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Synthesis and Characterization Nano Composite Decorated with Lithocholic acid Derivatives

Yusra A. Mohammed [1], Tareq Bandar Mahboob [2], Ali Fadhel Ahmed [3] Zahraa I. Abudal Kadhum[4]

[1] *College Of Basic Education, Almustansiriyah University ,Baghdad , Iraq*
[2] *Ministry of Education The General Directorate of Education in Salah Al-Deen, , Salah Al-Deen ,Iraq*
[3] *College Of Basic Education, Almustansiriyah University ,Baghdad , Iraq*
[4]*College Of Basic Education, Almustansiriyah University ,Baghdad , Iraq*

E-mail of the corresponding author: * dr.yusra.abdulghafoo@uomustansiriyah.edu.iq

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Abstract:

Graphene oxide (GO) nanocomposites, synthesized with lithocholic acid derivatives, present a promising material with unique physicochemical properties suitable for applications in biomedicine. This study explores the preparation, characterization, and potential applications of GO-based nanocomposites functionalized with lithocholic acid derivatives. Lithocholic acid (LA) interacts favorably with GO to create stable, biocompatible, and functional nanocomposites. Through chemical modification, LA derivatives were designed to enhance dispersibility, stability, and bioactivity, addressing common challenges in GO applications such as aggregation and cytotoxicity. Comprehensive characterization techniques including Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Scanning electron microscope (SEM) confirmed successful conjugation and morphological stability of the nanocomposites.

Keywords: Graphene Oxide Nanocomposites, Lithocholicacid, morphological stability of the nanocomposites, Graphene Oxide

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تحضير وتشخيص متراكبات نانوية مزينة بمشتقات حامض الليثوكوليك

يسرى عبد الغفور محمد[1] ، طارق بندر محبوب[2]،علي فاضل احمد[3]، زهراء اسماعيل عبد الكاظم[4]

[1]العلوم، التربية الاساسية، الجامعة المستنصرية، بغداد، العراق
[2]مديرية تربية صلاح الدين، صلاح الدين، العراق
[3]العلوم، التربية الاساسية، الجامعة المستنصرية، بغداد، العراق
[4]العلوم، التربية الاساسية، الجامعة المستنصرية، بغداد، العراق

مستخلص البحث:
مركبات الكرافين أوكسيد النانوية، التي يتم تصنيعها باستخدام مشتقات حمض الليثوكوليك، تمثل مادة واعدة بخصائص فيزيائية وكيميائية فريدة تجعلها مناسبة للتطبيقات في المجال الطبي الحيوي. تستعرض هذه الدراسة خصائص وتطبيقات محتملة لمركبات نانوية تعتمد على الكرافين أوكسيد وتم تعديلها بمشتقات حمض الليثوكوليك. يتفاعل حمض الليثوكوليك (LA) بشكل إيجابي مع الكرافين أوكسيد ليُنتج مركبات نانوية مستقرة، متوافقة حيوياً، وفعالة. من خلال التعديل الكيميائي، تم تصميم مشتقات حمض الليثوكوليك لتحسين قابلية التشتت، الاستقرار، والنشاط البيولوجي، مما يعالج التحديات الشائعة في تطبيقات الكرافين أوكسيد مثل التكتل والسُمية الخلوية. أكدت تقنيات التشخيص، بما في ذلك مطياف الأشعة تحت الحمراء (FTIR)، وحيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح (SEM)، نجاح الارتباط وثبات الشكل للمركبات النانوية

الكلمات المفتاحية: مركبات أوكسيد الجرافين النانوية، حمض الليثوكوليك، الثبات المورفولوجي للمركبات النانوية، أكسيد الجرافين نانو ثنائي أقطاب الجرافين،

1. Introduction

Nanocomposites can be described as a group of materials produced by mixing a percentage of one or more substances with the base material. The materials are well integrated with each other to obtain a homogeneous composite in which the particles of the materials are fully distributed. Nanocomposites are considered one of the most important advanced materials, opening future horizons for solving complex problems .Lithocholic acid (LCA) is a bile acid and an example of an organic carboxylic acid compound. It belongs to the family of secondary bile acids, which are formed in the intestine by the feat of bacteria on primary bile acids . *(Ridlon, J. M., Kang, D. J., & Hylemon, P. B. 2006)* such as cholic acid. The main structural features of lithocholic acid is Carboxyl group (-COOH), which defines it as a carboxylic acid. This functional group can donate a proton, giving the molecule its acidic properties and Steroid nucleus *(Lajczak-McGinley, N. K., Porru, E., Fallon, et al. 2020).*, Lithocholic acid has a steroidal structure with four fused rings (three cyclohexane rings and one cyclopentane ring), which is typical for bile acids and Hydroxy group, in position 3 of its steroidal structure, making it a relatively hydrophobic molecule.*(Sun, L., Li, F., Tan, W., Zhao, W., Li, Y., Zhu, X., ... & Wang, L. 2023)*

The benefit of the composite is to obtain new distinctive properties and advanced characteristics. Since the composites have the strength of th Compound's e additive and the flexibility of the compounds, they have attracted many manufacturers.*(Potts, J. R., Dreyer, D. R., Bielawski, C. W., & Ruoff, R. S. 2011)* Therefore, Compounds composites have been widely used in recent times. compounds composites are the addition of prepared materials that interact with graphene oxide to the compounds used in the study. These additives can improve the electrical conductivity properties, mechanical properties, thermal conductivity, flexibility, hardness, strength, etc., depending on the nanomaterials added to the compounds.*(Daradmare, S., Raj, S., Bhattacharyya, A. R., & Parida, S. 2018)*

2. Experimental

Graphene oxide: is prepared by modified Hammer method. Graphite 1gm, sodium nitrate(1.5 gm) and of sulfuric acid (46 ml) were mixed and strongly stirred at 0˚C for 15 minutes in a 500 ml reaction flask immersed in ice bath. Then potassium permanganate 6gm was added slowly to the above solution and cooled for 30minutes. After this, the suspended solution was stirred continuously for 2 hour at 35˚C, and water (46 ml) was added slowly to the suspension for 10 minutes and raised the temperature to 98 ˚C. The solution was left with stirring for 20 minutes. Subsequently, the suspension was diluted by warm water 140ml and stirring for 10minutes. After that, The solution was maintained at room temperature, treated with H2O215 ml (30%) to reduce residual permanganate to soluble manganese ions. Finally, the resulting suspension was filtered by centrifugation, washed with 10% HCL and distilled water.*(Ossonon, B. D., & Bélanger, D. 2017).*

Thiocarbohydrazide(TCH) : Add 5 mL of disulfide to a round flask with a capacity of 100 mL in an ice bath, then add to it 20 mL of hydrazine (80%) dropwise over(10 min) while stirring continuously. Heat the reaction mixture for (30 min). A yellowish-white precipitate form. Collect the precipitate by filtration and wash with ethanol until the precipitate turns white. Collect the final product and dissolve it in distilled water. The yield is 80%, with a melting point of 170°C.*(Ashour, M., Kandil, F., & Alazrak, A. 2024). (Jiříčková, A., Jankovský, O., Sofer, Z., & Sedmidubský, D. 2022).*

(3R,8R,9S,10S,13R,14S,17R)-17-((R)-4-(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)butan-2-yl)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-3-ol (TL) is prepared by heated TCH with Lithocholic acid in a flask*(Šarenac, T. M., & Mikov, M. 2018).(Smith, M.*

B. 2020). until melted *,(Thomas, W. P., & Pronin, S. V. 2021).* (time 10 min, m.p 145-147 , temperature 188 ^{o}c , color Light- Brown , Rf 0.65) . Nano composites (GO- TL) is prepared by put 0.1 g graphene oxide in 15 ml of dioxane. *(Chen, H., Du, W., Liu, J., Qu, L., & Li, C. 2019).(Wan, S., & Cheng, Q. 2017).* then the solution was placed in an ultrasonic bath for 2 hours .0.1 g from (TL) add then it was refluxed for 1 hour with continuous stirring, the precipitate was filtered and washed with ionic water (3 times x 10 ml for each wash) and dried.

3. Results and Discussion

Graphene oxide was examined using an X-ray diffraction. Fig (1) shows the X-ray diffraction pattern of graphene oxide. It was found that graphene oxide exhibits strong diffraction at 11.84° corresponding to a d-spacing of 0.75 nm. which is higher than that of graphite (0.33 nm) Fig. (3-2) indicates introduce functional groups (carboxylic acid, carbonyl, hydroxyl and epoxy) on the basal and edges planes of GO sheets *(Vagdevi, K., Devi, V. R., & Rao, K. V. 2017).(Zhou, J., Wu, D., & Guo, D. 2010).*

The functional groups of GO have been characterized with FT-IR. Fig. (2) shows the FT-IR spectrum of GO,. The stretching vibration of alcoholic and carboxylic OH appeared at 3433 cm-1 with broad and strong band.*(Latif, I. A., & Merza, S. H. 2018).* Atomic Force Microscope (AFM) was also used to examine the morphology and the thickness of the GO sheet.Fig(3). The maximum height of GO was 5.52 nm with smooth sheets-shaped in appearance as illustrated in the 3D-AFM image in Fig.(3-a). The length and thickness of the chose sheet in Fig.(3-b,c) was calculated depends on the section area analyses. The morphology of the GO (Fig.4) was studied by scanning electron microscopy (SEM). It was observed that particle size reduction on GO. As observed, the splitting of graphite stacks into layers is considerably higher in the GO . The layers within the layer are further disassembled and more crumpled structure is observed. This is the consequence of surface area (due to particle size reduction) that allow efficient oxidation during the process.

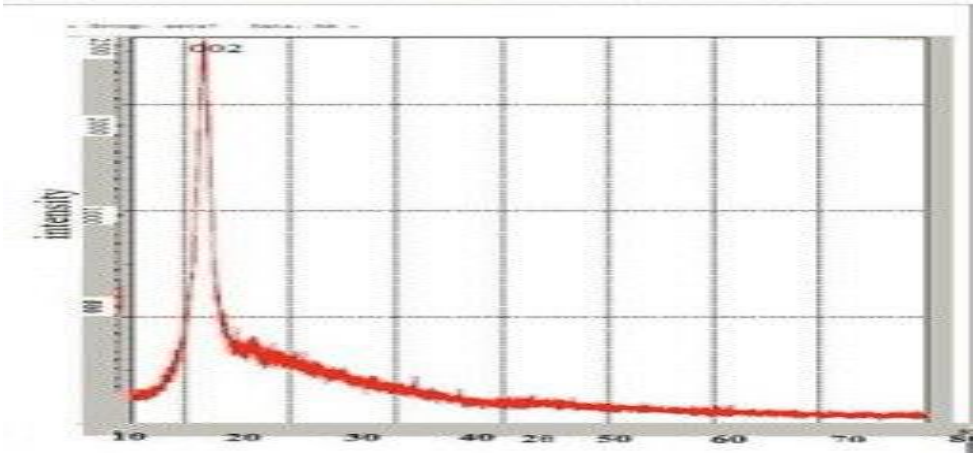


Fig (1) shows the (X-ray) diffraction pattern of graphene oxide

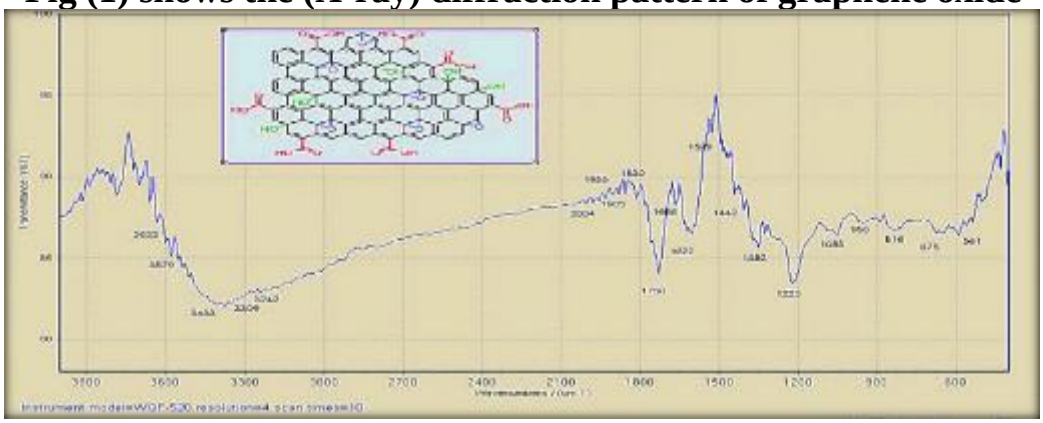
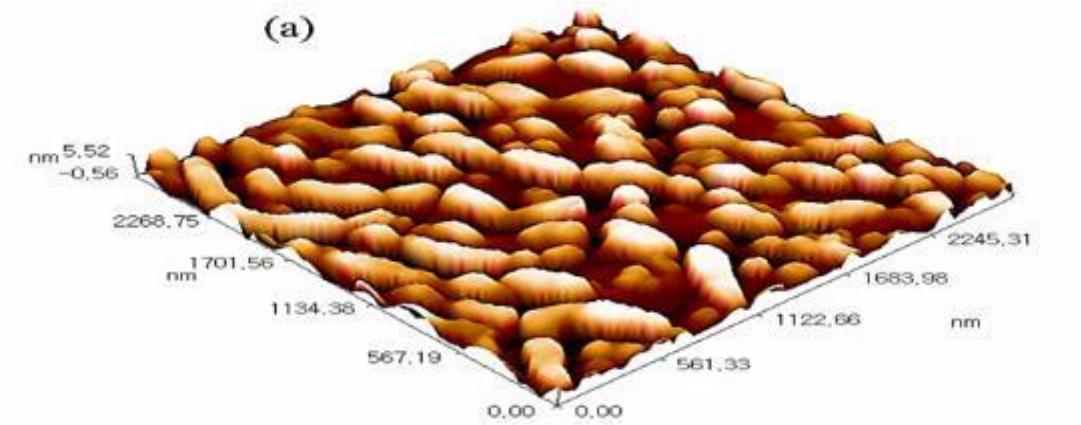


Fig. (2) shows the (FT-IR) spectrum of GO



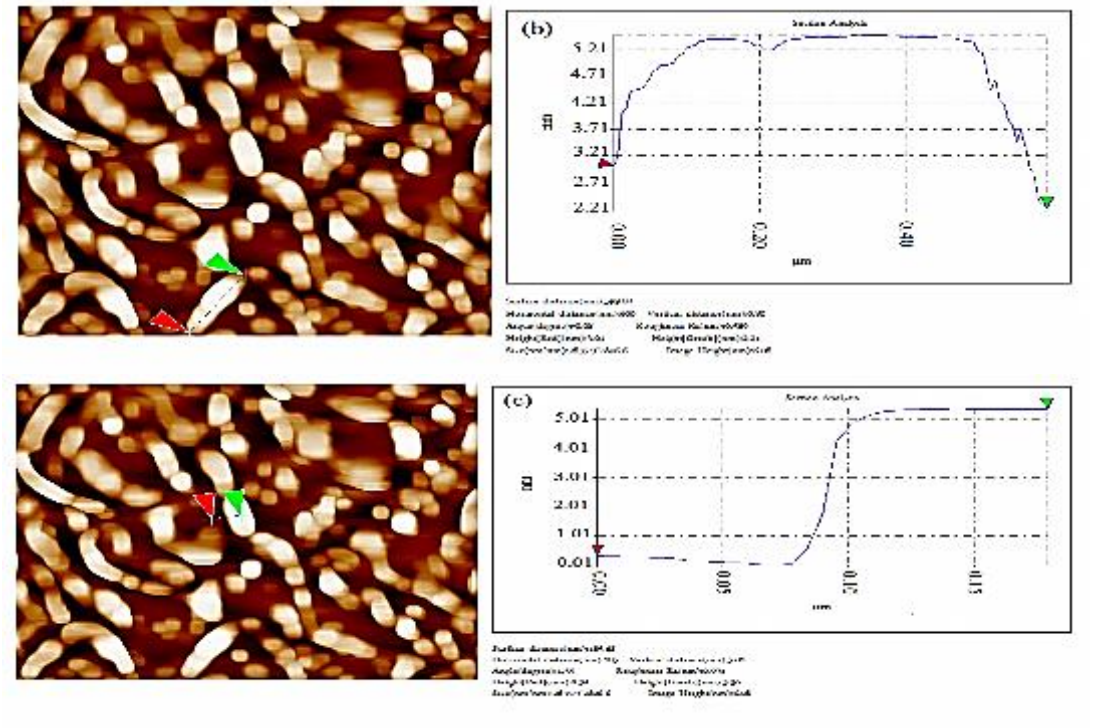


Fig. (3) AFM results of GO: (a, b ,c)

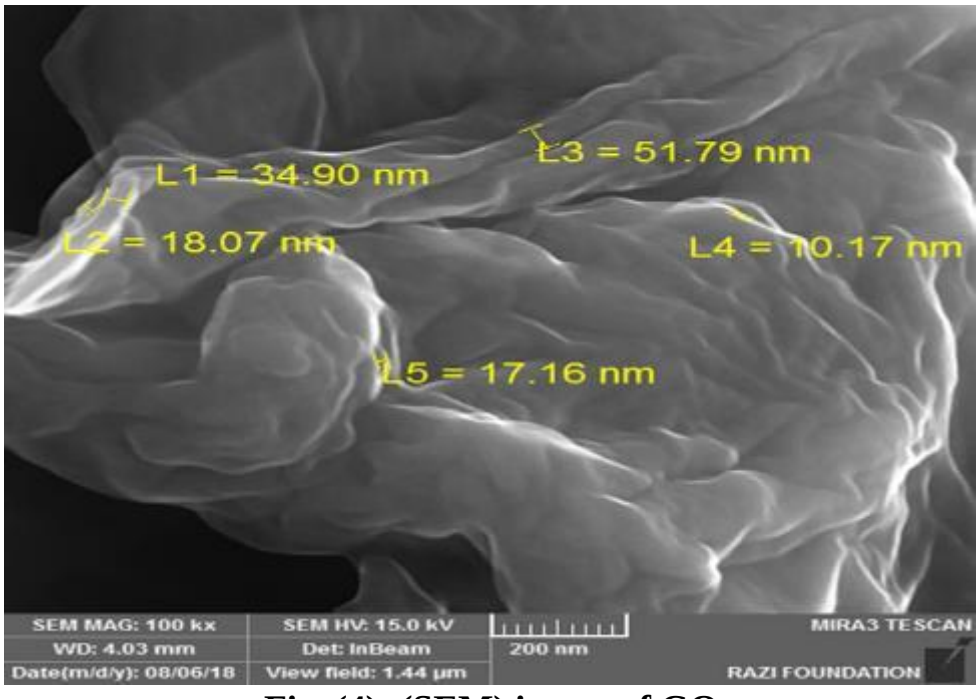


Fig. (4): (SEM) image of GO

Fig. (5) and Fig. (6) shows FT-IR spectrumof prepared TCH, (TL), The peaks at 3304, 3269, 3165 cm-1corsspondes to N-H and NH2 stretching vibrations respectively. The NH2 bending and wagging vibrations contributed to the two peaks at 1637 and 1139 cm- 1 respectively .The characteristic peaks 1531 and1490 cm-1 assigns to the coupled modes N-

H wagging and C-N stretching vibrations.*(Barot, K. P., Manna, K. S., & Ghate, M. D. 2017).* and stretching vibration of ν(OH) in (3373) cm-1, stretching vibration of ν(NH2) in (3275,3182) cm-1 The peak in 1662 assigns to the ν(C=N), The C=S stretching contributes to peak at 1172 cm-1, also the peak at 2927,2862 cm-1 to ν(CH2), and the peak in at 1033 cm-1 assigns to the ν(C-O) and beding vibration of δ(CH3) in (1367) cm-1 for (TL) .(*Pospieszny, T. 2015).* The 1HNMR spectrum(in DMSO-d6 as a solvent) of (TL) showed in Fig (7), a singlet signal at δ 5.50 ppm for NH2, , a singlet signal at δ (6.55) ppm for NH, a singlet signal at δ (7.22) ppm for OH,the singnal appeared at δ (0.49-2.27) ppm for aliphatic saturated protons. *(Moretti, A., Li, Q., Chmielowski, R.,et al 2018).(K umar, S., Wani, M. Y., Arranja, C. T., Castro, R. A., Paixão, J. A., & Sobral, A. J. 2018).*

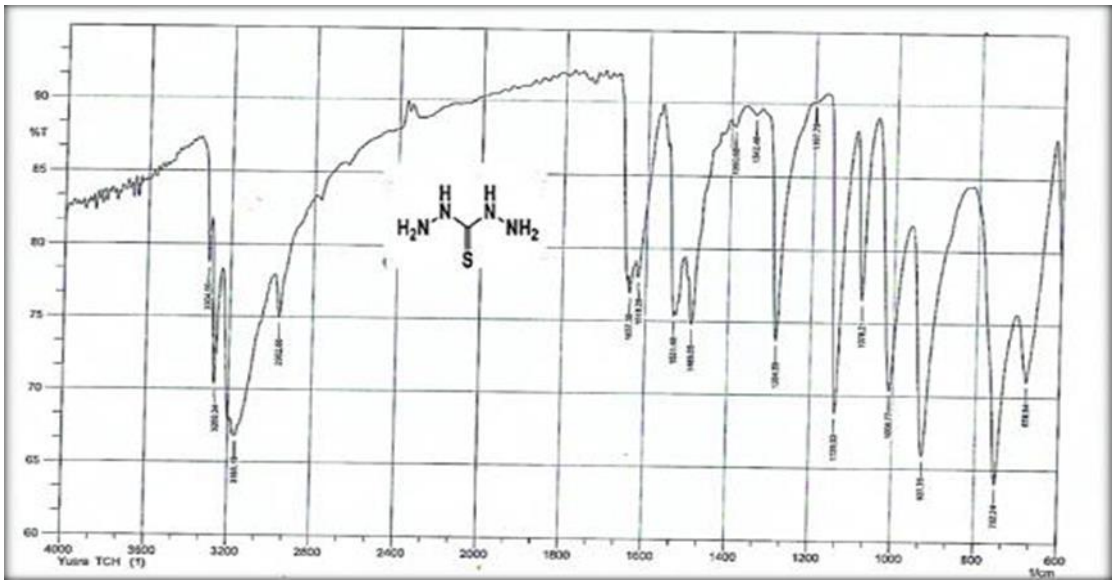


Fig. (5) shows FT-IR spectrum for prepared TCH

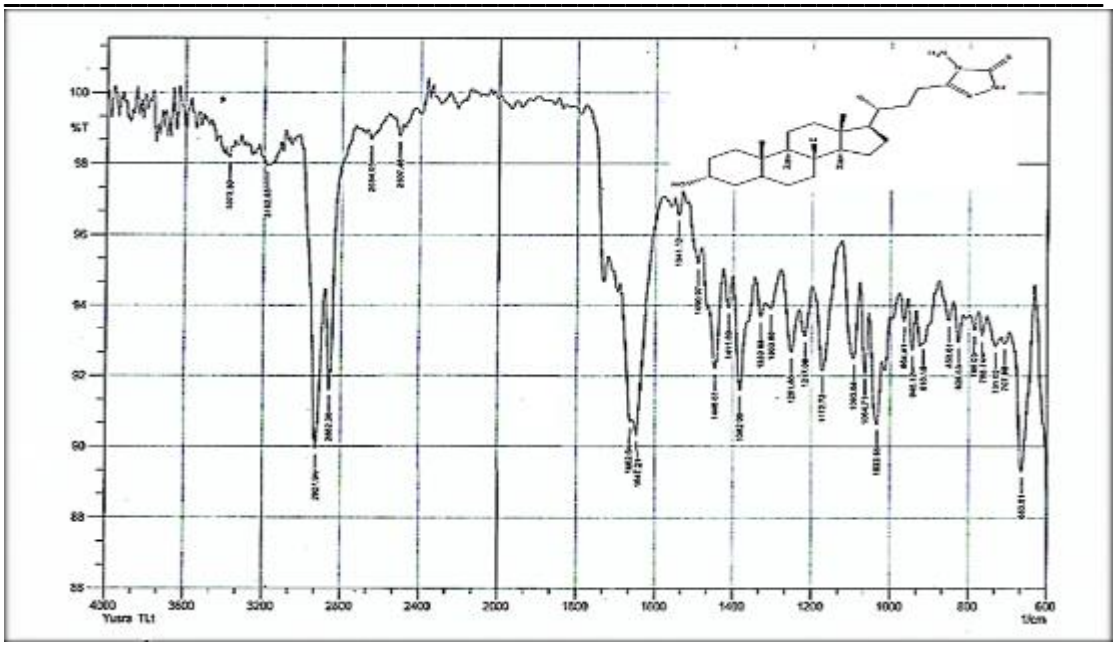


Fig. (6) shows (FT-IR spectrum) for prepared (TL)

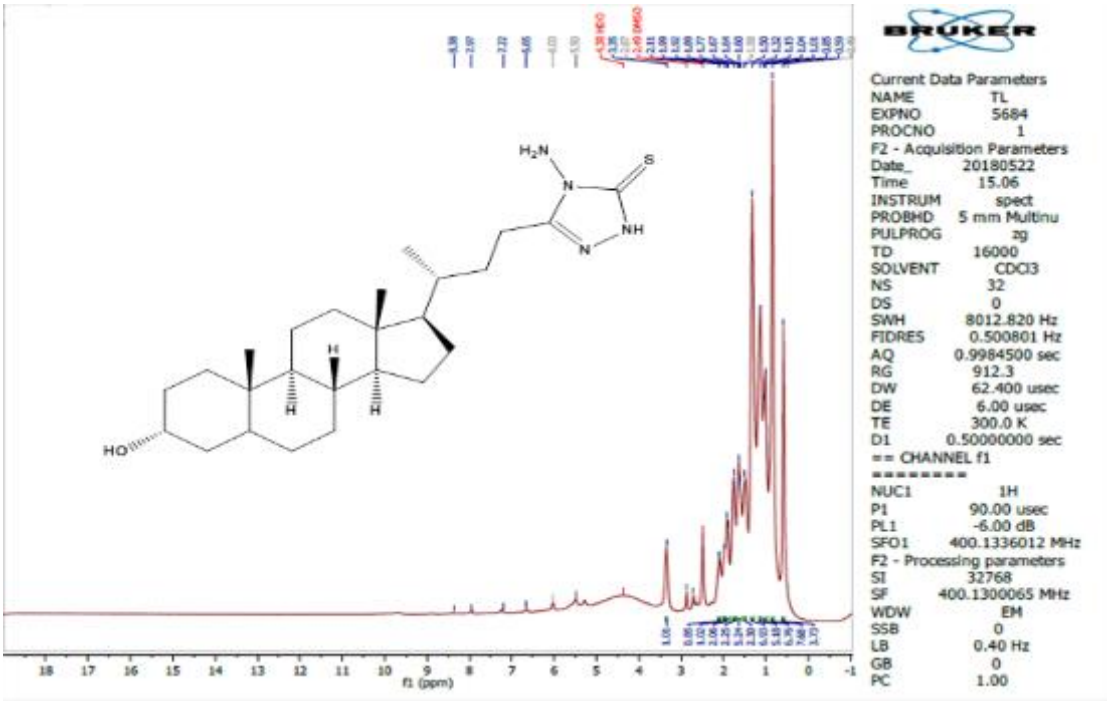
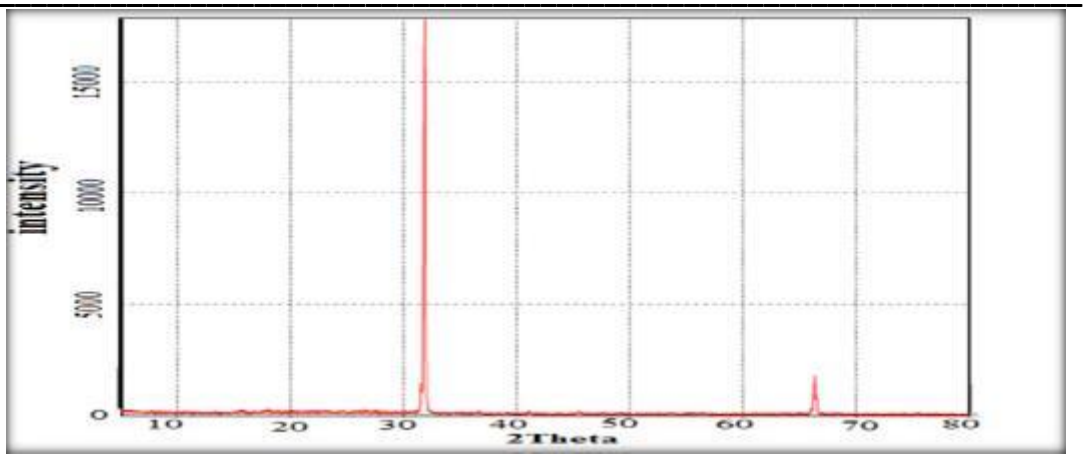


Fig (7)^{1}HNMR - spectrum of prepared TL

Figure (8) shows the (X-ray) diffraction pattern of (GO- TL), (2θ 31.54o, 31.85o ,66.30o) the d-spacing (0.28,0.28,0.14 nm . Fig. (9) shows the (FT-IR) spectrum of (GO- TL) illustrated the presence of a large peak at (3440,3467,3437) cm−1, Atomic Force Microscope (AFM) for (GO- TL) sheet.in Fig (10). The morphology of the (GO –TL).Fig.(11) was examined by scanning electron microscopy (SEM).



(GO- TL) Fig. (8). XRD patterns of

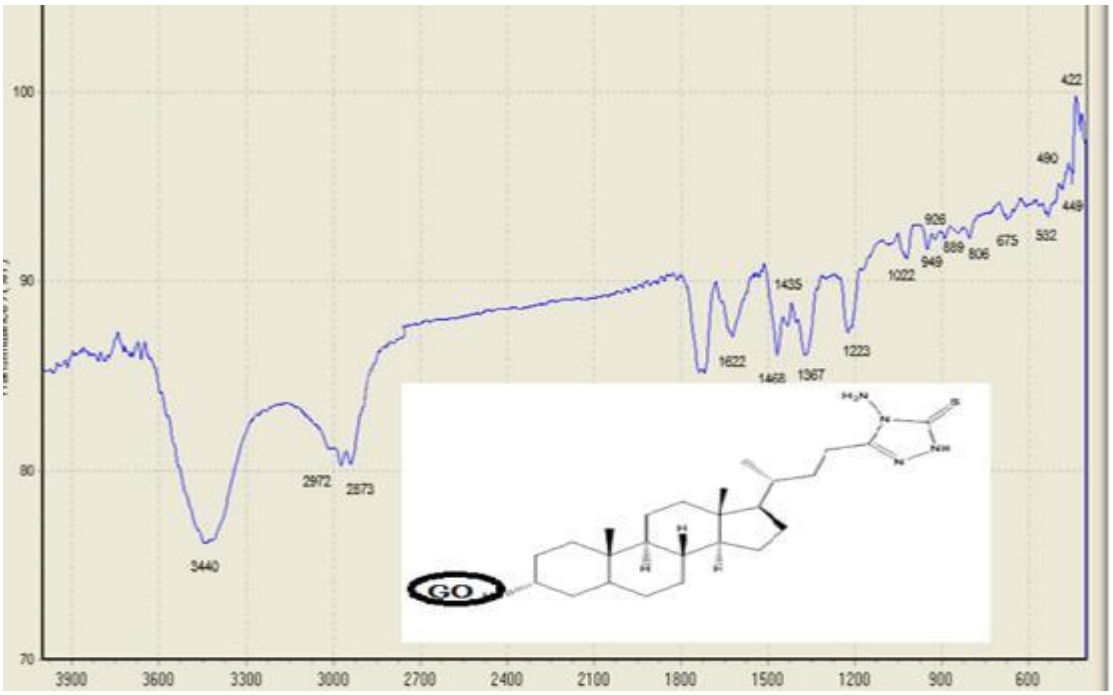


Fig. (9) (FT-IR) spectrum of (GO- TL)

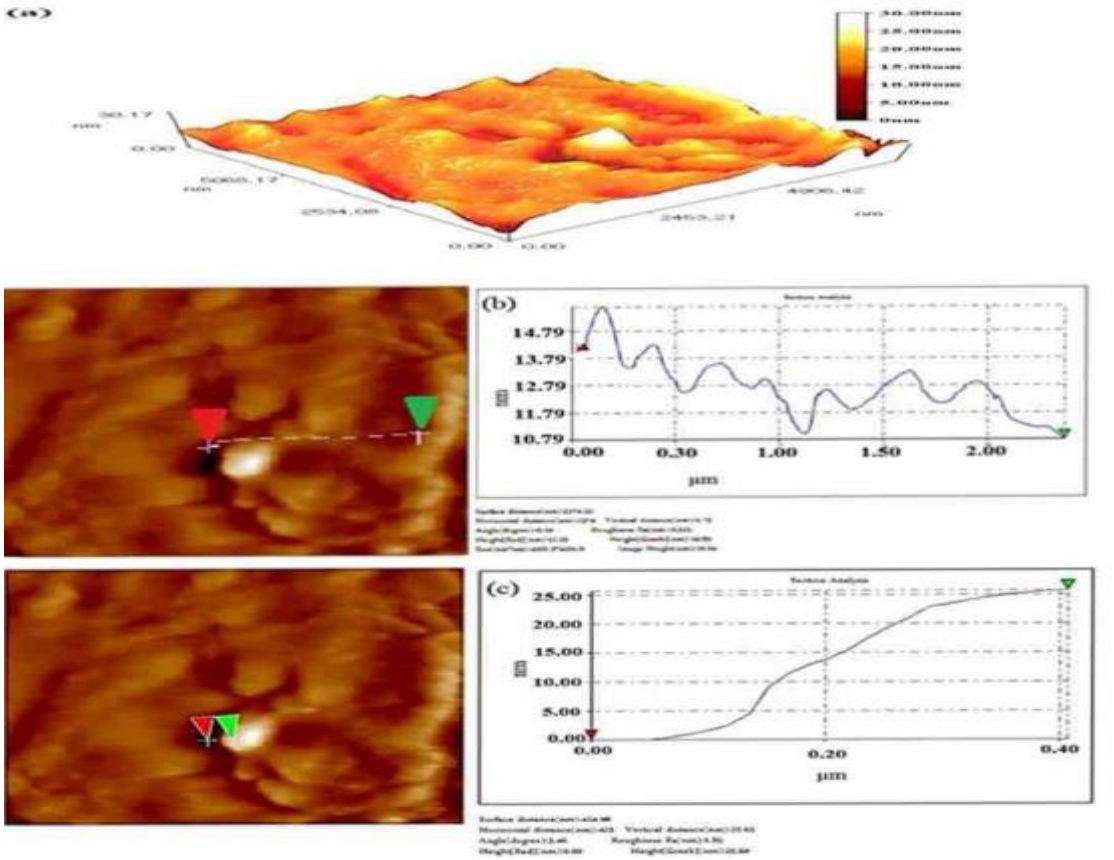


Fig. (10) AFM results of [GO-TL (a,b,c)]

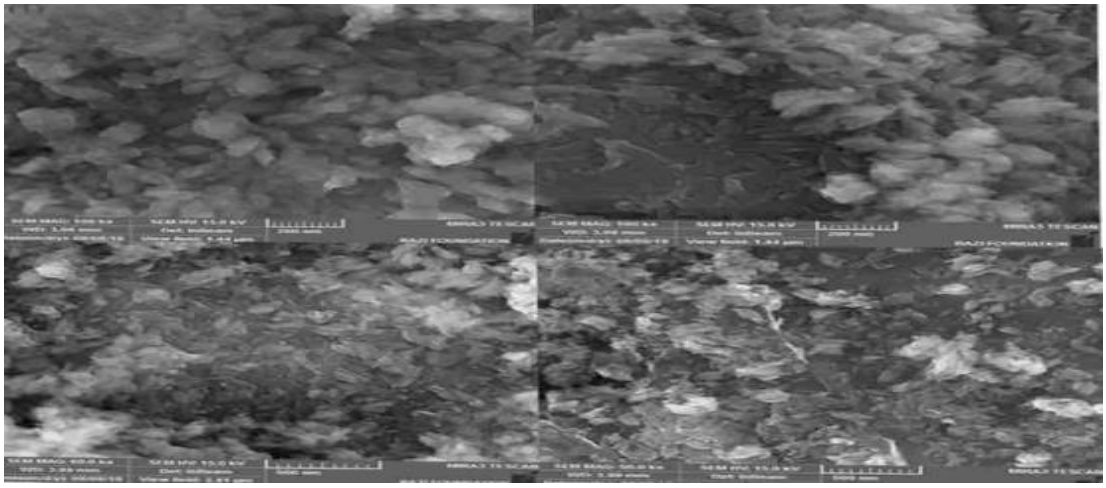


Fig. (11) (SEM) image of compound GO-TL

Conclusion:

The study concludes that graphene oxide (GO) nanocomposites functionalized with lithocholic acid derivatives represent a valuable material with versatile properties suited to various applications, including biomedicine, catalysis, and environmental remediation. The incorporation of lithocholic acid, a bile acid known for its biocompatibility and amphiphilic structure, into GO helps to enhance the nanocomposites' dispersibility, stability, and bioactivity. Key characterization techniques, including FTIR, XRD, and SEM, confirmed successful synthesis and functionalization. These GO-based nanocomposites demonstrate improved structural integrity and functional properties, addressing common challenges in GO applications such as aggregation and cytotoxicity, making them promising for advanced material applications.

Conflicts of Interest: None

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