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Study of the Biological Activity for Reduced Graphene Oxide Decorated with Some Amine Compounds and Triazoles - Schiff Bases

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Abstract
In the paper focuses on the synthesis and characterization of Schiff bases using 1,2,4-triazole and graphene oxide (rGO) compounds, along with their applications. Specifically, it explores the conversion of graphene-to-graphene oxide and reduced graphene oxide (RGO) and their reactions with thiocarbhydrazone. The study shows how to prepare Schiff bases from different aldehydes and thiocarbhydrazides and then combine them with RGO that has been modified with sulfadiazine to make nanocomposites. These synthesized compounds were investigated for structural properties using infrared spectroscopy, X-ray diffraction, and FESEM imaging. In addition, the antibacterial activity of the resulting materials was evaluated, showing significant bacterial inhibition, especially against E. coli, with varying efficacy depending on the concentration of the material.

Keyword: Sulfadiazine-linked reduced graphene oxide, Thiocarbazaide- Schiff base .

Introduction
Graphene comprises of 2D- level sheets; the thickness of the sheets is generally equivalent to the breadth of one carbon molecule. The carbon particles are in the condition of sp^2 hybridization. In actuality, carbon iota hybridization in GO is principally sp^3. This is because every carbon molecule in GO is being clung to four different iotas, framing a tetrahedral structure, which makes it non-conductive*(Zhu, Y., Murali, S., Cai ,W,2010)*. In adjoining districts, some carbon iotas are in a sp^2 game plan, where every carbon is clung to three others and structures a twofold bond with one, making a planar design of hexagons. These hexagonal rings resemble the honeycomb structure, forming a network of flat hexagonal surfaces similar to a honeycomb lattice, known as graphene. The sp hybridization exists in all types of graphene nanosheets in different forms. It is found in certain bonding sites or double bonds *(Wang, H., Yuan, X., et al,2013)*.This is because of the way

that nanographene has an electrical conductivity property, and consequently its valence and conduction groups are associated at six places where the energy hole is zero*(Fedirko, V. A,2014)*. In this way, it has high conductivity contrasted with other nanomaterials and non-nanomaterials, and even outperforms metals in conductivity*(Savage, N ,2012*). This is why it is referred to as a "carbon super" *(Nair, R. R., Blake, P., Grigorenko, A. N., et al,2008)*and it also has high optical transparency.

Thiocarbhydrazones can exist in thioenol and thioketone frames and can go about as connecting locales as different wellsprings of sulfur considering different underlying potential outcomes and stereochemistry going from tetrahedral to octahedral to square planar. Thiocarbhydrazide promptly consolidates with two carbonyl atoms to deliver Schiff bases. An enormous number of reports have seemed to portray the union of such mixtures by changing the aldehyde utilized*(Emera, A.; Khalil S.; Salib, K., J. Coord. 1995,et al 2000).*We have revealed the combination and portrayal of a progression of symmetric and uneven Schiff bases*(Esmadi, F.; Irshaidat, T., 2000, Esmadi, F.; Ali, O.; Irshaidat, T.,2010).*The natural action of a portion of these mixtures has been contemplated*(Saleh, N.; Khabour, O.; Esmadi, F.; Al-Kofahi, I., 2010).*Continuing with our benefit in Schiff base collects, we have loosened up the work to the mixture and depiction of potential Schiff bases-thiocarbhydrazides as ligands got from the development of thiocarbhydrazide and heterocyclic aldehydes with graphene oxide. In this unique circumstance, we blended another Schiff base from thiocarbhydrazones and other known thiocarbhydrazones and responded them with graphene oxide nanocomposites to study and analyze their coordination conduct. The organic action of the subsequent mixtures has likewise been examined.

Experimental

1**- The Methods for Preparation Nanoparticles (Graphite - Graphene Oxide - Graphene)**

1-1 Preparation of ground wheat straw and wheat straw graphite

The preparation process involved two steps:

First: Planning of wheat straw: New wheat stalks (WSS) 1 g referred to tests were gathered and washed a few times in refined water (3 × 50 mL) to eliminate silica/dust and different foreign substances. They are then spread and passed on to dry at room temperature of around 35°C to a steady weight is accomplished. From

that point onward, they were grounded and used for the evaluation of the procedures. This was often repeated severally until a required amount for all subsequent tests was provided *(Ban, D.S.2022).*

Second: Preparation of wheat straw-derived graphite: A suitable amount of ground wheat straw stalks was placed in a small ceramic crucible. This crucible was then placed at the center of a larger ceramic crucible, and the space between the crucibles was filled with a sufficient amount of powdered graphite. The crucibles were fired (heated to combustion temperature) in a furnace with a gradual temperature increase (12°C per minute) from 30°C to 700°C for one hour

1-2 Preparation of graphene oxide (GO):

Place 46 ml of amassed sulfuric corrosive in a fitting carafe in an ice shower until warm harmony is accomplished. Slowly add 0.0176 moles (1.5 g) of sodium nitrate north of 15 minutes, blending consistently at a temperature of 0°C. Then, progressively add 2 grams of arranged graphite to the combination north of 10 minutes. Slowly add 0.042 moles (6 grams) of potassium permanganate north of 15 minutes, keeping the temp. beneath 20°C. Leave the combination in the ice shower for 5 minutes, then permit attractive mixing at room temperature (25°C) for 2 hours.

Add 46 ml of refined water dropwise north of 20 minutes, then, at that point, raise the temperature to 98°C and keep warming for 20 minutes. In this manner, add 140 ml of warm refined water (50°C) and mix the combination for 10 minutes at room temperature. Then, at that point, add 15 ml of 30% hydrogen peroxide and mix for 30 minutes. Add 300 ml of refined water and leave the blend undisturbed for 24 hours. Gather the subsequent item utilizing a nanofiltration framework, wash the encourage once with 10% hydrochloric corrosive arrangement (30 ml), and wash multiple times with deionized water (30 ml each) until arriving at a pH of 7. At long last, gather by filtration and permit drying at a temperature of 60-70°C until a consistent weight is achieved *(Dalaf, A. H., Jumaa, F. H., & Jabbar, S. A. S.2018).*

1-3 Preparation of Reduced Graphene Oxide (RGO):

The sample was prepared by adding an appropriate amount into a ceramic crucible, then placing it in the microwave at 500°C for 3 minutes *(Bansal, S. A., Singh, A. P., & Kumar, S.2019).*

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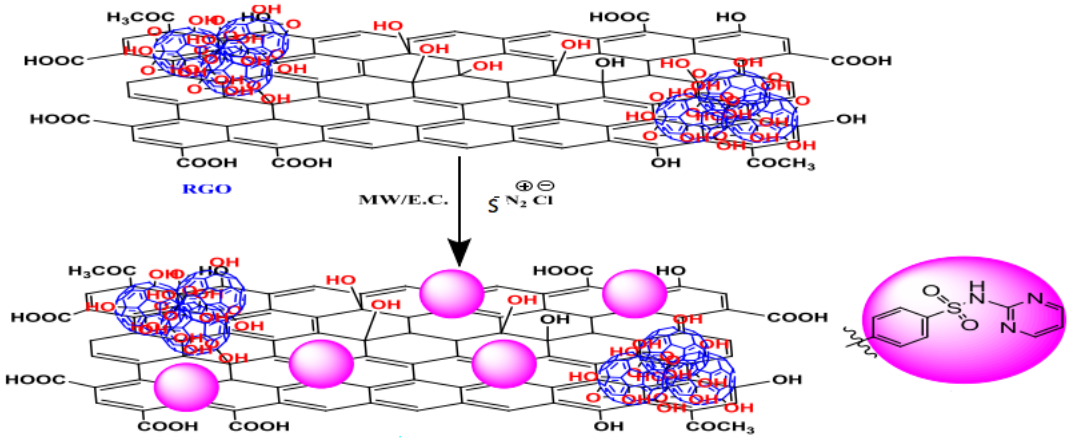
2- Preparation of Sulfadiazine-linked reduced graphene oxide (S – RGO)

Planning was completed by dissolving 0.2 grams of Sulfadiazine in a measuring glass containing an acidic arrangement (5:5 water: 37% concentrated hydrochloric corrosive), while keeping up with the arrangement temperature inside the scope of 0-5°C, with persistent mixing. In another recepticle, 0.07 grams of sodium nitrite was broken down in the littlest conceivable measure of refined water, then, at that point, added to the primary arrangement while keeping the temperature inside the scope of 0-5°C utilizing an ice shower, with persistent blending for 30 minutes in a dull climate. The subsequent item was not described however was utilized straightforwardly in the following stage:

0.1 grams of diminished graphene-oxide was broken up in 25 ml of refined water, then positioned in a ultrasonic shower until it turned out to be clear. The arrangement was then moved to an electrochemical cell comprising of two terminals associated with a voltage of 1.5-1.6 V, with consistent blending. The arrangement from Stage 1 was then added to the phone with cooling in an ice shower, and mixing was kept up with for 24 persistent hours. *(Dalaf, A. H., Jumaa, F. H., & Jabbar, S. A. S.2018).* The arrangement was washed a few times with deionized water, separated utilizing a nanofiltration vacuum, and left to dry until a steady weight was achieved.



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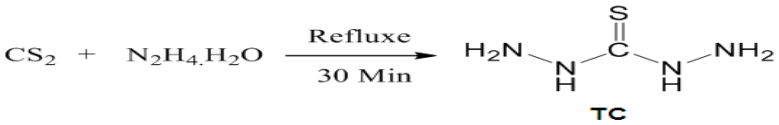
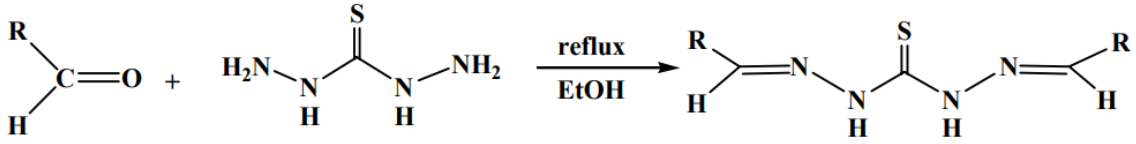
3- Preparation Thiocarbazaide (TC):

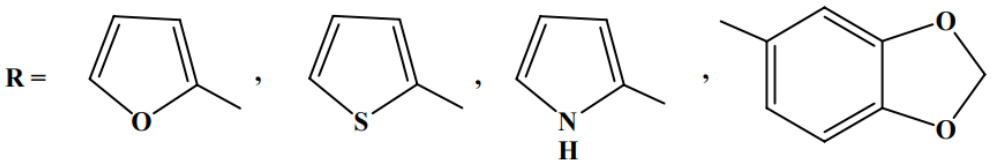
0.113 mol (5 ml) of carbon disulfide was added gradually over 10 min to (0.632) mol (20 ml) of aqueous hydrazine (80%) in a flask at 0°C with consistent blending. The response blend was warmed for 30 min, then cooled in an ice shower. The encourage shaped was gathered *(Sidiqi, K.; Khan, S.; Nami, S.; El-Ajaily, M.,2007)*



4-Preparation thiocarbazaide-schiff base bisfurfuralthiocarbohydrazone (tch-fu) bispyrrole-2-carboxaldehydethiocarbohydrazone(tch-py) bisthiophene-2-aldehyde thiocarbohydrazone (tch-th) and bispiperonaldehydethiocarbohydrazone(tch-pi).

For each compound 20 mmol of the contrasting aldehyde in 50 ml of ethanol was added to a warmed game plan of thiocarbohydrazide 10 mmol in 50 ml of ethanol the blend was then refluxed 2 hours for tch-fu 3 hours for tch-py and tchpi and 30 minutes for tch-th for tch-fu water was used as the dissolvable while for tch-py several drops of acidic destructives were added preceding refluxing .The resulting solids were recrystallized from a comparable dissolvable used in the reaction totally washed with ethanol followed by ether and subsequently dried for the present at 70°C *(Esmadi, Fatima 2013).*





5- Preparation Sulfadiazine - Reduced Graphene Oxide - Thiocarbazaide- Schiff base.

Four thiocarbohydrazone Schiff bases were synthesized. Among them, bis(piperonaldehyde) thiocarbohydrazone (Tch-Pi) appears to be novel, as no prior reports exist to the best of our knowledge. The remaining compounds, however, have been documented in previous studies *(Mahdi, M. F., & Raauf, A. M. 2024, Bacchi, A.; Carcelli, M.; Pelagatti, P.; Pelizzi, at al1999, Skoog, D. A.; West, D. M.; Holer, F. J.,1988).*

5-1 Preparation Sulfadiazine - Reduced Graphene Oxide - Thiocarbazaide- bis(piperonaldehyde) (S-RGO- Tch-Pi).

It was prepared using the thermal melting method in the solid phase, where the reaction took place until the color of the melt and the nature of its consistency changed. The reaction took place between Sulfadiazine - Reduced Graphene Oxide and Thiocarbazaide- bis(piperonaldehyde).

Results and Discussion

While the infrared (IR) range of the base substance [Graphite], a band was seen at 3404 cm^{-1}, credited to the Goodness bunch. Assimilation groups were likewise seen at 3080 cm^{-1}, relating to the extending of the fragrant $C-H$ bond, and at 2964 and 2844 cm^{-1}, related with the aliphatic $C-H$ bunch. Furthermore, an assimilation band was noted at 1666 cm^{-1}, relating to the extending of the carbonyl $(C=O)$ bond. Groups were likewise seen at 1583 and 1479 cm^{-1}, connected with the extending of the sweet-smelling C=C bond, and a retention band at 1353 cm^{-1}, relating to the O-C gathering. These groups were near those announced in the study *(Mohan, A. N., & Panicker, S. Facile 2019).*

The X-beam range of the compound [Graphite] showed a worth of 2θ point at 28.3867, an interlayer separating d=3.14418, and various layers of n=7. Fig (1)

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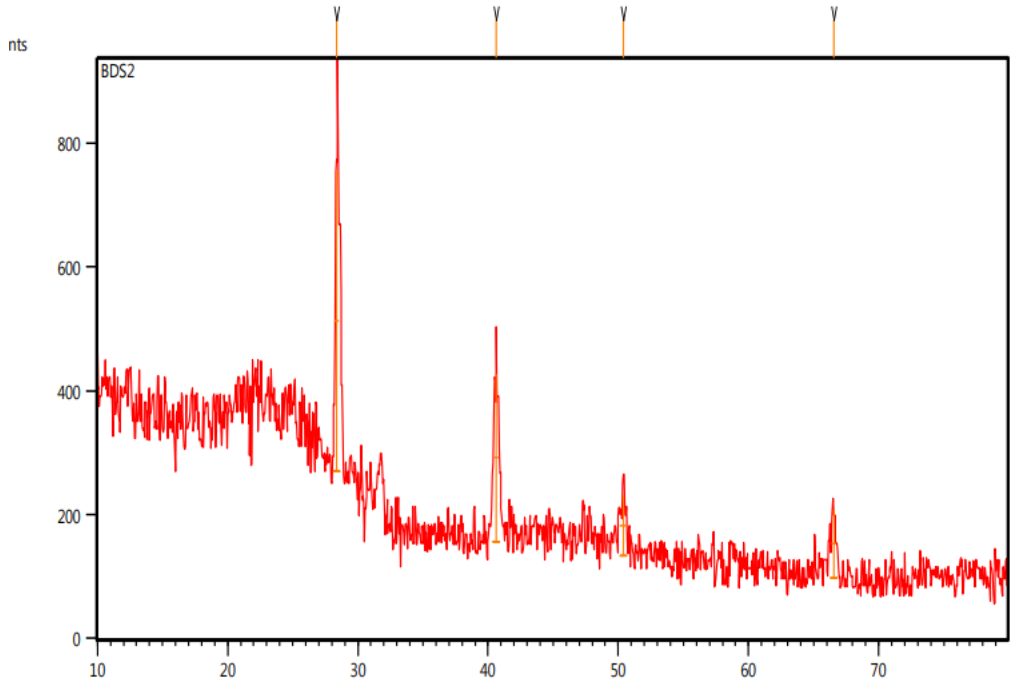

Fig(1) : The X-ray spectrum of the Graphite

From the FESEM morphological images of graphite, large cracks were observed in a, which is attributed to the loss of part of the sample's weight by heat, with possible re-bonding to form the sheets by radical mechanics, with clear cracks remaining b, with nano-thickness of the sheets c, and the presence of spherical structures of different sizes on the surfaceD.Fig(2)

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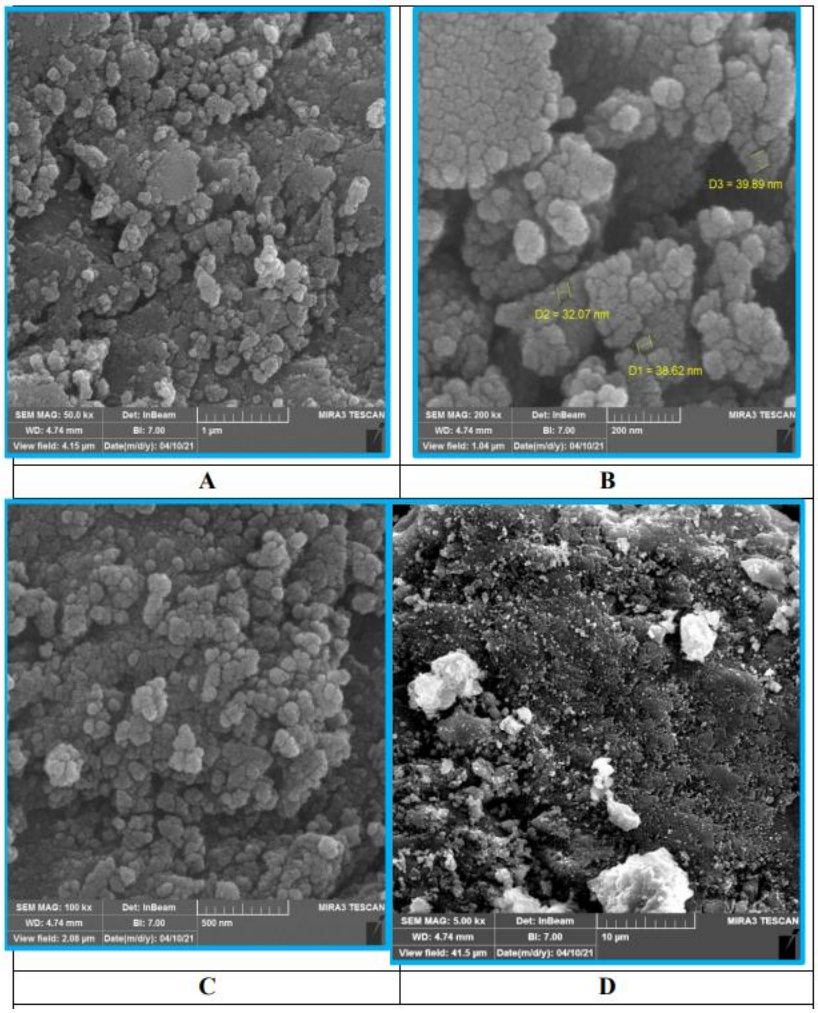

Fig(2) : FESEM morphological images of graphite

While focusing on the infrared reach (IR) of the compound(GO), it was seen that A band appeared at a repeat of (3409) cm^{-1},a band showed up at a recurrence of (3409) cm^{-1}, has a place with the (Gracious) bunch, and the presence of retention groups at a recurrence of (3043) cm , has a place with the extending of the fragrant (CH) bond, and the presence of two groups at a recurrence of (2964, 2871) cm^{-1}has a place with the aliphatic (CH) bunch, as well as the presence of an ingestion band at a recurrence of (1724) cm^{-1}, has a place with the extending of the carboxylic (O = C) carbonyl bond, and the presence of two assimilation groups at a recurrence of (1589) 1488) cm^{-1}, has a place with the extending of the sweet-

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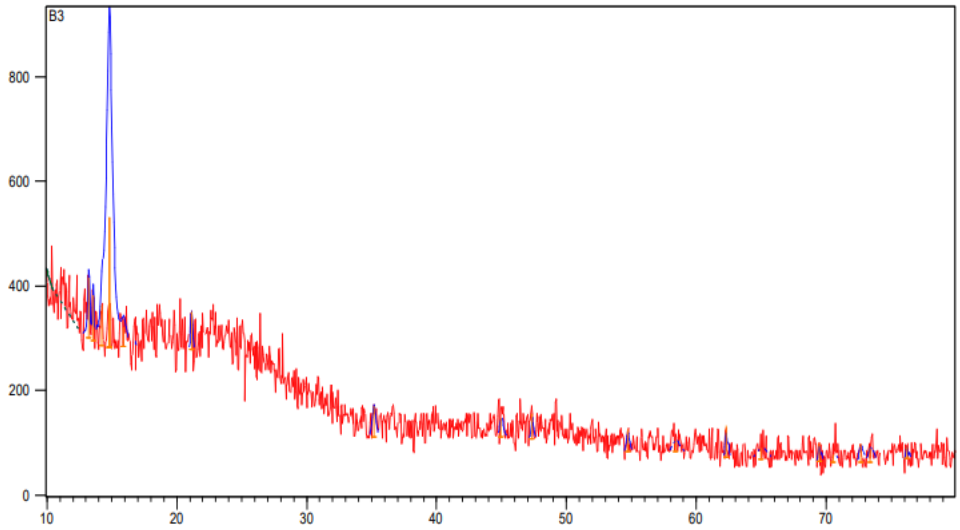
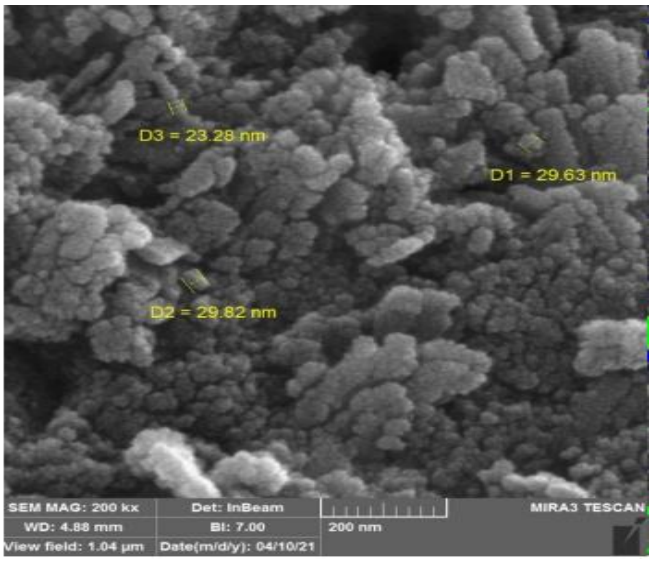
smelling (C = C) bond, and the presence of The assimilation band at recurrence (1328) cm^{-1} has a place with the (O – C) bunch, and the presence of the retention band at recurrence (1244) cm 152 has a place with the (C-O-C) bunch.

The X-beam range of the compound [GO] showed a worth of 2θ point at14.7859 and for interlayer distances of d=5.99144 and number of layers of n=1. It was noticed that these qualities are near the writing *(Chen, R., Wang, X., Li, X., Wang, H., He, M., Yang, L., & Wei, D. A.2021)* Fig(3)



Fig(3) : X-ray spectrum of the compound [GO]

From the FESEM morphological pictures of the GO example, it was seen that the nanoscale aspects of the sheet thickness were protected with a particular width of the framed sheets. The totals coming about because of the oxidation with potassium permanganate (carboxyl gatherings) were gathered. Fig(4)



Fig(4) : FESEM morphological images of the GO sample

While the infrared range (IR) of the [RGO], it was seen that a band at 3396 cm^{-1} for (OH), The presence of a band at a recurrence of 3058 cm^{-1} is expected to the fragrant (CH) , and the presence of two groups at 2941-2846 cm^{-1} is aliphatic (CH) , and The appearance of an absorption band at (1720) cm^{-1} for carboxylic carbonyl gathering. *(Saleh, R. H., Rashid, W. M., Dalaf, A. H.,at al 2020, Hasan, H. A., Ali, K. F., & Mehdi, W. A.2024).*

The X-beam range of the [RGO] composite showed a worth of 2θ point at 23.37 and for interlayer distances of d=3.80357. It was noticed that these qualities are near the writing. Fig(5)

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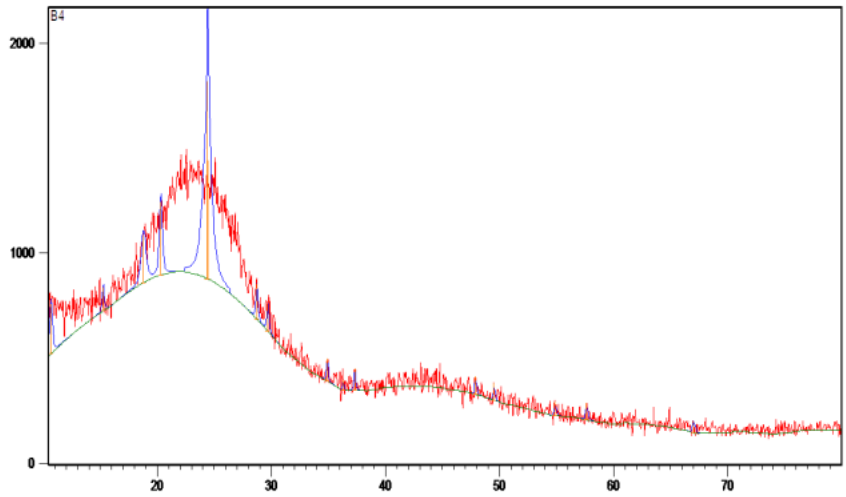

Fig(5): X-ray spectrum of the [RGO]

From the FESEM morphological pictures of the RGO , two peculiarities were noticed: the first is the high equatorially (level high) of the outer layer of the sheet A with the surface breaks remaining. The second is spherical particles gathered on the surface to give another type of shape C with the presence of regular and close holes above the surfaces of the plates with close diameters D. Fig(6)

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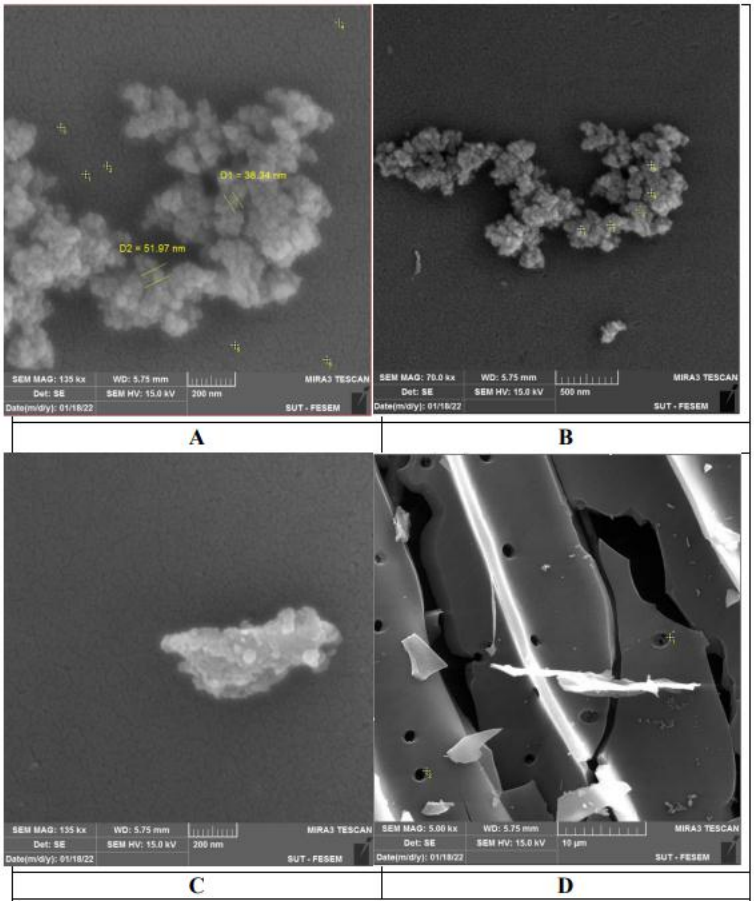

Fig (6) : FESEM morphological images of the RGO

While the infrared (IR) range of the compound [S-RGO], a band was seen at 3330 cm^{-1} to (OH), and a band at 3191 cm^{-1} to the NH. A band showed up at 3064 cm^{-1}, to the Aromatic CH ,and 2923 and 2864 cm^{-1} related with the aliphatic CH. An assimilation band at 1718 cm^{-1} was noticed, to the carboxylic carbonyl group ($C=O$), and a band at 1649 cm^{-1}, to the Azo methine ($N=C$). Two absorption groups showed up at 1587 and 1494 cm^{-1}, comparing to the aromatic $C=C$ bond. the band at 1325 cm^{-1} was noticed, relating to the SO_2 bunch, and the band at 1247 cm^{-1}, ascribed to the epoxy O-C . An assimilation band at 1143 cm^{-1} was noticed, relating to the N-C bond.*(Mahmood, S. A., Rauf, A. M., & Aburjai, T. 2024)*

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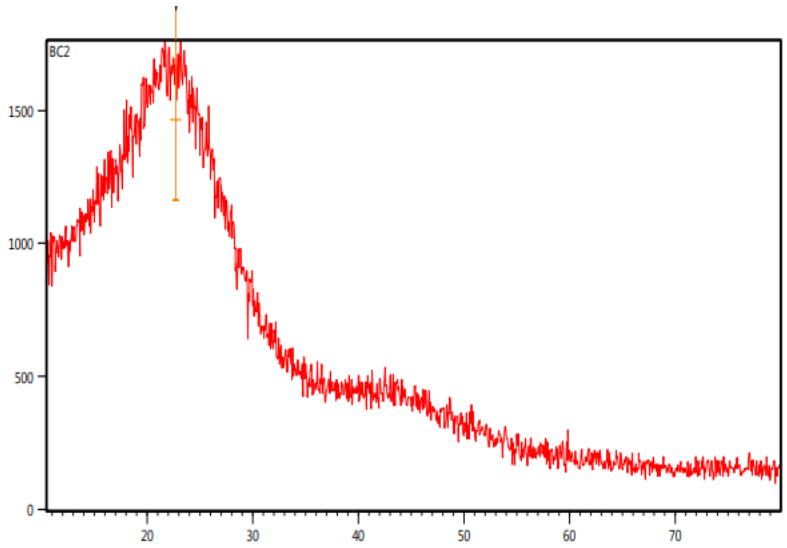
The X-beam diffraction range of the compound [S-RGO] shows a 2θ (22.73), with an interplanar separating of 3.91237 (d),and number of layers= 2. Fig(7).



Fig(7): X-ray diffraction spectrum of the compound [S-RGO]

FESEM morphological images of the sample[S-RGO] showed thickening on the edges of the sheet A with the presence of undulations in the areas of sheet cracks exclusively B with limited cracks within the sheet with good amplitude C. Also, the spread of the decoration was irregularly distributed on the sheet D.*(Al-Dulaimi, A. F., Al-Somaidaie, G. H., & Jumaa'h, M. M.2022)* Fig(8)

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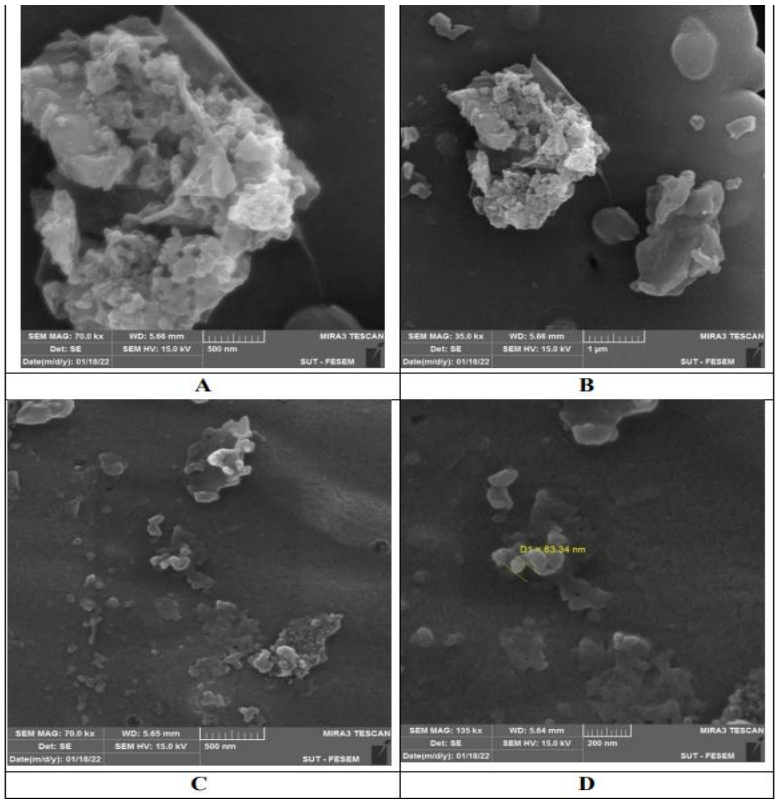
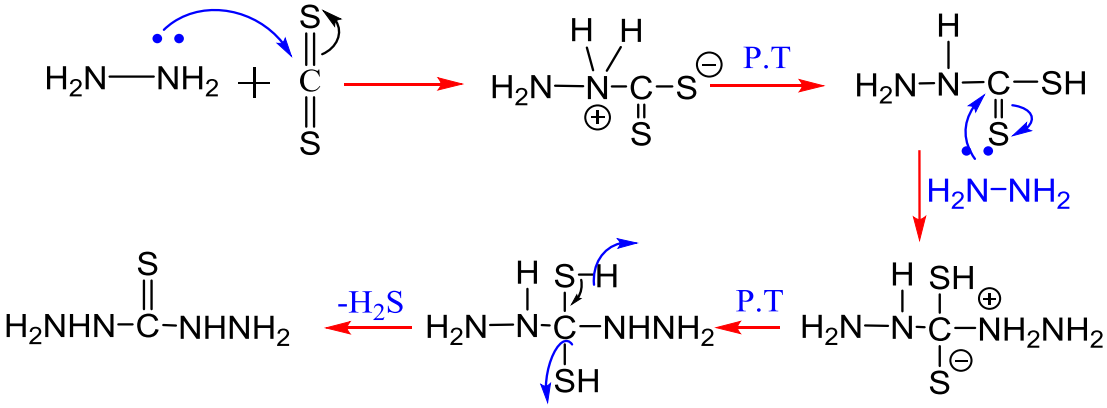


Fig(8): FESEM morphological images of the sample[S-RGO]

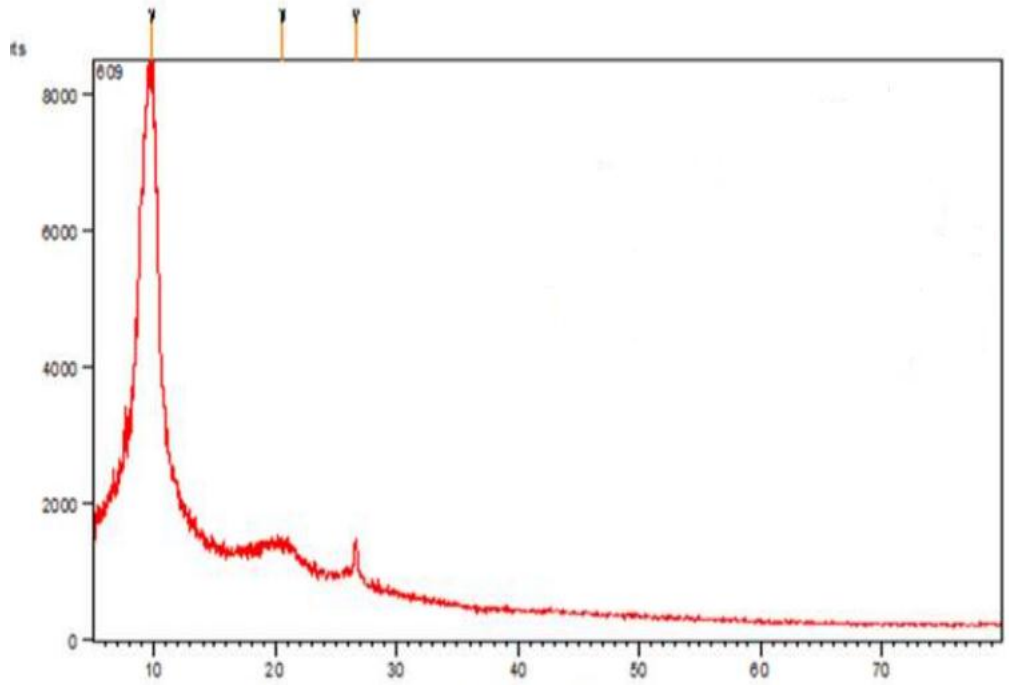
Thiocarbazide TC was prepared by condensation of a mixture of carbon disulfide and aqueous hydrazine, where white crystals were obtained according to the mechanism below:



Identified by appearance and some physical properties, such as melting point and color, as well as through infrared (IR) spectroscopy measurements. The IR

spectrum displayed peaks at 3273 and 3305 cm^{-1}, corresponding to the symmetric stretching frequency of the first amine group (νNH_2), while the second amine stretching appeared at 3204 cm^{-1}. Additionally, a peak at 1284 cm^{-1} was associated with the stretching of the $C = S$ bond ($\nu C{=}S$). The scissoring bending frequency of the (νNH_2) group was observed at 1643-1140 cm^{-1}, along with an overtone vibration for the NH bending at 3174 cm^{-1}, and a wagging (νNH) bending at 933 cm^{-1}, showing characteristic intensity. The data matched those reported in the literature (Esmadi, Fatima.2013).Fig(9)

The IR range for [Tch-Pi] showed two gatherings at 3147 and 3240 cm^{-1} (N-H. A band which appeared at 2994 cm^{-1} to $C - H$ of fragrant vibration and a band at 1626 cm^{-1} to $(C = N)$ vibration of the azomethine pack. A social event of three gatherings appeared in the locale 1519-1593 cm^{-1} for $C = C$ aromatic. A band as a result of $C - O$ vibration appeared at 1252 cm^{-1} and another band at 771 cm^{-1} to $C = S$ bond vibration. A weak band at 2600 cm^{-1} was given out to $S - H$ bond vibration of the thiol tautomer. While the infrared (IR) range of the compound [S-RGO-Tch-Pi], a band was seen at 3330 cm^{-1} comparing to theOH, and a band at 3191 cm^{-1} credited to the NH bunch. A band showed up at 3064 cm^{-1}, relating to the fragrant CH bunch, three band appeared in the region 1519-1593 cm^{-1} to Aromatic $C = C$ vibrations. A band due to $C - O$ vibration appeared at 1252 cm^{-1} and another band at 771 cm^{-1} was attributed to $C = S$ bond vibration.The X-beam range of the compound [S-RGO-Tch-Pi] showed a point worth of 2θ = 20.6, 26.4 ,9.8, with a slight expansion in the distances between the layers d = 0.9013 nm, which makes sense of the lessening in the quantity of layers n = 4 and shows that the material has a lower thickness and more prominent harshness as in the Fig(10)



Fig(10): X-ray spectrum of the compound [S-RGO- Tch-Pi]

From the FESEM morphological pictures of the example [S-RGO- Tch-Pi], it was noted that there is a high roughness attributed to the additional peeling of the sample. This may lead to an increase in the surface area with an increase in the thickness on the edges of the plate with the presence of accumulation on the surface as well as the appearance of cracks and accumulation on its edges and on the edges of the plates.Fig(11)

Fig(11):FESEM morphological images of the sample[S-RGO- Tch-Pi]

Antibacterial activity

In the communication of microscopic organisms with[S-RGO] and [S-RGO-Tch-Pi] in suspensions (Table 1), a deficiency of bacterial cell feasibility was uncovered in a focus and time-subordinate way, with a more prominent loss of reasonability of Gram-positive microorganisms.

Table 1 Bactericidal character of [RGO],[S-RGO] and [S-RGO-Tch-Pi]

Material	Bacterial cell model	Comments
RGO	$E.coli$	68.2% hindrance utilizing plate counting at [RGO] = 85 $\mu g\ mL^{-1}$, loss of cell honesty,
$S-RGO$	$E.coli$	100 percent obliteration at 4 $mg\ mL^{-1}$
$S-RGO-Tch-Pi$	$E.coli$	97.9 % restraint utilizing plate counting at [S-RGO-Tch-Pi]= 85 $\mu g\ mL^{-1}$; loss of cell respectability; showed higher antibacterial movement.

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دراسة الفعالية البايولوجية لأوكسيد الكرافين المختزل المزين ببعض مركبات الأمين والترايازولات- قواعد شيف

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مستخلص البحث:
يركز البحث على تصنيع وتشخيص قواعد شيف باستخدام مركبات 1،2،4-ترايازول وأكسيد الجرافين (rGO)، إلى جانب تطبيقاتها. وعلى وجه التحديد، يستكشف البحث تحويل الجرافين إلى أكسيد الجرافين وأكسيد الجرافين المختزل وتفاعلاتهما مع ثيوكاربهيدرازون. تسلط الدراسة الضوء على طرق تخليق قواعد شيف من مختلف الألدهيدات وثيوكاربهيدرازيدات، تليها تراكباها مع RGO المعدل بالسلفاديازين لتكوين مركبات نانوية. تم تشخيص هذه المركبات المصنعة من حيث الخصائص البنيوية باستخدام التحليل الطيفي بالأشعة تحت الحمراء، والحيود بالأشعة السينية، وتصوير FESEM. بالإضافة إلى ذلك، تم تقييم النشاط المضاد للبكتيريا للمواد الناتجة، مما يدل على تثبيط بكتيري كبير، وخاصة ضد E. coli ، مع فعالية متفاوتة اعتمادًا على تركيز المادة.
الكلمات المفتاحية: أكسيد الجرافين المختزل المرتبط بالسلفاديازين، ثيوكاربازيد- قاعدة شيف.