 carbons and the extent of graphitization, whereas the D band displays the carbon skeleton's flaws. The intensity ratio of the D band to the G band (ID/IG) can be used to determine the extent of the sp2 domain's defects, creases, edges, and average size. The two bands of the GOTM compound are D = 1365 cm-1 and G = 1598 cm-1. On the other hand, GO and GOTM were to have ID/IG values of 0.90 and 0.99, respectively. The increase in intensity ratio (ID/IG) indicates that the sp2-hybridized carbon skeleton in GO experienced substantial alterations following the attachment to its surface [24, 25].

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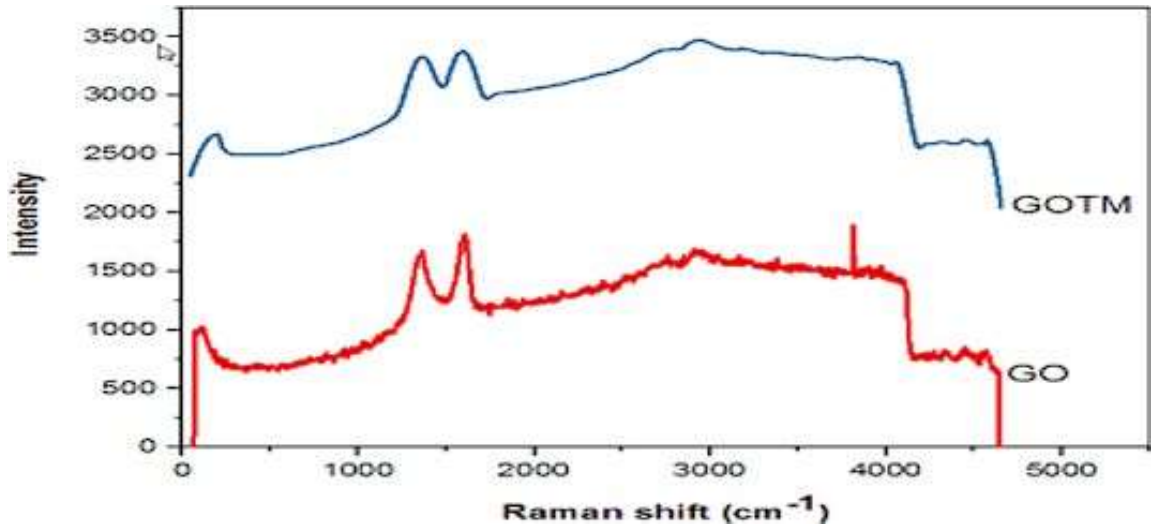

Figure 7. *Raman shift spectra for GO and GOTM.*

XRD Diffraction

The XRD patterns of GO and GOTM are seen in Figure 6. With a spacing of 8, GO displayed a distinct peak at 11.15°. When GO and GOTM are compared, different peak values and interlayer spacing emerge. The GOTM peak (12.9°) was found to be higher than the GO peak, with a slight decrease in interlayer spacing (6.9). This implies that the surface of GO was effectively bound with the Schiff base product of graphene oxide during the change [12, 26, 27].

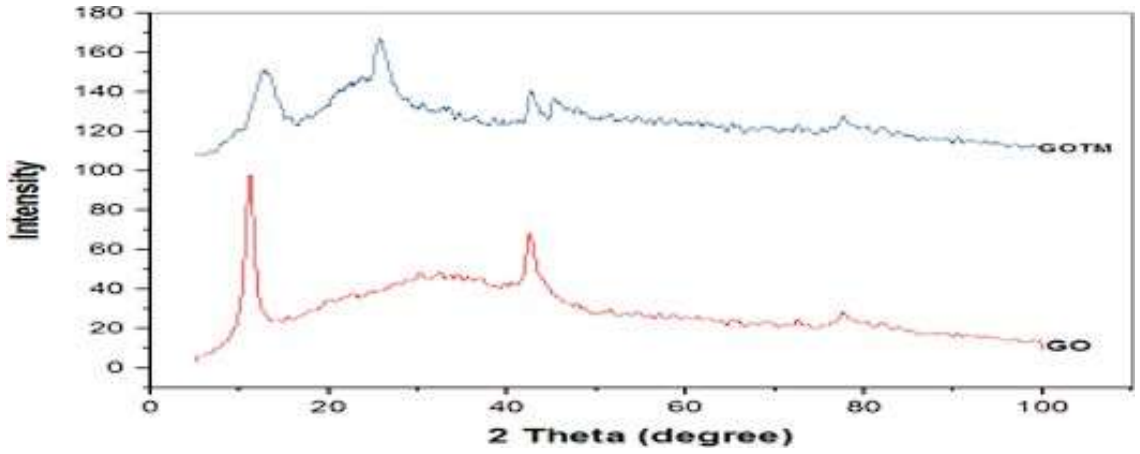

Figure 8. *XRD spectra for GO and GOTM.*

Computational analysis

Through applications in computational chemistry, density functional theory (DFT) provides the foundation for understanding various chemical concepts used in many branches of chemistry. Gaussian-09 software, which includes considerable geometric optimizations, was used to perform quantum chemical calculations to assess the compatibility between theory and experiment. In addition, various factors, such as total energy "E", electronic chemical potential "μ", overall hardness "η", molecular orbital energy band gap (EHL), and overall softness "S", characterize reactivity and stability. The values of the investigated overall electrophilicity indices, "ω", "HOMO", and "LUMO", are presented in the figures. Table 2 presents the electrical properties of GO and GOTM [28, 29].

Properties	ETotal	EHOMO	ELUMO	EH- L	μ	η	S	ω
eV								
GO	62040.43619	4.55628	2.95706	1.59921	3.75667	0.79961	1.250	8.824

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GOTM	17464.546	8.24505	3.1837	3.1837	5.6225	2.5806	0.375	1.878

Table 1. *The electronic characteristics of GO and GOTM.*

Conclusion

FT-IR, XRD, Raman shift, EDX, and FESEM were among the various techniques employed to characterize the GO and GOTM compounds. Theoretically, the GO and GOMT's stability and reactivity were examined at the (DFT) calculation level. Global parameters such as ionization energy ("I"), molecular hardness ("η"), electron affinity ("ω"), frontier molecular orbital topologies, and energy gaps ("EH-L") were used to determine the differences in reactivity between the three compounds and local ionization energy. Applying reactivity indices from DFT calculations has been shown to enhance our comprehension of chemical reactivity.

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