International Journal of Sustainable Applied Sciences (IJSAS)
Vol. 3, No. 6, 2025: 333-344

Preparation of Arylidene Sulfacetamide Reduced Graphene Oxide Nanocomposites and Measure some of its Optical Properties

Tareq Bandar Mahmood[1], Yusra A Mohammed[2], Ali Fadel Ahmad[3], Ghazwan Hasan Al-Somaidaie[4]
[1]Directorate of Education in Salah Al-Din, Salah Al-Din
[2]College of Basic Education, Almustansiriyah University, Baghdad
[3]College of Education for Pure Sciences, University of Anbar, Anbar
[4]College of Education for Pure Sciences, Tikrit University, Salah Al-Din
Corresponding author: Yusra A Mohammed Yusra199881@gmail.com

A R T I C L E I N F O

Keywords: Nanocomposites, Reduced Graphene Oxide, Arylidenesulfacetamides

Received : 19, April
Revised : 20, May
Accepted: 30, June

A B S T R A C T

Nanocomposites based on Schiff base reactions with reduced graphene oxide were prepared, and their optical and mechanical properties were studied. The preparation methods included synthesizing Schiff bases using sulfacetamide and aromatic benzaldehydes, preparing graphene oxide via the modified Hummer's method, and reducing it with aqueous hydrazine. Infrared spectroscopy (FTIR) and (NMR) analyses confirmed the successful formation of the nanocomposites. The results revealed that the prepared composites exhibit unique properties, with optical transmittance increasing variably between 49% and 89%, and energy band gaps ranging from 3.5 to 4.0 eV. Scanning Electron Microscope (SEM) images demonstrated that these composites possess distinctive nanostructures, enhancing their hydrogen bonding capability and increasing their effective surface area. These properties make the composites suitable for diverse applications in optical, magnetic, and mechanical fields.

DOI: https://doi.org/10.59890/ijsas.v3i6.53
ISSN-E: 3025-5597
https://dmimultitechpublisher.my.id/index.php/ijsas

INTRODUCTION

Nanoscience is known for its interest in materials whose dimensions or one of its three dimensions (Z, Y, X) are within the nano dimensions so that they do not exceed 100 nm. It also studies their chemical and physical properties and knows the phenomena arising from this transformation in their sizes (1). The word nano is originally a Greek word meaning "dwarf" (Nanos) indicating small things (2), (3) This nanomaterial significantly improves their properties for example, adding carbon nanotubes changes the electrical and thermal conductivity properties of the material (4). Adding other types of nanoparticles may improve the optical properties and electrical insulation properties as well as mechanical properties such as hardness and strength. Graphene oxide reduction Reduced graphene oxide consists of thin layers of graphene oxide without oxygen-containing groups. It is a type of nanocarbon, and it is a sheet or several sheets, such that the number of sheets stacked on top of each other does not exceed 10sheets.

LITERATURE REVIEW

These sheets contain different effective functional groups such as epoxy, carboxyl, carbonyl and alcohol groups (5). Imines are known as Schiff bases or azomethines because they contain the azomethine group, which was first prepared in 1864 by the German scientist Hug Schif (6). The structural formula for Schiff bases is (RR'C=NR''), as each of (R, R', R'') can be hydrogen, alkyl groups, aryl groups or heterogeneous rings, and they may be similar or different (7).

METHODOLOGY

Preparation of N-(4-(Benzylidene) Amino Phenyl Acetamide Substitutes (T1-T3)

Mixing (5.35 g, 0.025 mol) of sulfacetamide (13,14,15) with 0.025 mol of suitable benzaldehyde substitutes, placed in a heat-resistant glass beaker without using solvent, where the mixture was heated in a sand bath, quietly and for (6-11) minutes until the melting point with slow movement until the nature of the molten reactant's changes in terms of colour, texture and shape. The product was collected and recrystallized with Eth-OH (8).

Preparation of Graphene oxide: GO (Modified Hammer Method) (T4)

A flask with a capacity of (600 ml) is placed in an ice bath and 46 ml of concentrated sulfuric acid is added with magnetic stirring, then 1.5 grams of sodium nitrate. After 15 minutes, 1 gram of graphite is added gradually over 12 minutes, then 5 grams of potassium permanganate is added to the mixture gradually over 10 minutes, with caution and maintaining a temperature below 20 degrees Celsius. After completing the addition, leave the mixture in the ice bath for 5 minutes, lift the mixture from the ice bath and leave it to stir magnetically for 2 hours, then add 46 ml of distilled water very slowly using a dropper for 20 minutes, then raise the temperature to 98°C for 20 minutes, then add 140 ml of warm distilled water 52°C and leave it to stir for 10 minutes at room temperature, then add 15 ml of hydrogen peroxide 30% and leave it to stir for 30 minutes, then add 300 ml of distilled water and leave the mixture for 24

hours, pour and collect once, then wash the precipitate with a 10% solution of hydrochloric acid once, then with deionized water five times (45 ml ^{x}5) using a centrifuge to pH = 7 for the filtrate.

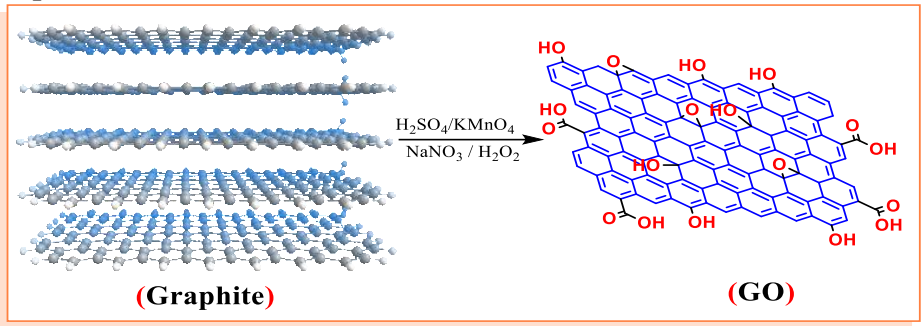

Figure 1. Preparation of Graphene oxide: GO (Modified Hammer Method) (T4)

Preparation of Graphene Oxide (T5)

0.1 g of graphene oxide is taken and placed in a round flask with a capacity of (50) ml and (1) ml of concentrated hydrochloric acid is added to it. Stir until the solution is homogeneous (it becomes a clear solution without suspended particles), then one mL of hydrazine (80%) is added to it and then heated at a temperature of 100) °C in the presence of a reflective condenser for two hours. The result is collected and washed with deionized water several times to remove excess hydrazine using a centrifuge, and it is dried in an oven at a temperature of (100) °C for (12) hours.

Preparation of Arylidene-Sulfacetamide Nanocomposites (T6 – T8)

0.101 g of reduced graphene oxide was dissolved in 10 ml of ethyl glycol with ultrasound for 15 minutes or until a clear nano solution was obtained. The prepared Schiff base derivatives (T3, T2 T1) were dissolved separately in (10) ml of ethyl glycol with ultrasound for fragmentation and to obtain clear solutions. Then the materials were spread in the sequence mentioned for the prepared solutions in the work method on the glass slide using a Spin coater device at a rotation rate of (6000 rpm) to obtain a thin layer. Then the sample was placed immediately after spreading in the oven at a temperature of 60-70 C for 15 minutes. After that, the sample was taken out and placed at room temperature until reaching a temperature close to the temperature of the glass slide. After that, the second layer was spread with the same steps.

RESULTS AND DISCUSSION

The infrared spectrum (FT-IR) of the compounds (T1-T3) prepared in (Fig1,2,3) showed the band of the (NH2) group compared to the novel compound, with the look of a new absorption range at 1685-1631 cm of the (C=N) group, and the 3498-3228 cm-^{1} which was assigned to the stretching of the (NH) group, and the appearance of a range at 1600-1519 cm-^{1} which was assigned to the (Ar-C=C) group.

The proton nuclear magnetic resonance spectrum also showed (1H-NMR) for the complex (T1), a single signal at the displacement of δ 1.67 is due to the proton (CH3), and the appearance of a high single signal at the displacement δ 2.51 is due to the protons of the solvent (DMSO), and the

appearance of multiple signals at the displacement δ (8.22, 7.78, 7.51, 7.31) is due to the protons of the aromatic rings, and the appearance of a signal at the displacement δ (8.39) is due to the proton (N=CH), and the appearance of a signal at the displacement δ (8.83) which is due to the proton (NH). The proton nuclear magnetic resonance (1H-NMR) spectrum of the compound (T3) showed a single signal at the displacement (δ 1.67) due to the proton (CH3), a high single signal at the displacement (δ 2.51) due to the protons of the solvent (DMSO), a single signal at the displacement (δ 3.01) due to the proton (N-CH3), the appearance of multiple signals at the displacement (δ 7.41, 7.15, 6.78, 6.48) due to the protons of the aromatic rings, the appearance of a signal at the displacement (δ 7.72) due to the proton (N=CH), and the appearance of a signal at the displacement (δ 8.41) due to the proton (NH).

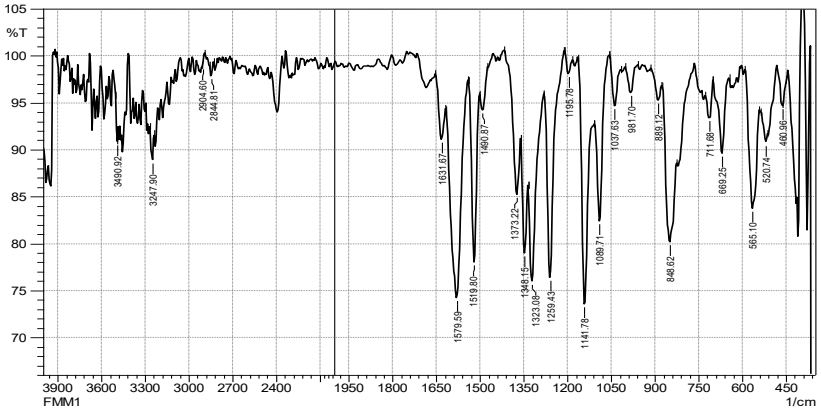

Figure 2. FT-IR Spectrum of Compound (T1)

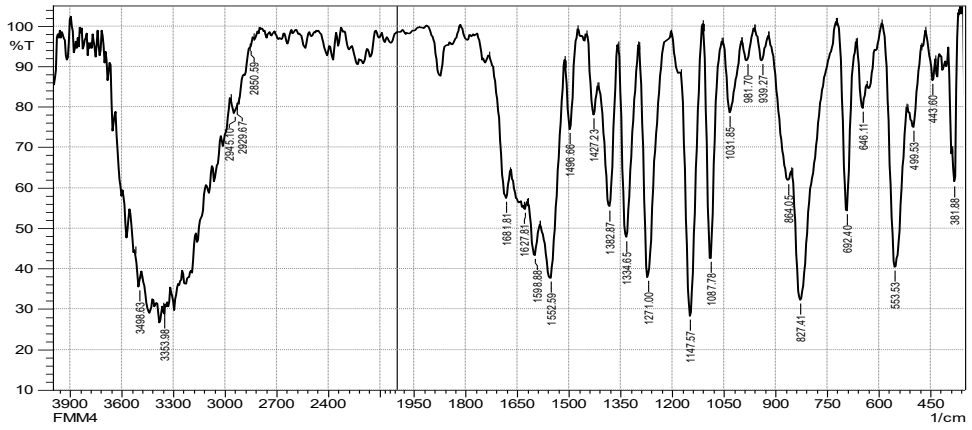

Figure 3. FT-IR Spectrum of Compound (T2)

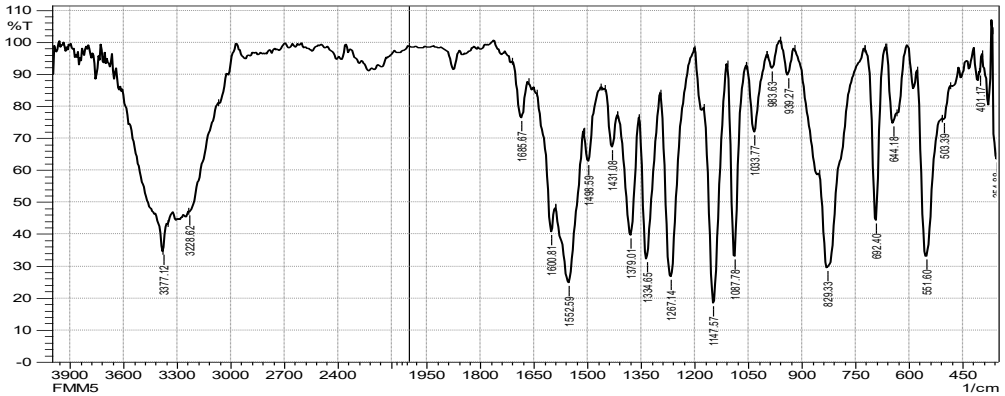

Figure 4. FT-IR Spectrum of Compound (T3)

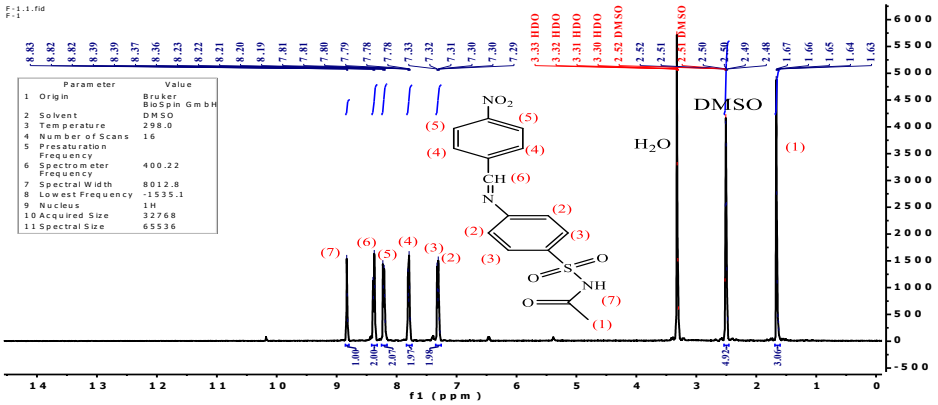

Figure 5. 1H NMR Spectrum of Compound (T1)

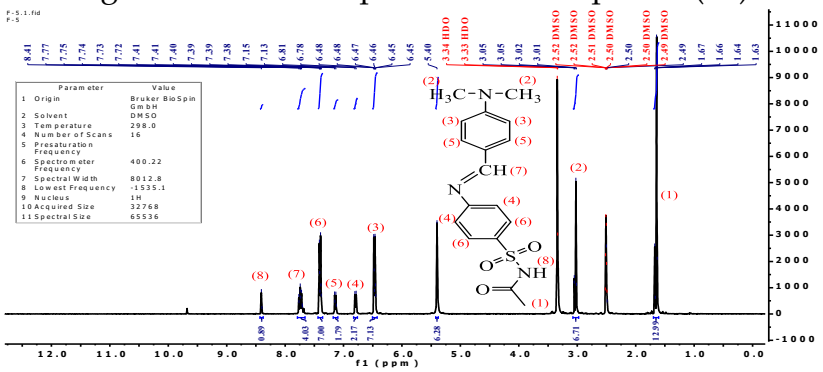

Figure 6. 1H NMR Spectrum of Compound (T3)

X-ray for graphene oxide GO showed an angle value of 2θ=11.82, in shows a value of 2θ at 32.26, for RGO.

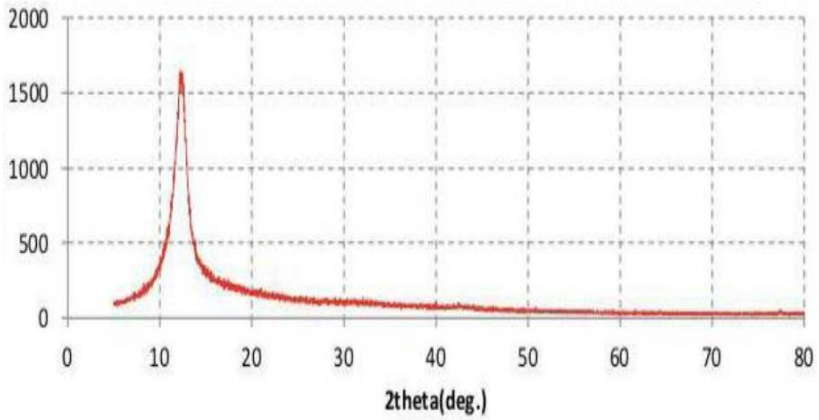

Figure 7. XRD Spectrum of Graphene Oxide

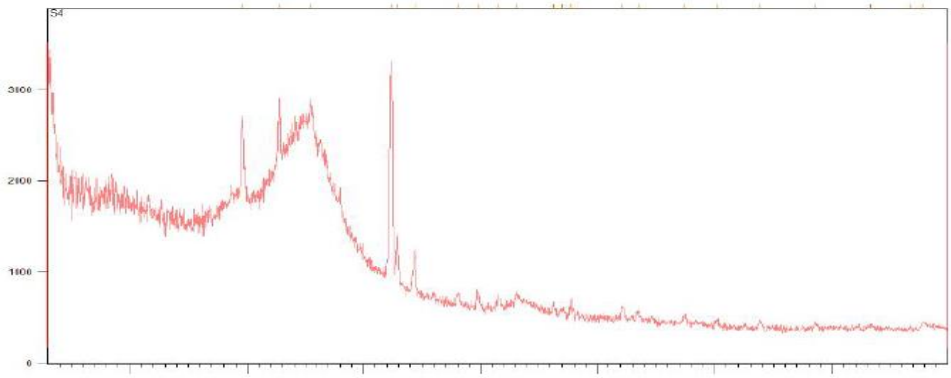

Figure 8. XRD Spectrum of rGO

It is also noted the differences in the expected density of the grains and thus their ability to possess the expected effective surface area. From the scanning electron microscope (SEM) images of the slide sample (T6).

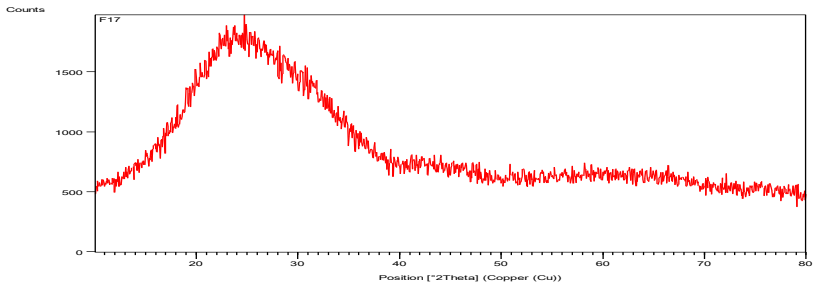

Figure 9. XRD Spectrum of Sample (T6)

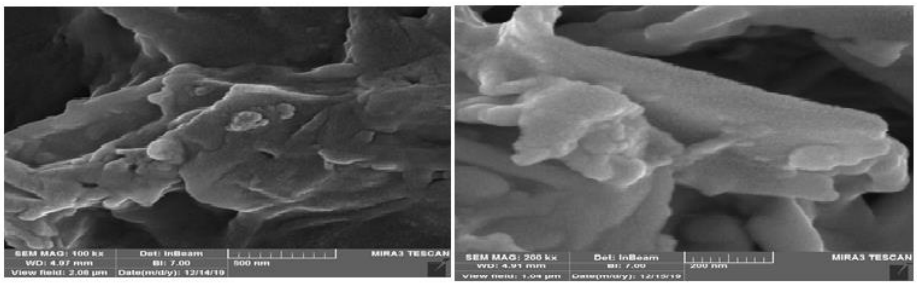

Figure 10. SEM Images of Sample (T6)

The composite T7 showed different angle values from GO, and the doping with the aromatic compound gave values for the interlayer distances of 0.34 A°, 0.39 A°, which differs from the value of GO, which had a d value of 0.74 and angle values of 25.77, 22.01 and an average grain size of 24.52 nm and a number of 62 plates. SEM images of sample (T7) show the presence of convergence between the plates with the separation of the layers.

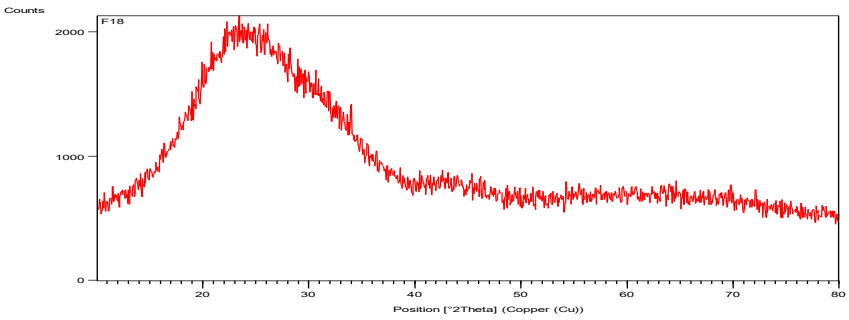

Figure 11. XRD Spectrum of Sample (T7)

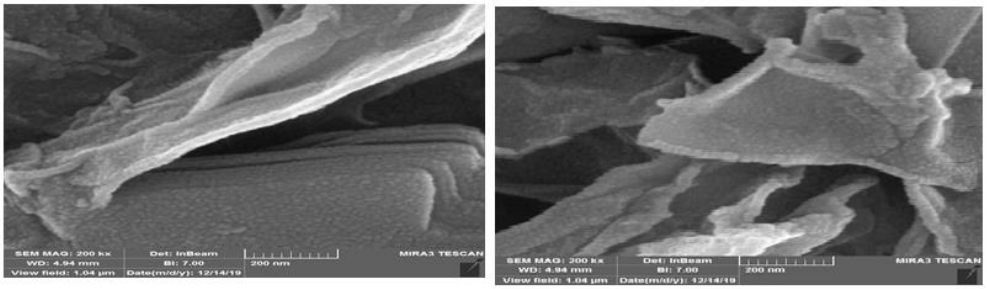

Figure 12. SEM Images of Sample (T7)

The composite T8 showed different angular values from GO, and the doping with the aromatic compound gave values for the inter-spaces of 0.30 A°, 0.34 A°, 0.44 A°, which differs from the value of GO, which had a (d) value of 0.74 and angular values of 19.90, 29.01, 25.02 and an average grain size of 40.19 nm and a number of 91 plates.

The sample (T8) showed morphological images of the SEM microscope showing the role of compound (T4), which differs in its composition from (T7, T6) As it carries an electron-pushing group, the spacing between the sheets and layers in Figure (13- a) and Figure (13-b) may be attributed to the appearance of a relatively high grain size with a decrease in the number of sheets This is supported by the nanometric measurements of the sheets, which reach 39.67 nm, with a homogeneous spread that reduces the bonds between the sheets and increases the possibility of a high surface area, which may be attributed to the fact that the polarized single-end pushing groups reduce the bonds between the layers and increase the surface area and increase the grain size of the reduced graphene oxide nanosheets.

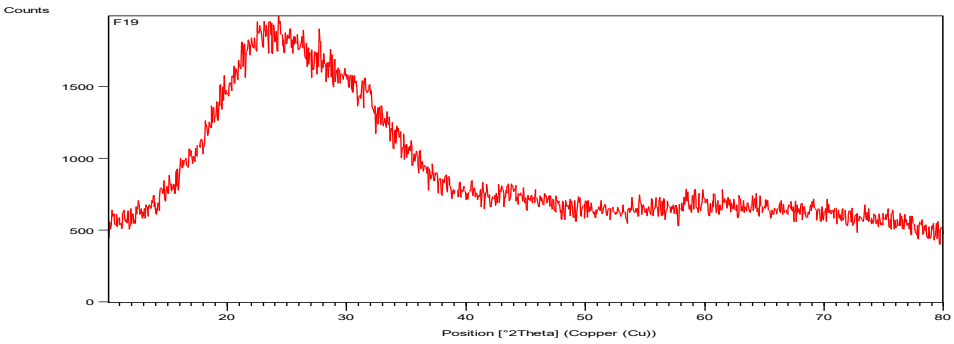

Figure 13. XRD Spectrum of Sample (T8)

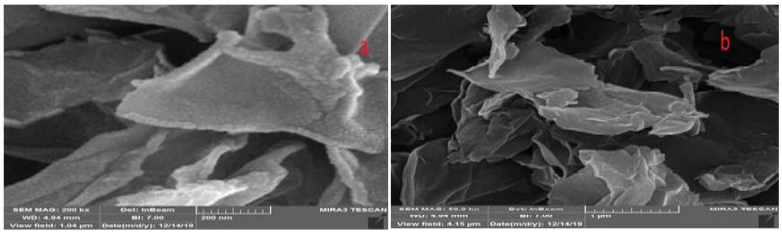

Figure 14. SEM Images of Sample (T8) (a,b)

Measuring Optical Efficiency with a (UV-Visible)

Sample T6 showed varying transmittance of (77-89)% of light intensity, as shown in the figure below:

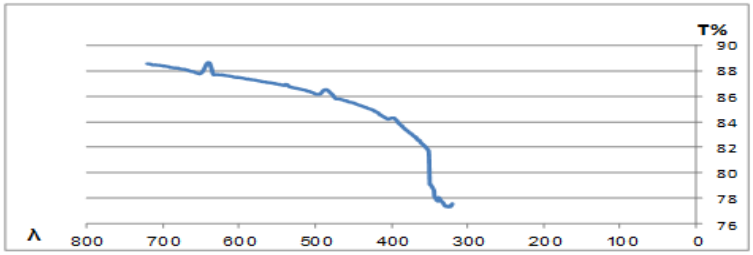

Figure 15. Light Intensity Transmittance of Sample T6

Sample T6 also showed an absorption ratio of wavelengths (0.06- 0.13)% as shown in the figure below:

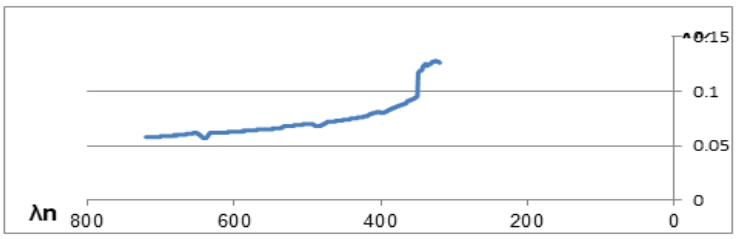

Figure 16. Wavelength Absorbance of Sample T6

As a result, it was found that the energy gap of sample T6 equals e.v (3.5) as shown in the figure below:

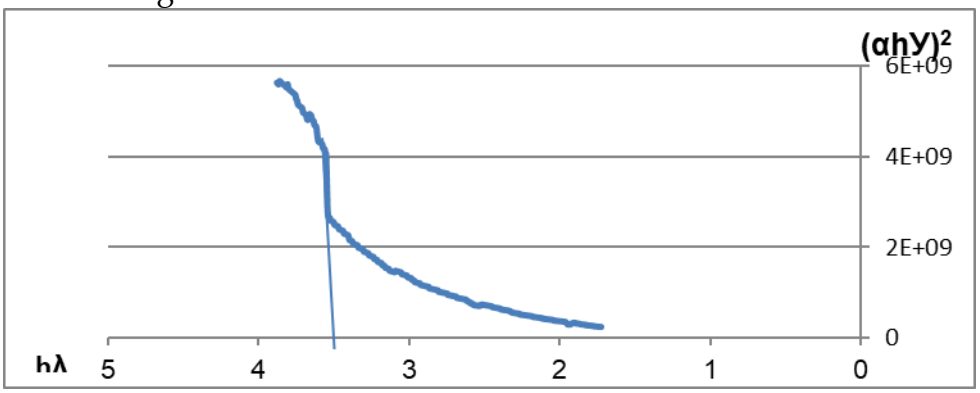

Figure 17. Energy Gap of Sample T6

Sample T7 showed varying transmittance of (49- 60)% of light intensity, as shown in the figure below:

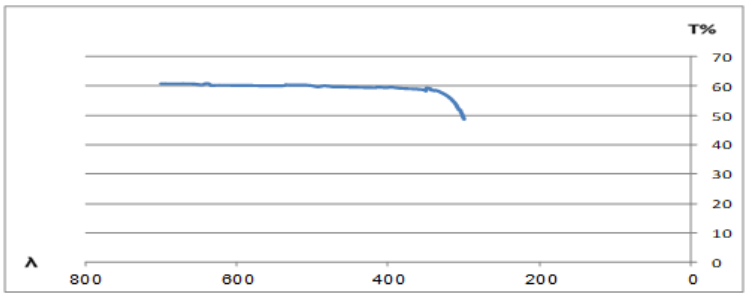

Figure 18. Light Intensity Transmittance of Sample T7

Sample T7 also showed a ratio Absorption of wavelengths (0.22- 0.32) % as shown in the figure below:

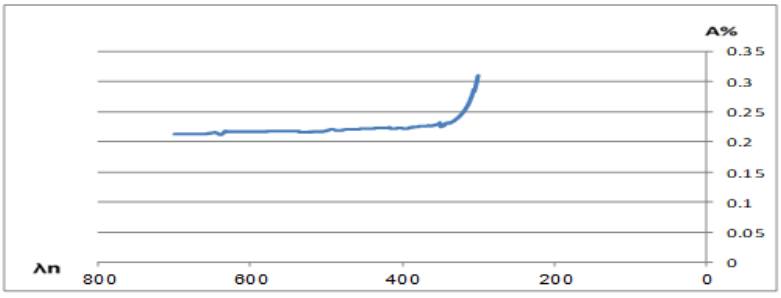

Figure 19. Wavelength Absorbance of Sample T7

As a result, it was found that the energy gap of sample T7 equals e.v (3.7) as shown in the figure below:

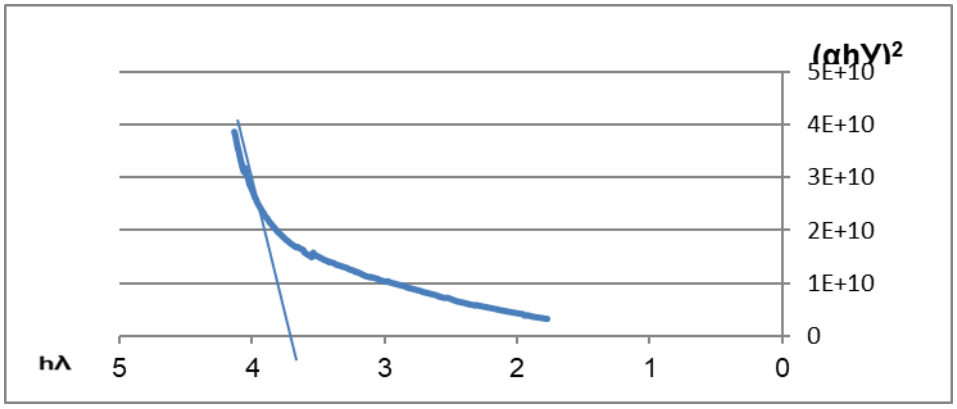

Figure 20. Energy Gap of Sample T7

Sample T8 showed a varying range of transmittance of (60-80)% of light intensity, as shown in the figure below:

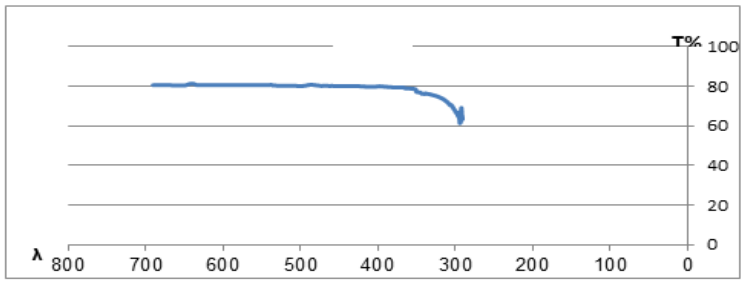

Figure 21. Light Intensity Transmittance of Sample T8

Sample T8 also showed an absorption ratio of wavelengths (0.1- 30.2)% as shown in the figure below:

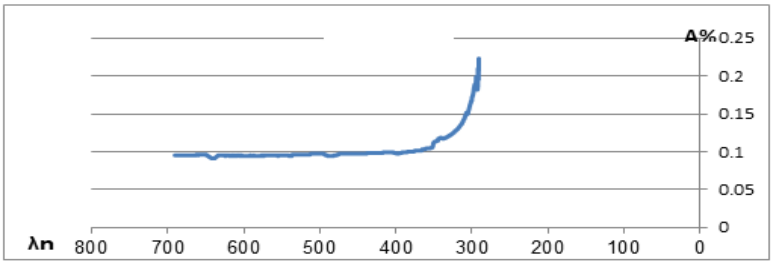

Figure 22. Wavelength Absorbance of Sample T8

As a result, it was found that the energy gap of sample T8 equals e.v (4.0) as shown in the figure below:

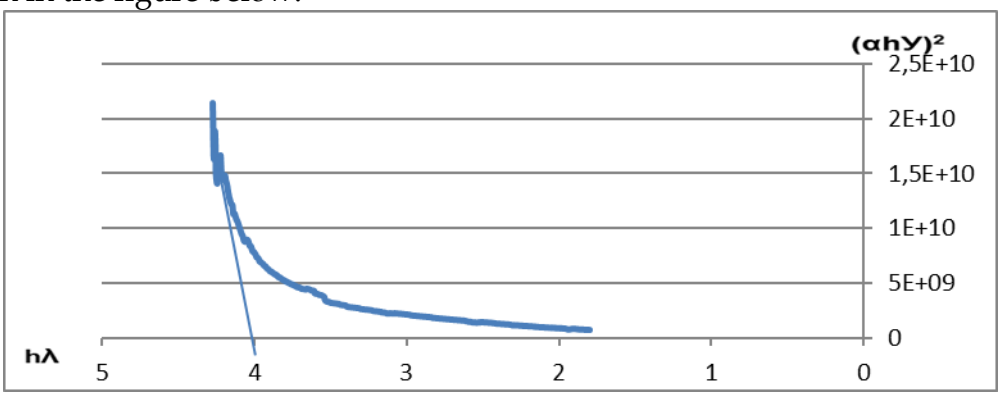

Figure 23. Energy Gap of Sample T8

Table (1) represents Values of transmittance, absorbance and energy gaps for the prepared nanocomposite membranes (T6-T8):

Table 1. Represents Values of Transmittance

Sample	% Transmittance	% Absorption	Energy gap
T_6	(89−77)	(0.13 −0.06)	(3.5 e.v)
T_7	(60−49)	(0.32 −0.22)	(3.7 e.v)
T_8	(80−60)	(0.23 −0.1)	(4.0 e.v)

From observing the values of both transmittance and absorbance in Table (1), it is clear that the transmittance was at a higher value for the complexes (T8-T6) on the graphene membranes (77- 89)% with an absorbance value that is the lowest among the prepared complexes (0.06 - 0.13)%. This may be attributed to the fact that the complexes (T8-T6) are highly polarizable, which is due to the number of heterogeneous atoms in them, where the number of Heteroatoms in the complexes as shown in the table below:

Table 2. Number of Heteroatoms in the Prepared Complexes (T6-T8)

complexes	Number of heteroatoms
T_6	9 Het.Atom
T_7	7 Het.Atom
T_8	7 Het.Atom

CONCLUSIONS AND RECOMMENDATIONS

This study successfully synthesized nanocomposites based on Schiff compounds (arylidene sulfacetamide) and reduced graphene oxide (rGO), and evaluated their optical properties and morphological structures. The synthesis was carried out through a modified Hummer method to produce graphene oxide, which was then reduced with hydrazine. The composites were made by mixing rGO with Schiff compounds synthesized from sulfacetamide and aromatic benzaldehyde.

Characterization using FTIR and NMR showed the successful formation of C=N bonds and aromatic structures. XRD and SEM results showed that the composites had nano grain sizes and well-ordered layer structures, which contributed to the increase in surface area and hydrogen bonding ability.

Optical property tests showed that the light transmittance of the composites varied between 49% and 89%, and the energy band gap ranged from 3.5 eV to 4.0 eV. Composites T6 and T8 showed the highest transmittance and lowest absorbance values, presumably due to the higher hetero atom content and high polarity of the compounds, which allowed for better optical efficiency. With the combination of stable nanocomposite structure and good optical properties, this material has the potential to be used in advanced optical, magnetic and mechanical applications in the future.

FURTHER STUDY

This research still has limitations so further research is still needed on this topic "Preparation of Arylidene Sulfacetamide Reduced Graphene Oxide Nanocomposites and Measure some of its Optical Properties".

REFERENCES

Aissa, B. (2024). Recent progress in the growth and applications of graphene as a smart material: a review: gas-phase growth, graphene material, smart applications. Frontiers in Materials and Energy, 1(003).

Ansam A. Hashim a, Rashid Jamei. (2024). Synthesis and characterization of zinc-titanium oxide nanoparticles blended methylcellulose derived from Albizia tree as an antibacterial natural polymer composite. Tikrit Journal of Pure Science. https://doi.org/10.25130/tjps.v29i1.1543

Armile, N., Chowdhury, R. R., & Desai, S. (2025). A Comprehensive Review of Quality Control and Reliability Research in Micro–Nano Technology. Technologies, 13(3), 94 https://doi.org/10.3390/technologies13030094

Bedi, P., Alanazi, A., Bose, R., & Pramanik, T. (2022). Recent Development of Synthetic Strategies Towards the Synthesis of Azomethine Analogues: A Brief Review. https://https/doi.org/10.33263/BRIAC134.350

Dhameliya, T. M., Bharadia, H. A., Shah, J. K., Sureja, D. K., & Bodiwala, K. B. (2024). Future of environment with carbon allotropes. Nirma University Journal of Pharmaceutical Sciences, https://doi.org/10.5281/

Haitham L. Al-Hayali, Abdulwahhab J. Al-Hamadany, Muntaha M. Al-Kattan (2018) Effect of Some Synthesized Pyrrolidines in Growth of L. infantum Promastigotes, Tikrit Journal of Pure Science.

Liqaa Adnan Mohammed. (2016). Effect of the addition of silver nanoparticles on the biological activity of Thio carbohydrazide derivatives,

M. G. Mukhlif, M.Sc. Thesis, College of Education for pure sciences–University of Anbar, (2017).

Manousi, N., Plastiras, O. E., Deliyanni, E. A., & Zachariadis, G. A. (2021). Green bioanalytical applications of graphene oxide for the extraction of small organic molecules. Molecules, 26(9), 2790.

N. Sangshetti, J., Altamash Shakeel Ahmad, A., A. Kalam Khan, F., & Zaheer, Z. (2015). Synthesis and biological activities of substituted benzoxazepine: a review. Mini-Reviews in Organic Chemistry, 12(4), 345-354.

Pyrzynska, K. (2023). Preconcentration and removal of Pb (II) ions from aqueous solutions using graphene-based nanomaterials. Materials, 16(3), 1078. https://doi.org/10.3390/ma16031078

Radandima, A., Arofah, S. K., Amalia, H. A. R., & Nurbaiti, U. (2021, June). nanocomposite of bioactive glass/forsterite from raw material sand and

egg shell for bone and dental implants. In Journal of Physics: Conference Series (Vol. 1918, No. 2, p. 022018).

Rady, H. S., Misbah, M. H., & El-Kemary, M. (2023). Gram-scale synthesis of highly doped chlorine graphene quantum dots: Synthesis and photoluminescence properties. Carbon, 214, 118341.

Sharma, P. K., Dorlikar, S., Rawat, P., Malik, V., Vats, N., Sharma, M., ... & Kaushik, A. K. (2021). Nanotechnology and its application: a review. Nanotechnology in cancer management, 1-33.

Singh, V., Joung, D., Zhai, L., Das, S., Khondaker, S. I., & Seal, S. (2011). Graphene based materials: past, present and future. Progress in materials science, 56(8), 1178-1271.