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Preparing some nanocomposite decorated with pharmaceutical compounds and evaluating their biological effectiveness

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Abstract

The study involves the preparation of nanocomposites decorated with pharmaceutical compounds and their evaluation for biological effectiveness. These composites primarily utilize materials such as graphene and reduced graphene oxide (RGO). The research highlights the synthesis of these materials, their characterization through various techniques like X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Atomic Force Microscopy (AFM), and their application in antibacterial medical masks and bacterial biological activity evaluation. The results show that the nanocomposites have strong antibacterial properties against both gram-positive and gram-negative bacteria. At higher concentrations, they showed higher inhibition rates than regular antibiotics.

Keywords: Graphene, Reduced Graphene Oxide, nano composites.

1. Introduction

Nanomaterials are considered advanced chemical materials that can be produced with dimensions ranging from 1 nm to 100 nm. The small sizes and scales of these materials have led them to behave differently from traditional large-sized materials whose dimensions exceed 100 nm, and to have strong qualities and properties. Excellence cannot be found together in traditional materials, and the smaller the scale of nanomaterials, the greater the effectiveness. *(M. sherif EL- Eskandarany, 2009)*

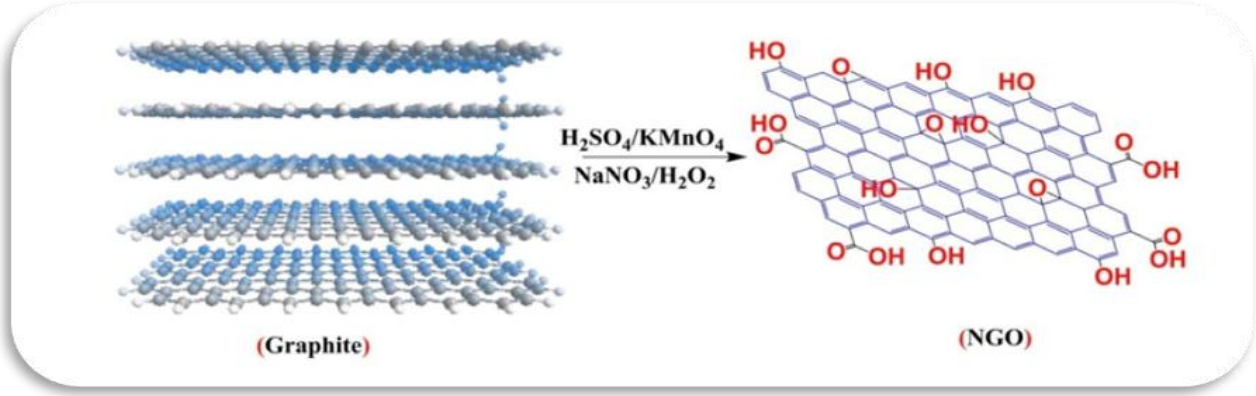
Examples of nanoscale objects are those that are the width of deoxyribonucleic acid or that are 10 hydrogen atoms in diameter *(Moniri, M., Boroumand Moghaddam,(A., Azizi et al 2017)* and nanotechnology is the application of scientific concepts and methods in the process of development at the nanoscale level, and industrial fields and sectors, such as the manufacture of foldable devices and nanorobots.

The emergence of new applications for nanotechnology has enabled researchers to control cell behavior and repair and develop human tissues, and it has also provided services in the field of energy and water purification. Nanotechnology has also been used to study the structure of cancer cells and treat them. Through nanotechnology, we can treat pollution and increase the efficiency of removing pollutants from soil and purifying seawater and groundwater .This technology also enables us to study and diagnose cells infected with diseases*(Basu, S., & Bhattacharyya, P. 2012).*

2. Material and Methods

2.1 Synthesis Graphene Oxide (GO)

The concentration of sulfuric acid (H2SO4) was introduced to a 600 ml beaker while magnetic stirring was maintained in a snow bath. Following fifteen minutes of stirring at room temperature, one gramme of graphite was added slowly over a period of ten minutes. After 10 minutes of careful stirring, add 6 grams of potassium permanganate; maintain a temperature below 10 degrees Celsius for another two hours. Removing the liquid from the ice bath after 15 minutes, add 46 milliliters of distilled water while stirring continuously. To bring it down to 50 degrees Celsius, a nice but still chilly temperature, after heating to 98 degrees Celsius, 140 milliliters of distilled water are added. Continue mixing for another 30 minutes after adding 15 milliliters of 30% hydrogen peroxide (H2O2) after 10 minutes. Dispense 150 milliliters of distilled water into each of the cups. At the end of the 24-hour period, drain the mixture and rinse it once with 10% (HCl) to remove any residual precipitate. Drying at 60–70 degrees Celsius followed five washes with anionic water to obtain the acidic function (pH=7). *(L. Shahriary and A. A. Athawale, 2014)*



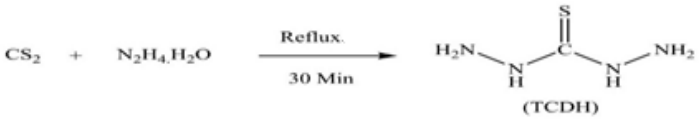
2.2 Synthesis reduced graphene oxide (RGO)

Blend 0.1 g of Hummer-prepared graphene oxide with 1 ml of hydrochloric acid in a 50 ml heat-resistant circular flask. Make sure to stir the mixture continuously until it becomes a clear solution free of plankton. One milliliter of hydrazine hydrate (80% concentration) should be added next. For two hours, use an inverter condenser to heat it at 100 °C. Sort the acquired NRGO. To get rid of any extra hydrazine, rinse the area three times with deionized water. Lastly, bake the mixture at 100°C for 12 hours to dry it. (*L. Tang, Z. Yang, F. Duan, and M. Chen, 2017)*

2.3 Synthesis Thiocarbodihydrazide TCDH

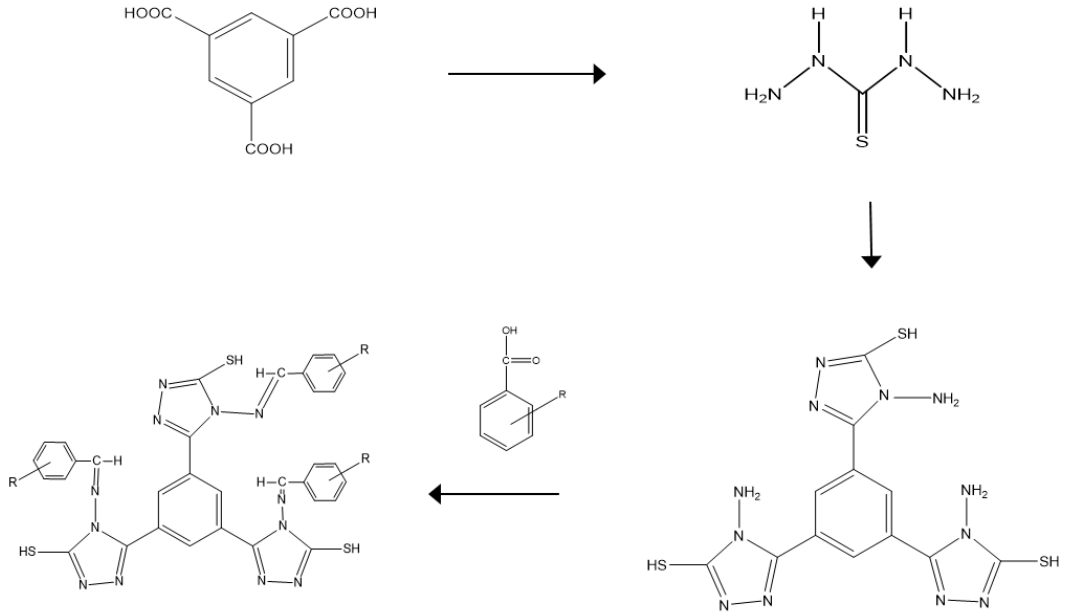
The following steps were taken in a 100 ml round flask that had been chilled in an ice bath: 20 ml of 80% hydrazine, 5 ml of carbon disulfide, and 10 minutes of magnetic stirring. The liquid was cooled to room temperature after 30 minutes, at which point a yellow precipitate had developed. Recrystallization with distilled water was followed by drying at 50 °C. Consistent with the literature, the melting point was determined to be

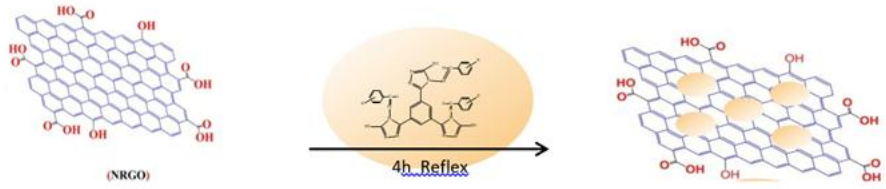
between 172 and 170 degrees Celsius. composed of 66 percent of the overall output. *(R. Nalawade, A. Nalawade, R. Shjendra, M. Anuse, 2017)*



2.4 Synthesis 5,5'-(5-(4-(((E)-benzylidene)amino)-5-mercapto-4H-1,2,4-triazol-3-yl)-1,3-phenylene)bis(4-(benzylideneamino)-4H-1,2,4-triazole-3-thiol)

The following reactants were combined in a heat-resistant flask: 0.01% pyridine-6,2-dicarboxylic acid and 0.02% TDCH, or 0.01% benzene-5,3,1-tricarboxylic acid and 0.03% TDCH. After that, the mixture was stirred constantly in an oil bath for ten to fifteen minutes until it reached melting point. The material was left to cool to room temperature until the melted state changed in terms of color and consistency. The next step was to apply a 10% sodium bicarbonate solution. To get rid of any residual acidity, the product was rinsed with ethanol many times after filtration. Afterwards, a thermal oven set at 60 Co was used to dry it. *(H. N. Moorthy, U. B. Vittal, C. Karthikeyan 2017)*





2.5 Synthesis of reduced graphene oxide nanocomposites

After 30 minutes of ultrasonic treatment at 50 Hz, 10 ml of distilled water is added to 0.3 mole of created nano-reduced graphene oxide (A2). This is followed by four hours of heating at 100 C°. The product undergoes three washes with 10 ml of deionized water following collection. After that, it's dried in an oven set at 80 degrees Celsius until its weight stays the same. *(Rashad, S. F., Al-Somaidaie, Gh. H. 2019) ,(Manousi, N., Rosenberg, E., Deliyanni, E. A., & Zachariadis, G. A. 2020)*

2.6 Biological applications of nanocomposites

2.6.1 Synthesis an antibacterial lab coat based on graphene pharmaceutical compounds

Methyldopa [A5,A6] colloidal solution synthesised at 50, 100, and 150 ppm concentrations An off-the-shelf lab coat was saturated with methyldopa solutions ranging from 50 ppm to 150 ppm. *(Rajabi, H., Jafari, S. M., Feizy, J., Ghorbani, M., & Mohajeri, S. A. 2020)*

2.6.2 Synthesis of colloidal solution using Methylcellulose

To create a transparent colloidal solution, heat 150 ml of deionized water to 90°C. Add 0.8042 g of methylcellulose (a 2% solution has a dynamic viscosity of 4000 cl), and stir it well to dissolve. Next, bring the mixture to a solid state by cooling it in an ice bath while stirring continuously. An equal volume of deionized water and 0.02 grams of the nanomaterial were combined in a beaker and let to sit for 60 minutes. The colloidal liquid was supplemented with methylcellulose once it had cooled. Mix was added to the reaction mixture after fifteen to twenty minutes of sitting at room temperature. When combined with three or five drops of a diluted hydrazine solution (1 ml in 100 ml of DI water), the combination can be stirred at room temperature for 30 minutes to produce a silver colloidal solution of methyl-dopa nanopharmaceutical compounds. *(Hiragond, C. B., Kshirsagar et al.,2018*)

2.6.3 Synthesis the colloidal solution using Xanthan gum

Stir vigorously to disperse 0.8042 g of Xanthan gum in the solvent, and then add 0.02 g of nanomaterial to a beaker with 150 ml of DI water that is at laboratory temperature. The material should have the dynamic viscosity of a 2% solution, which is 4000 cl. Once the 60 minutes are up, add the DI water colloidal substance to the 50 ml of deionized water in the beaker and agitate the mixture at room temperature for 30 minutes. Eventually, a silver colloidal solution of methyl-dopa pharmaceutical nanocomposites will form. *(El-Sheikh, M. A., Al-Enezy et, al 2020)*

2.6.4 Synthesis a medical lab coat containing the nanocolloidal solution

The methyldopa nanocompounds solution was used to generate three distinct concentrations: 100, 50, and 150 ppm. Lab coats, each about 5 by 5 cm in size, were immersed for five to seven hours in the solutions that had been previously made. After being left at room temperature for 30 minutes, the lab coat parts were dried in the open air. A lab coat treated with a 50 ppm solution of colloidal methyl-dopa nanopharmaceutical is referred to as A50, and it will be utilized in future antimicrobial investigations against both Gram-positive and Gram-negative bacteria. The A100 and A150 labels indicate that the lab coats were treated with solutions containing 100 and 150 parts per million, respectively. (Shirmohammadli, F., Nikzad et al .,2021)

2.7 Bacterial biological activity

2.7.1 Bacterial isolates

Two bacterial species, one Gram-positive and one Gram-negative, were utilized in this investigation. Their acquisition and diagnosis were completed at Samarra's General Company for Pharmaceutical Industry's Biological Activity Department; they were then activated just before usage. Bacillus, Salmonella, and Shikella are the gram-positive bacteria.

2.7.2 Synthesis of Bacterial inoculation

The bacterial suspension was prepared using a colony that was at least 24 hours old. After obtaining a smear of the bacteria using a lubricant, 10 ml of the prepared solution (typical saline) was supplemented with the particular strain of bacteria. A further day of incubation was subsequently added to the mixture. *(Aftan, M. M., Talloh, A. A ,et,al 2021) , (Aftan, M. M., Toma, M. A., Dalaf, A. H., Abdullah, E. Q., & Salih, H. K. 2021)*

2.7.3 Synthesis bacterial plates to test the biological activity of the prepared compounds

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To find out how effective the synthetic compounds were biologically, the cylinder plate method was employed, as detailed in the antibiotics-microbial testing part of the US Pharmacopeia (USP). A nutrition medium containing chemicals that bacteria can utilize for feeding is the essence of all this. *(Khalaf, S. D., Ahmed, N. A. A. S., & Dalaf, A. H. 2021)*

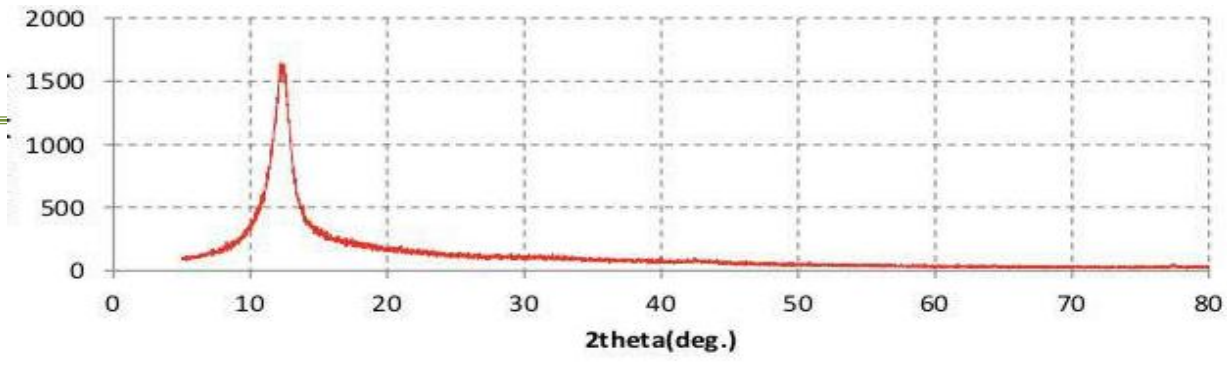
The following items have all been steriled and prepared using an autoclave:

The medium's pH level was determined to be (0.1 ± 6.6) following sterilization. It was then chilled to a temperature between 37 and 40 degrees Celsius. The following day, the bacterial suspension was introduced. This was achieved by meticulously choosing and inserting a swab of the target bacterium. After incubating for 24 hours in 10 ml of pre-made liquid medium, the suspension is transferred to the 100 ml basic nutrient medium. Twenty milliliters of medium is added to every Petri dish once the bacteria have been placed. Following the steps outlined in paragraph 4.6.2, 6 mm lab coat discs are manufactured when the medium has cooled. A standard antibiotic was utilized for comparison, and these discs were submerged in three different concentrations of the compounds (50, 100, and 150 ppm). The process is inhibited by Gentamicin sulfate at the same concentrations previously. Bacteria carrying Gram-positive and Gram-negative plasmids are both killed by it. The chemical was standardized after being cultured on particular media by the General Company for Pharmaceutical Manufacturing's Samarra quality control laboratories; it has many medical applications (Antibiotic Disc) The plates were placed in an incubator set at 37°C for 24 hours to determine the amount of antibacterial efficiency. The next step was to determine the inhibition diameter in millimeters using a piece of measuring equipment known as a zone reader.

3. Result and Discussion

3.1 Synthesis and characterization of graphene oxide nanosheets

X-ray diffraction of graphene oxide, (GO), showed an angle value of $2\theta = 11.82^{o}$ and a distance between the layers of d=0.74. Increasing the distance between the layers indicates oxidation, and then the grain size is calculated, which is equal to 5.43.



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Fig(1): X-ray diffraction of graphene oxide (GO)

The SEM morphological images of the compound (GO) show a peeling process resulting from oxidation and separation between the layers. Oxidation also appears on the edges and surfaces, with greater clarity of the rings on the plates.

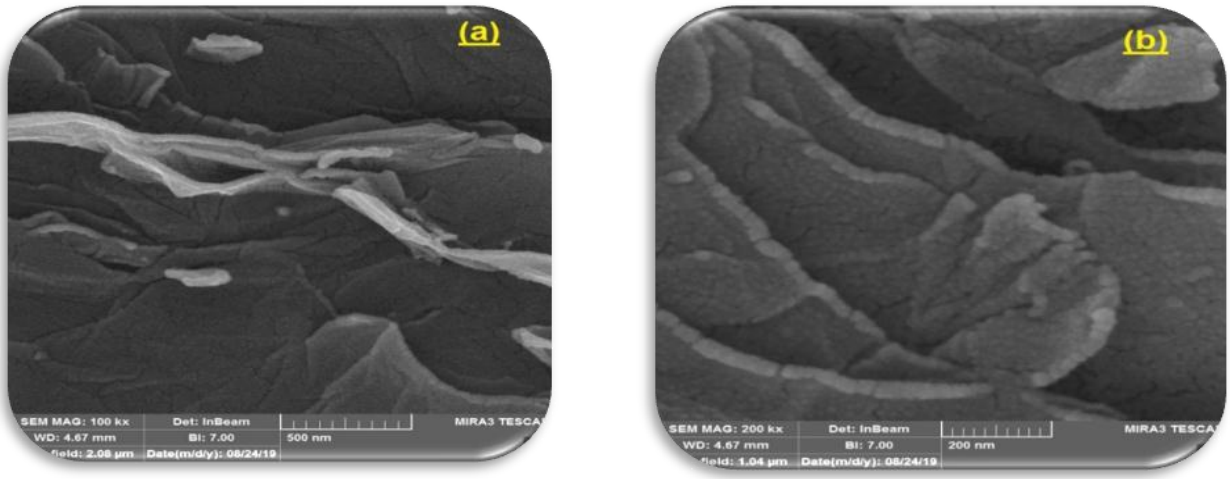


Fig (2) shows SEM images of the compound (GO)

AFM images of compound (GO) show a large sheet area (a) with the peeling process evident from observing the height of the sheet edges (b)

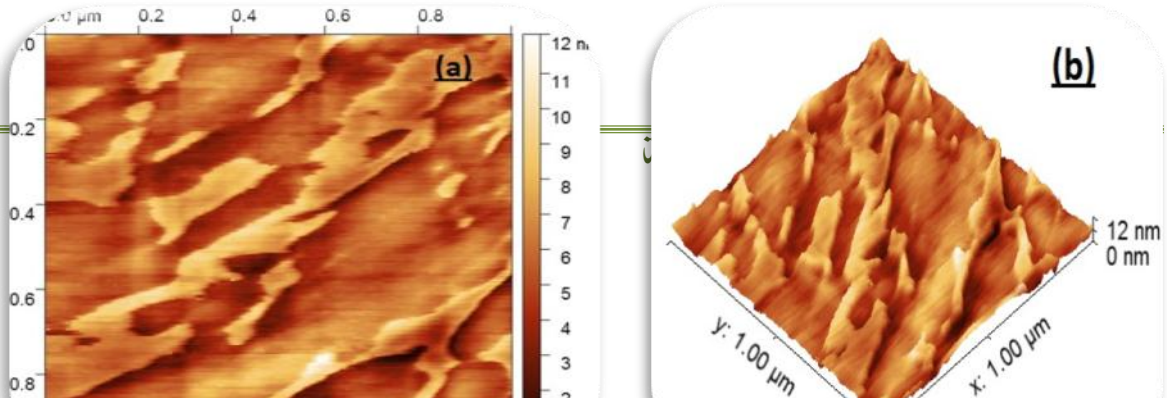
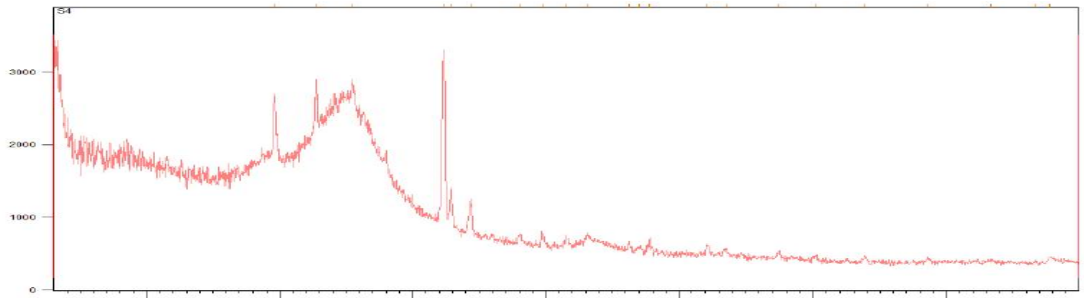


Fig (3) shows AFM images of the compound (GO)

3.2 Synthesis and characterization of reduced graphene oxide (RGO)

The X-ray diffraction of RGO shows an angular value at $2\theta=32.26^{o}$, indicating the arrangement of the sheets along the stacking direction. This confirms that the sample is made of a few layers of RGO, with an interlayer distance of 0.74, which is smaller than GO due to the removal of most of the functional groups.



Fig(4): X-ray diffraction of RGO

SEM images of compound (RGO) showed a decrease in the thickness of the sheets to 27.07 nm (a), with clear wrinkles on the sheets, while the oxidation state remained at the edges and in some areas (b).

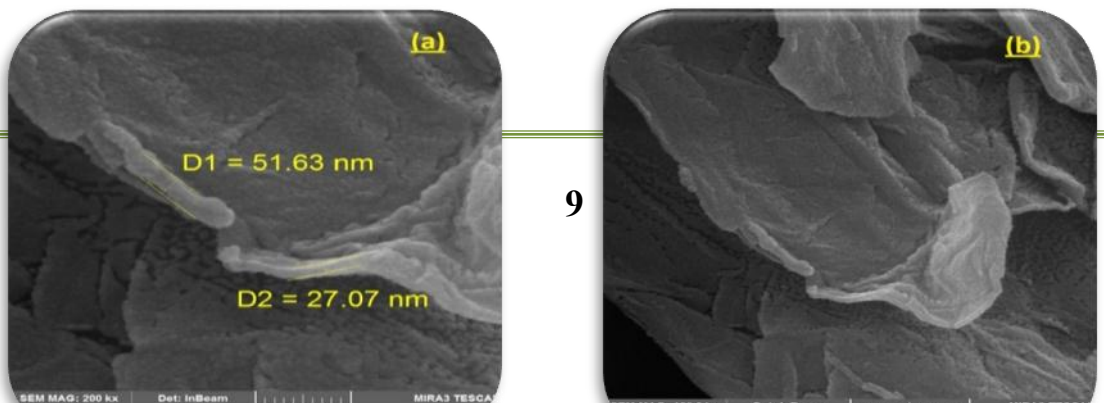
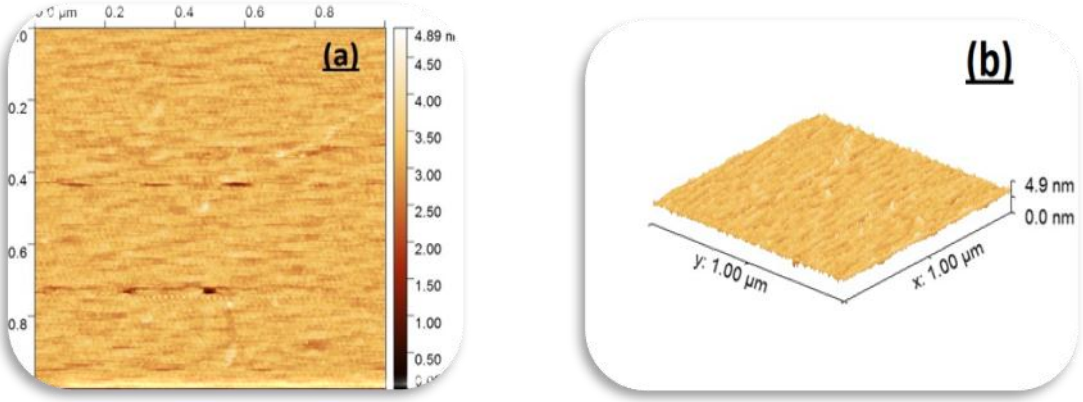


Fig (5) shows SEM images of the compound (A2)

As for the AFM images, they showed that the RGO in some areas on the surface still retained the oxygen groups (a) (b).



Fig(6): AFM images, RGO

3.3 Synthesis and characterization of reduced graphene oxide nanocomposites(A3)

Compound A3 showed different X-ray diffraction angle values than RGO, as the doping with the aromatic compound gave interlayer spacing values of 0.27, 0.22 A^{o}, which differed from the values of RGO, which had a d value of 0.71, with angle values of 30.71 and 30.29, with a grain size of 20.95 and a number of layers of 64.1

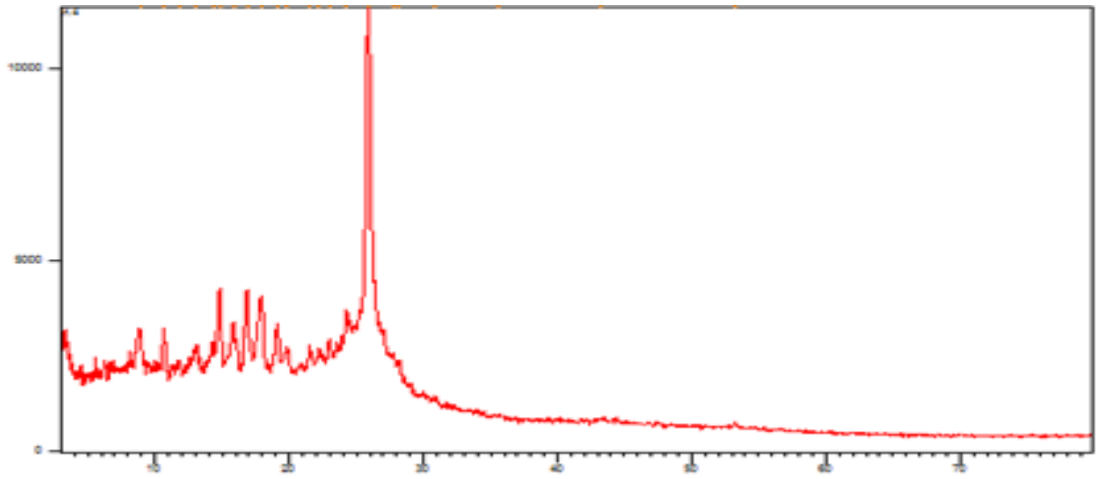


Fig (7) shows the XRD spectrum of the compound (A3)

SEM images show a combination of nanosheets measuring 28.60 with bends and layer stacking without fracture and grooves in the overlapping material on the surface.

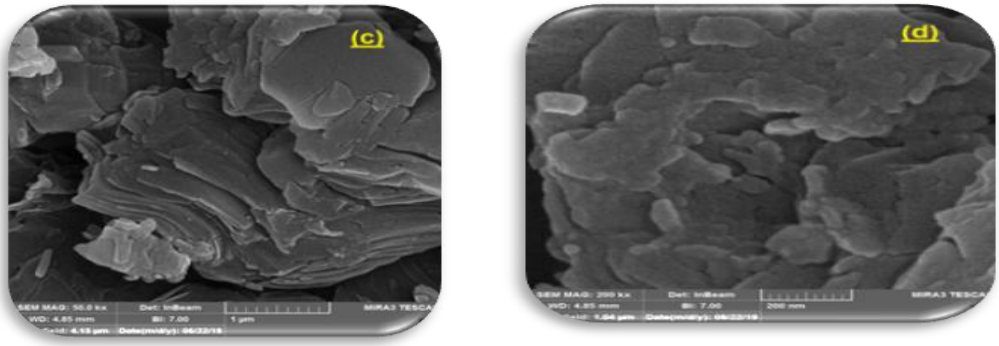


Fig (8) shows the SEM of the compound (A3)

3.4 Synthesis and characterization of reduced graphene oxide nanocomposites(A4)

The X-ray spectrum of the compound showed different angle values and the doping with the aromatic compound gave values for the distances (0.66, 0.22) A^{o}, and the d values were 0.77, and the grain size was 18.36 with a number of layers of 21.66.

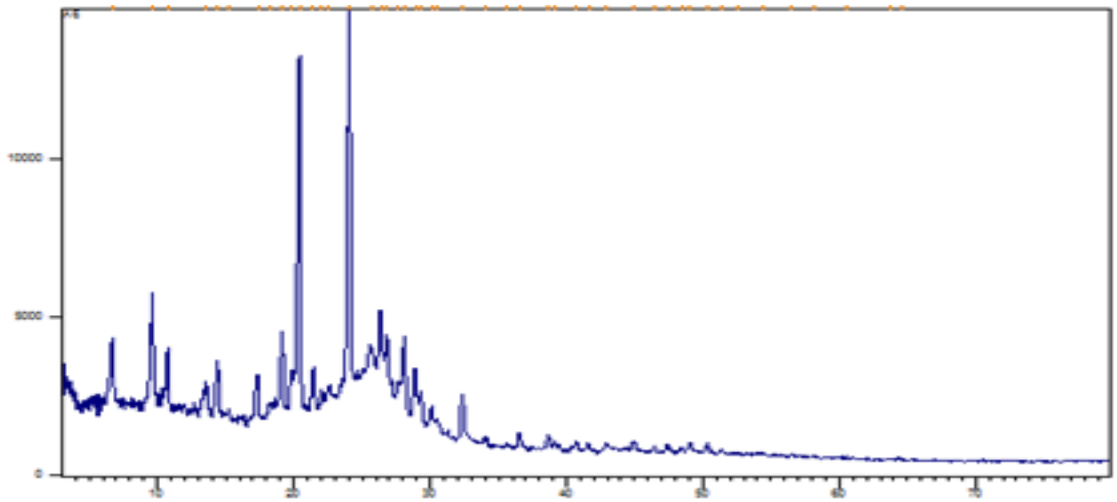


Fig (9) shows the XRD spectrum of the compound (A4)

In the SEM images, it was shown that there are spherical aggregates on the surface and edges of the RGO sheets, with grooves in the composite material. It was noted from XRD calculations that the grain size increases after adding the composite material and leads to an increase in the convergence of the layered sheets, and with it the number of layers in the layered aggregates increases.

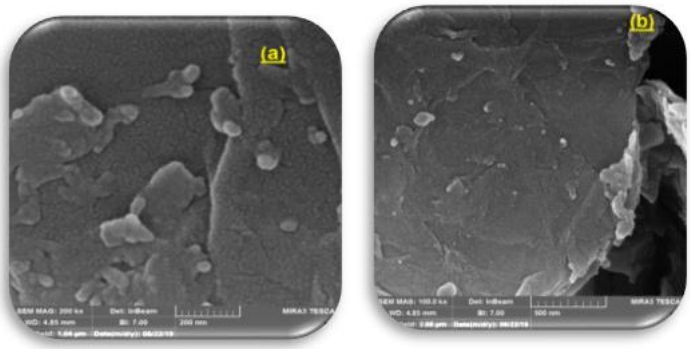


Fig (10) shows the SEM of the compound (A4)

3.5 Evaluation of the bacterial biological activity of some prepared compounds

Biologically, it works against Gram-positive and Gram-negative bacteria in distinct ways. We test the produced chemicals for their biological efficacy. In this case, we employed three distinct bacterial strains: Bacillus, Shigella, and Salmonella. The medical significance of certain microbes led us to select them. Numerous diseases are caused by these microorganisms. Compounds (A3, A4) show that they can block the growth of bacteria, both good and bad; the effect is stronger at larger concentrations. Additionally, we employed

concentrations of (50, 100, 150) ppm, with 150 ppm exhibiting the greatest inhibition and 50 ppm the least. Gentamicin is the antibiotic that is used to compare the efficiency of inhibition .At a concentration of 150 ppm, compound (A4) inhibited bacilli the most, whereas compound (A3) inhibited negative bacteria the most, with a difference of 18.8%. This is because the compound has a large surface area (A3).

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تحضير بعض المتراكبات النانوية المزينة ببعض المركبات الدوائية وتقييم فعاليتها الحيوية

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مستخلص البحث:
تتضمن الدراسة تحضير مركبات نانوية مزينة بمركبات دوائية وتقييم فعاليتها البيولوجية . تستخدم هذه المركبات في المقام الأول مواد نانوية كاوكسيد الكرافين المختزل . يسلط البحث الضوء على تركيب هذه المواد وتشخيصها من خلال تقنيات مختلفة مثل حيود الأشعة السينية(XRD) , والمجهر الإلكتروني الماسح , (SEM)ومجهرالقوة الذرية (AFM) . تشير النتائج إلى فعاليتها كمركبات مضادة للبكتيريا ، حيث أظهرت المركبات النانوية معدلات تثبيط أعلى عند تركيزات متزايدة مقارنة بالمضادات الحيوية القياسية.

الكلمات المفتاحية: الكرافين، أوكسيد الكرافين المختزل، المتراكبات النانوية.

ملاحظة : هل البحث مستل من رسالة ماجستير او اطروحة دكتوراه ؟ كلا