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Preparation and Diagnosis of a Nano-Pharmaceutical Compound Derived From Graphene Oxide

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Annotation: In this research, a pharmaceutical nanocomposite derived from graphene oxide was prepared and characterized using the halbridiol drug. The first step involved synthesizing graphene oxide using the modified Hummers method. Next, the composite was created by reacting the graphene oxide with the halbridiol drug in deionized water as the solvent, utilizing sublimation. The validity of the prepared compound was confirmed through characterization using various spectroscopic methods, including FT-IR, FESEM, and XRD.

Keywords: graphene oxide, graphite, nanocomposites, halbridiol.

Introduction

Graphene oxide is a hydrophilic, non-conductive carbon-based material characterized by a graphene network structure that contains various functional groups, including alcohols, ketone carbonyls, and carboxyl groups [(1)], as illustrated in Figure 1. It is particularly interesting for the creation of adsorbents because it allows for excellent functionalization, leading to chemically modified graphene in the form of a yellow solid. The carbon-to-oxygen (C:O) ratio in graphene oxide ranges from 2:1 to 2:9. Although it retains the layered structure of graphite, the interlayer spacing is larger and more irregular.

The bulk material can spontaneously disperse in basic solutions or can be dispersed through sonication in polar solvents, resulting in monomolecular sheets referred to as graphene oxide. This term draws a comparison to graphene, which is a single-layer form of graphite. The structure and properties of graphene oxide depend on the specific synthesis method and the degree of oxidation [(2)]. Typically, it maintains the layered structure of native graphite; however, the layers are twisted, and the interlayer spacing is approximately twice as large (~0.7 nm) as that of graphite [(3)].

The modified Hummers method, developed by Hummers and Offerman, is a safer, faster, and more efficient synthesis technique. It utilizes a mixture of sulfuric acid (H2SO4), sodium nitrate (NaNO3), and potassium permanganate (KMnO4), and this method remains widely used, often with some adjustments. Larger graphene oxide monolayers with a highly rigid carbon structure and lower concentrations of residual impurities can be synthesized in inert containers using highly pure reactants and solvents [(4)].

Graphene oxide (GO) is an intriguing material with numerous applications[(5)], including electronic devices, supercapacitors, water desalination membranes[(6)], composite materials[(8-7)], electrocatalysts[(9)], catalyst carriers[(10)], gas sensors[(11)], and a variety of medical treatments [(13-12)] .

Haloperidol, a commonly used first-generation antipsychotic, works by inhibiting dopamine D2 receptors in the brain to exert its antipsychotic effects. It is primarily used to treat the positive symptoms of schizophrenia, such as hallucinations and delusions. Haloperidol has received FDA approval for various clinical applications, and it also has several off-label uses.[(14)]

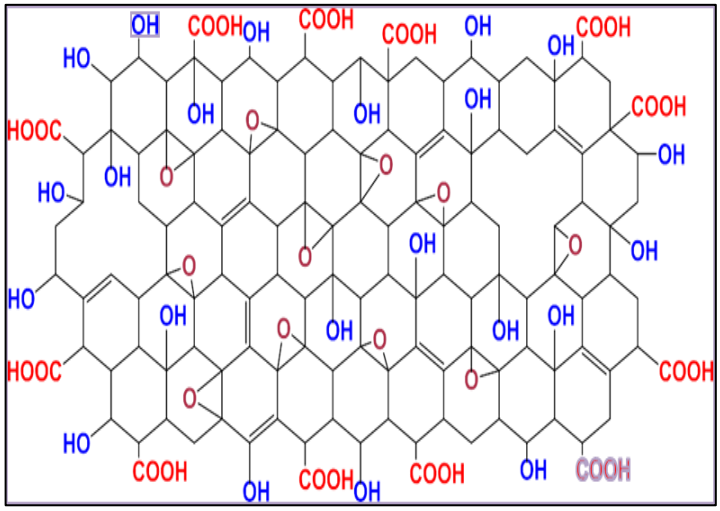

Figure 1. Chemical structure of graphene oxide

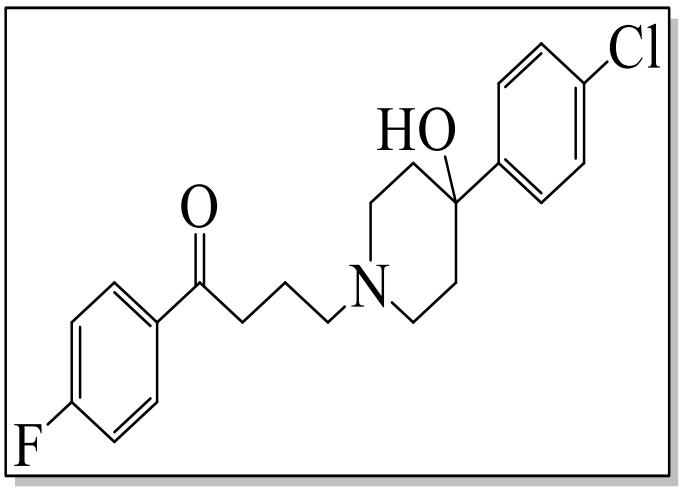

Figure 2. Structural formula of haloperidol

Experimental part

Preparation of Graphene oxide by Hummer method [GO]

To begin the procedure, place 46 ml of concentrated sulfuric acid in a suitable beaker and immerse it in an ice bath until the temperature is uniform. Gradually add 1.5 g of sodium nitrate over 15 minutes while stirring at 0°C. Next, slowly introduce 1 g of graphite dust over 10 minutes. Carefully add 6 g of potassium permanganate to the mixture over 15 minutes, ensuring

that the temperature remains below 20°C.

After this, keep the mixture in the ice bath for 5 minutes. Then, please remove it from the ice bath and allow it to stir magnetically for 2 hours. Slowly add 46 ml of distilled water to the mixture over 20 minutes, followed by raising the temperature to 98°C for 20 minutes.

Next, introduce 140 ml of warm distilled water and stir for an additional 10 minutes at room temperature. Add 15 ml of 30% hydrogen peroxide and continue stirring for 30 minutes. Then, add 300 ml of distilled water and let the mixture sit for 24 hours.

After this period, collect the precipitate by centrifugation at 6000 rpm. Wash the precipitate with a 10% hydrochloric acid solution once, followed by five washes with deionized water (45 ml each) until the pH reaches 7. Finally, dry the material at a temperature of 60-70°C until a constant weight is achieved[15].

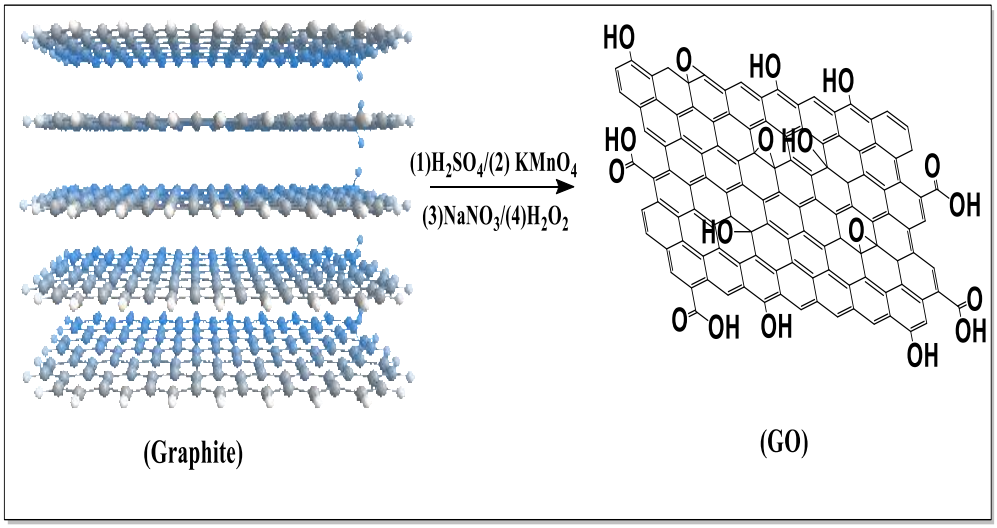

Figure 3. Steps for preparing graphene oxide using the Hummer method

Preparation of graphene-drug complex [GOHA]

The pharmaceutical halbridiol was mixed with 0.1 g of graphene oxide (GO) at a 1:1 ratio in 15 ml of deionized water. The mixture was sonicated for 30 minutes until it became homogeneous. Next, the solution was heated at 100°C for 2 hours. The resulting product was collected through centrifugation, washed with deionized water, and then dried at 50°C, as shown in Figure 4[16].

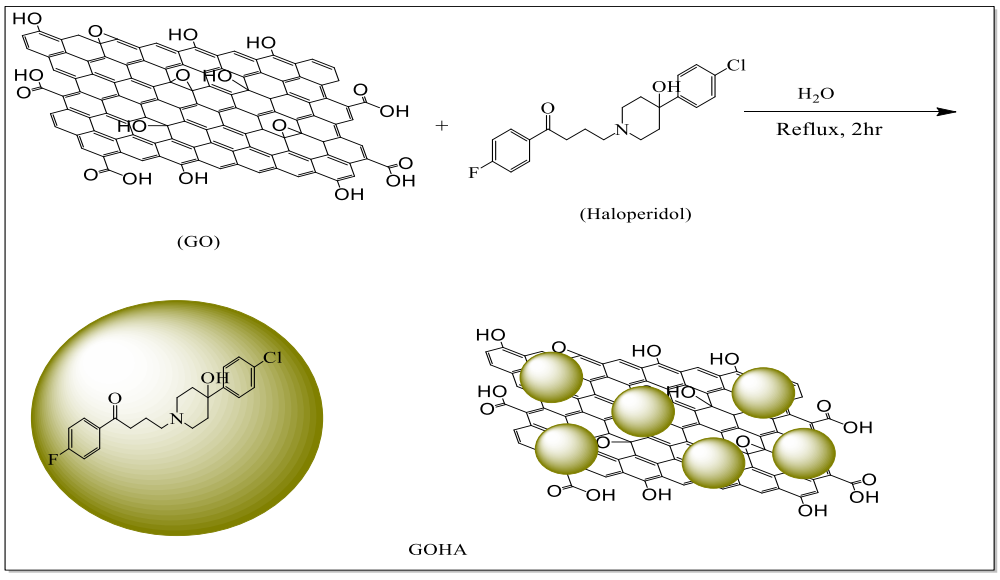

Figure 4. Steps for preparing the graphene drug complex GOHA

Results and Discussion

The infrared spectrum (FT-IR) of the GOHA compound in Figure (5) showed a distinct band due to the stretching of the phenolic bond present in the structure of the pharmaceutical drug halbridiol as well as present on the graphene oxide sheet (O-H) at (3334-3599) cm-1, and a distinct and clear band due to a group due to the stretching of the aliphatic (C-H) bond in its symmetrical and asymmetrical types at (2850-2920) cm-1, and the appearance of a band at (1732) cm-1 due to the stretching of the carboxylic (C=H) bond present in the graphene oxide sheet, and the appearance of a band at (1672) cm-1 due to the stretching of the ketone (C=O) bond present in the structure of the pharmaceutical drug halbridiol, and the appearance of a band at (1627-1408) cm-1 due to the stretching of the aromatic (C=C) bond present in the structure of the pharmaceutical drug halbridiol as well as located on a graphene oxide sheet

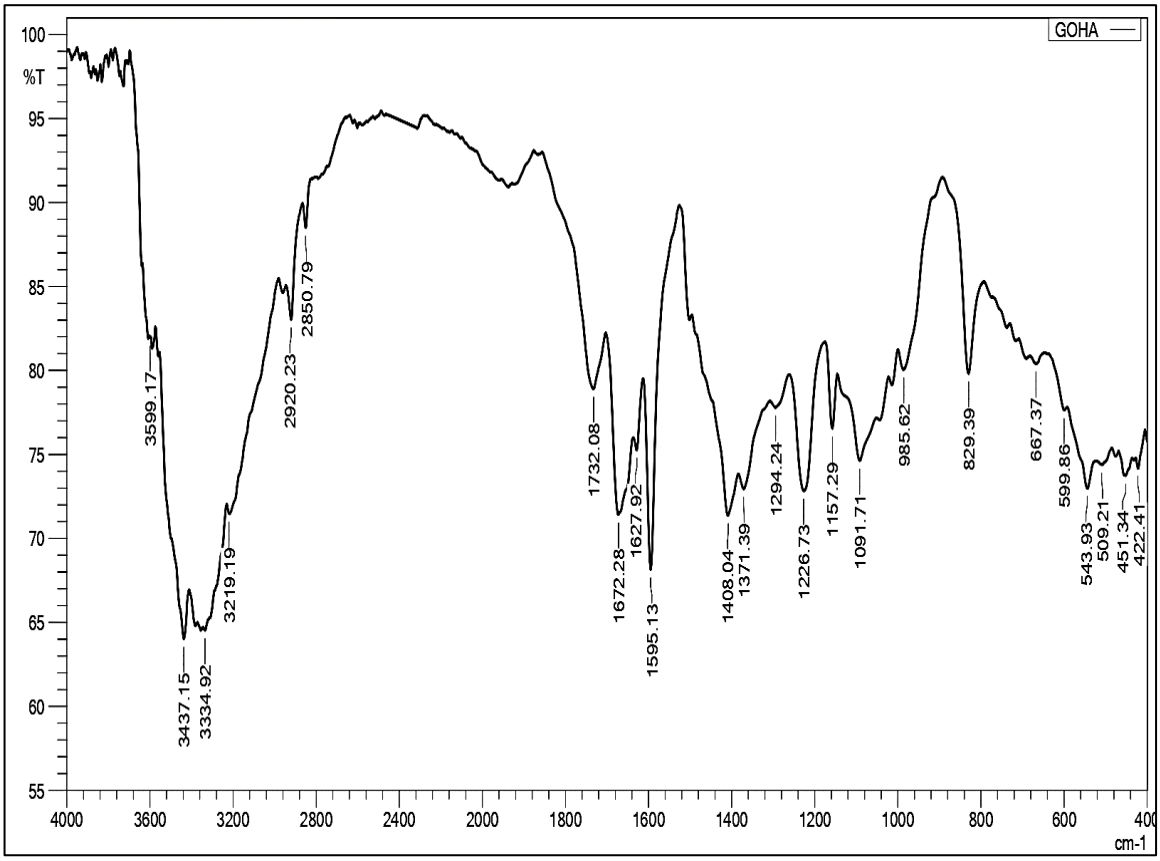

Figure 5. FTIR spectrum of GOHA compound

Results of XRD

The X-ray spectrum of GOHA revealed a 2θ angle value of 14.00068, corresponding to an interlayer distance of d = 6.32039 nm. Additionally, the grain size was measured at 17.39 nm, with the number of layers being n = 2.75. These values are consistent with those found in the literature (161), as shown in Figures 6 and 7 and Table 1. The thickness of the GOHA sheets (t) was calculated using the classical Debye-Scherrer equation, which is provided below:

$D = \lambda\, k/\beta cos\theta$

where k represents the Scherrer constant and depends on the crystal shape and size distribution (form factor). β is the full angular width at half the maximum peak intensity (FWHM), λ = 0.1541 nm is the wavelength of the Cu Kα radiation source, and θ is the Bragg angle (degrees). Diffraction was recorded in the range of 10-80 degrees. The thickness was found to be 8.59 nm. The number of stacked layers (n) can be estimated from the following relationship:

n = t/d

Table 1 X-ray measurement results for GOHA compound

Number of layers n	Granular size D	Space between layers d	2Θ	Comp.
2.75	17.39	6.32039	14.00068	GOHA

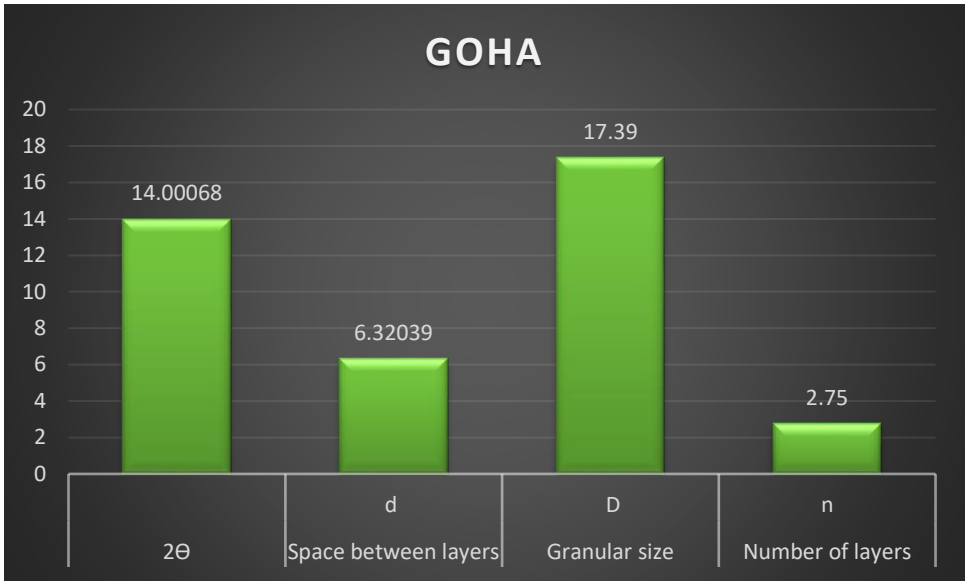

Figure 6. Results of X-ray measurements of the GOHA compound.

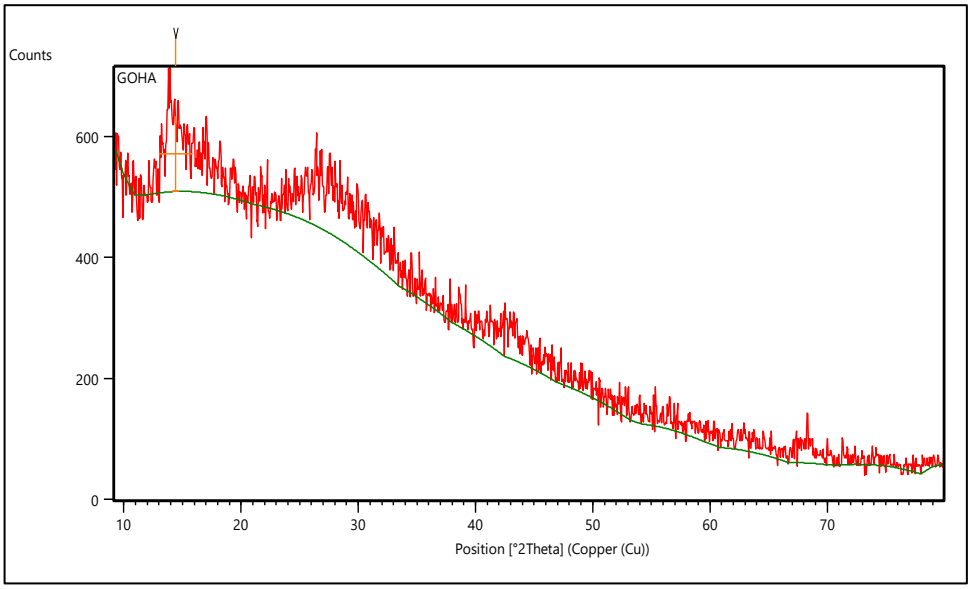

Figure 7. X-ray spectrum of the compound GOHA

Morphological images of the GOHA complex showed:

1. The irregularities observed on the surface of the plate are due to the varying ability of oxidizing agents to penetrate and oxidize different layers. This capability depends on the differences in interlayer bonding strength within the graphite.
2. The roughness on the surface of the plate is less pronounced than on the edges. This is because carboxylic groups are more effective at forming hydrogen and polar bonds with the drug compared to the surface of the plate.

3. The presence of potential drug clusters at the edges can be attributed to layered accumulation. This phenomenon results in increased peeling and a corresponding decrease in grain size, as illustrated in Figure 8.

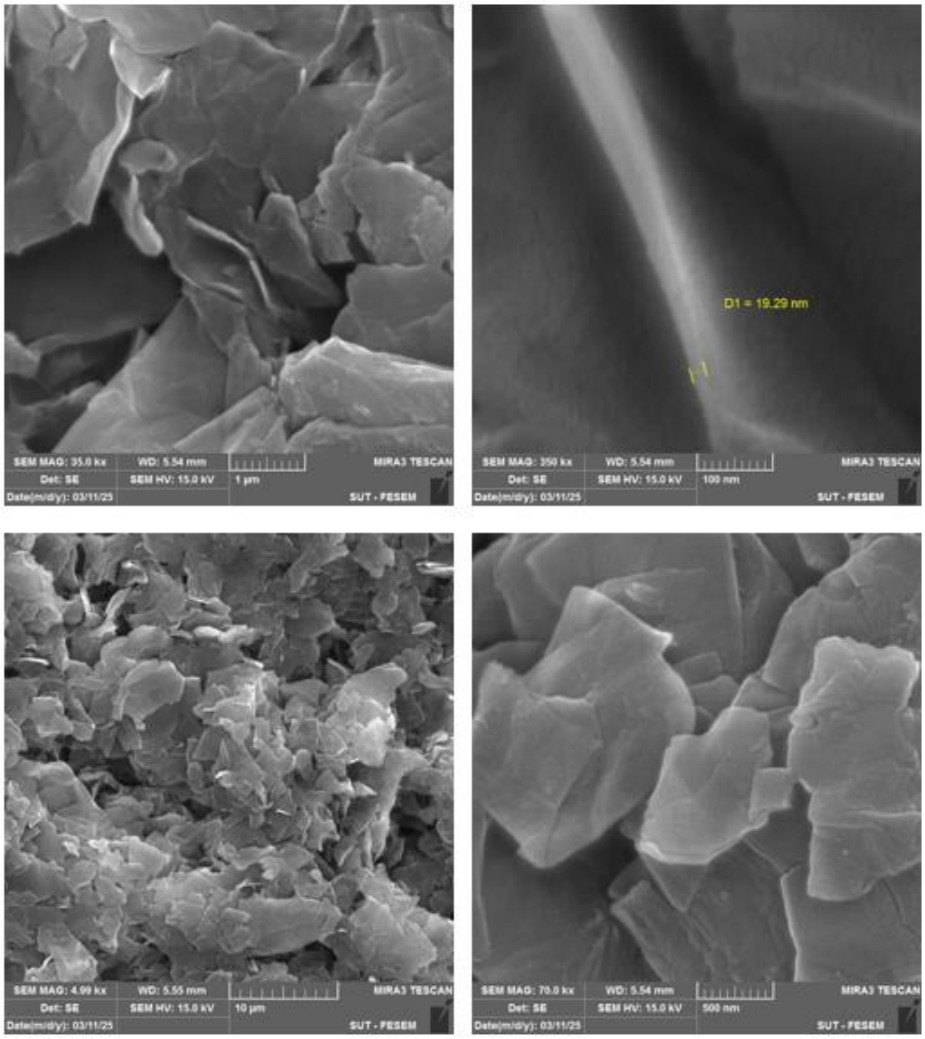

Figure 8. FESEM images of the GOHA composite.

Conclusion

Spectral examinations confirmed the validity of the prepared compound, as the compound showed a low particle size with a high peeling percentage, which confirms that the compound has various and wide medical applications.

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