Iraqi Journal of Physics, 2025 *Vol. 23, No.1, PP. 78-84*
https://doi.org/10.30723/ijp.v23i1.1298
P-ISSN: 2070-4003
E-ISSN: 2664-5548

Characterization of Graphene/Epoxy Nanocomposites

Malik Rashid Khasheet[1a*] and Estabraq Talib Abdullah[1b]
[1]*Department of Physics, College of Science, University of Baghdad, Baghdad, Iraq*
[a*]Corresponding author: malek.khshit2304@sc.uobaghdad.edu.iq

Dr. Estabraq Talib Abdullah is a journal editor, but he did not participate in the peer review process other than as an author. The authors declare no other conflict of interest.

Abstract

Graphene nanoplatelets have received extensive attention from the scientific community in the 21st century because of their two-dimensional planar structure, high surface area, functionalization abilities, and remarkable electrical, mechanical, and thermal properties. In this work, the fabrications of graphene platelets/epoxy nanocomposites, with weight ratios of (0.002, 0.004, 0.006, and 0.008), were synthesized by the combined action of centrifuge and sonication techniques to reduce the aggregation and agglomeration of the graphene. Fourier-transform infrared spectroscopy (FTIR) verifies the prepared samples by the presence of main, distinguishing peaks associated with vibrational groups. Scanning electron microscopic (SEM) shows a good dispersion of the graphene in the epoxy, where it is homogeneously dispersed by exhibiting less agglomeration and aggregation. Atomic force microscopy (AFM) shows the average Ra value of the epoxy resin was 29 nm. In comparison, the graphene-based composites were less than 5 nm, significantly decreasing the surface roughness by adding a small amount of nanofillers. Surface roughness shows that epoxy is rougher than nanocomposites.

Article Info.

Keywords:
Graphene, Epoxy, Ultrasonication, Contact Angle, AFM.

Article history:
Received: May,11,2024
Revised: Jul. 18, 2024
Accepted: Jul. 27, 2024
Published Mar 01,2025

1. Introduction

Epoxy polymer, compared to other fibres, has excellent mechanical and thermal properties, wear and chemical resistance, and low cost [1]. These properties make the epoxy polymer attractive to apply in many applications, such as coating and adhesives and can be used as structural materials and matrix composites [2]. Epoxy has a high degree of crosslinking because it belongs to thermoset polymer, making it high in rigidity and strength. However, this crosslinking structure makes the polymer brittle with a high ability to crack, which eventually limits its applications [3, 4]. Due to this limitation, many scientists and researchers are concerned about improving the toughness of epoxy by adding filler to the polymer.

Single graphite layers arranged in a hexagonal lattice of carbon atoms joined in the sp2 configuration are known as graphene [5]. It is known as the thinnest of materials, and it has many interesting applications [6, 7]. It was first known when Geim and Novoselov won the Nobel Prize in 2010 for preparing a single graphene sheet by peeling off a sheet of graphite using scotch tape [8]. Excellent graphene properties include superior mechanical properties, including ultimate strength (139GPa), Young modulus (1TPa), intrinsic strength (130MPa), and higher electrical and thermal conductivities than copper materials [8]. Many researchers have proved that even a small amount of graphene can enhance the electrical, mechanical, and optical properties of matrix materials due to its high aspect ratio [9-11].

The extraordinary improvement of the polymer characteristics at low filler fractions has drawn the attention of most academia and industry in recent years to polymer/graphene-based nanocomposite [12-14]. Epoxy resins typically consist of epoxy groups at both terminal extremities and hydroxyl groups at the midpoint of the molecule. Aromatic rings are essential in epoxy due to their exceptional resistance to heat and chemicals. In epoxy resins, there is also some branching at the end groups, which is primarily dependent on the commercially available resin and nanofiller. Due to its indispensable function in the field of polymer composites, graphene has been regarded as an indispensable reinforcing material among all nanomaterials, including CNTs, MXene, and nano-silica. For instance, it enhances the bonding of filaments with the epoxy matrix, thereby improving its function as reinforcing filler. Additionally, the polymer beneath the graphene sheets is safeguarded from atomic oxygen erosion. Various factors, including the filler content fraction, aspect ratio of the graphene, and uniform dispersion of the graphene, influence the ultimate properties of graphene/epoxy [15, 16].

This work involved the preparation of an epoxy composite with different quantities of graphene (0.002, 0.004, 0.006, and 0.008) utilizing ultrasonication and stirring procedures. The focus of the study was to analyze the structural and morphological properties of the resulting nanocomposite.

2. Experimental Part

2.1. Materials

Graphene sheets with a 608nm thickness and a 15 µm average particle diameter were purchased from Skyspring Nanomaterials Inc. (USA). Epoxy resin (6105) was obtained from DOW Chemicals (USA), and methyl-hexahydrophthalic anhydride (MHHPA) used as a hardener was provided by Zhejiang Alpharm Chemical Technology Co., Ltd. (China).

2.2. Samples Preparation

Epoxy-based nanocomposites reinforced with different weight ratios of graphene (0.002, 0.004, 0.006, and 0.008) were prepared using the following procedure. The desired concentrations of graphene were poured into a liquid epoxy that was ultrasonicated for 1 hour. The solution was further processed with mechanical stirring and sonicating for 3 hours to separate the aggregation of nanofillers and achieve good dispersion. Finally, MHHP, the hardener, was added to the mixture at a ratio of 1:3 and ultrasonicated for another 15 minutes. The resulting graphene-epoxy mixture samples were cast into (0.1×0.5) discs and cured at 70°C for 1 hour. The samples were detonated as variables, as in Table 1.

Table 1: Symbols of the studied samples.

Materials wt%	Symbols
Graphene	A1
Epoxy	A2
0. 2 nanocomposites	A3
0. 4 nanocomposites	A4
0. 6 nanocomposites	A5
0. 8 nanocomposites	A6

2.3. Characterization

The structural properties of the samples were investigated using a Fourier-Transformer Infrared (FTIR) spectroscope (type- Shimadzu 8400) in the frequency range 4000-400cm^{-1} by pelletizing a homogenized powder of the synthesized samples and KBr. The surface morphology of the prepared samples was studied using a Scanning Electron Microscope (SEM) (FEI-Company Netherlands inspect S50). Atomic

Force Microscopic (AFM) tests were done to analyse the surface morphology. A contact angle test of the pure and nanocomposite samples was conducted to understand the surface modification on adding graphene based on the ASTM D7334.

3. Results and Discussion

3.1. SEM Analysis

SEM analysis was used to examine the sample and identify the distribution of graphene inside the polymer matrix. The SEM images are shown in Fig.1. Many studies showed that seeking graphene sheet in microstructure is a challenge during dispersion in polymer matrix [17-20]. The images show that graphene had a non-crystalline distribution within the epoxy, resulting in a smooth and homogeneous epoxy surface without any visible cracks. The surface appeared sleek, with a tendency toward cohesion due to the homogeneity between the epoxy and graphene. Furthermore, the nanoparticles displayed a uniform distribution throughout the sample, homogeneously dispersed by exhibiting lesser agglomeration and aggregation, and there was no evidence of graphene clustering on the epoxy surface. This uniformity was consistent with an increase in the graphene-to-epoxy ratio [21, 22]. Moreover, the actual size distribution of the different graphene can easily be distinguished by their appearance, as seen in Fig. 1.

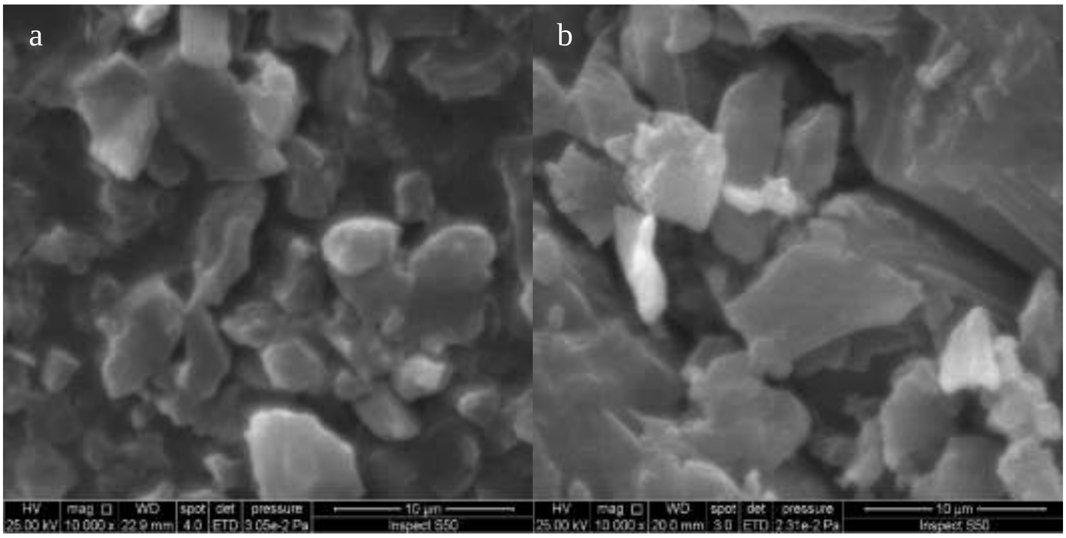

Figure 1: SEM images of (a) A4 and (b) A6 nanocomposites.

3.2. FTIR Analysis

FTIR spectroscopy examines multi-component functional groups and provides helpful information about the mechanism of interaction and the content of the material phase in a variety of bond types found in all samples. Fig.2 exhibits FTIR spectra of pure graphene, pure epoxy and epoxy-graphene composites with different graphene concentrations. The spectra revealed distinct bands of bending and stretching vibrations associated with the functional groups generated in the synthesized films.

For epoxy, the peaks at 3421.72 cm^{-1} correspond to O-H stretching. The peak at 3050 cm^{-1} indicates the $-CH-O-CH_2-$ stretching of the epoxy ring, and at 2966 and 2925 cm^{-1} of the asymmetric stretching of the $-CH_3-$ and $-CH_2-$ bonds are identified. The symmetric stretching of these two groups has a visible peak at 2958.80 cm^{-1}, corresponding to C-H (alkanes) stretching. Between 2000 and 1500 cm^{-1}, there are the overtones of the C-H bonds of the aromatic ring and the signals of the stretching of C=C-H always belong to the benzene ring. 1247.94 cm^{-1} peak corresponds to C-O stretching, the peaks at 1510.28 cm^{-1}corresponds to the C=C stretching, the peak at 956.69 cm^{-1} is for the bond $-CH-O-CH_2-$ which belongs to the epoxy ring [17, 18].

For the epoxy-graphene composite, the peaks at 1558.48 cm^{-1} correspond to C=C stretching. It indicates the presence of graphene in the sample, where the C=C bond is an essential part of the graphene structure and peaks at 3425.58 cm^{-1} corresponding to O-H stretching. This indicates the ability of the mixture to achieve good surface adhesion. OH groups have the ability to interact effectively with other surfaces, leading to improved adhesion and good corrosion resistance [23, 24].

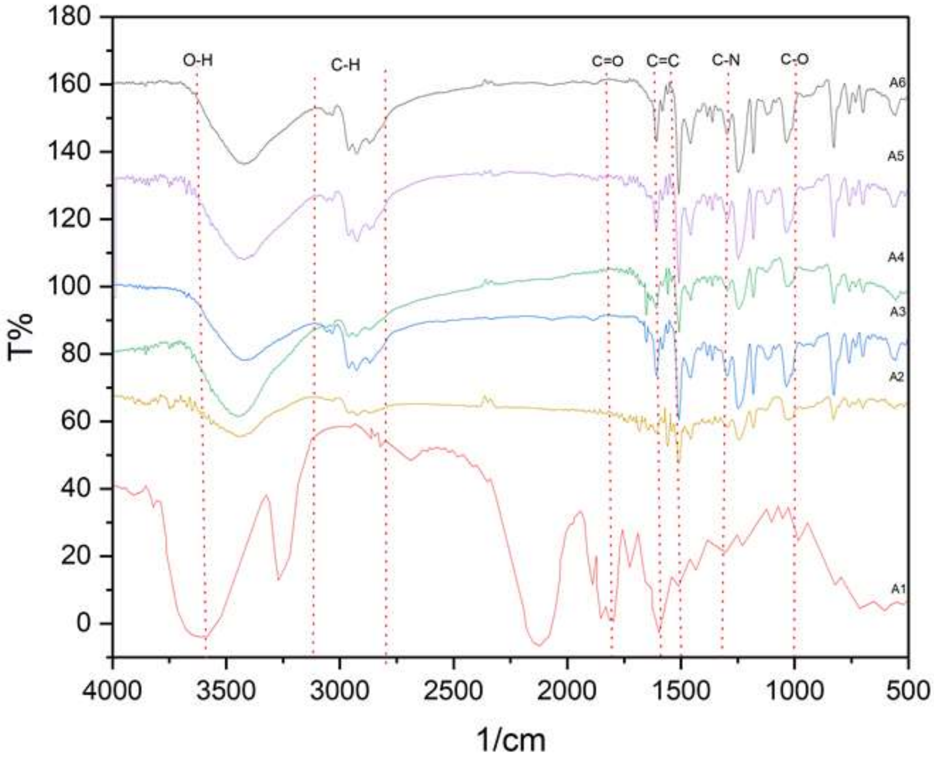

Figure 2: FTIR spectra of pure graphene, epoxy, and the epoxy-graphene nanocomposites of different graphene concentrations.

3.3. AFM Analysis and Surface Roughness

Fig. 3 shows the AFM images of pure epoxy and epoxy-graphene nanocomposite samples. The surface of epoxy resin seems to be rougher than the other nanocomposite samples (see Fig.3a). The averaged roughness (S_a) value of the epoxy resin was 244.2 nm. While the values of epoxy-graphene nanocomposites were less. The results showed that adding a small amount of graphene significantly increased the surface roughness [25].

Fig. 4 shows the contact angle of the epoxy and nanocomposite samples. The epoxy contact angle was about 67 degrees, confirming the hydrophilic nature of epoxy. The nanocomposites, with different graphene concentrations, displayed a different trend, including a reduction in the contact angle except for the nanocomposite with a graphene concentration of 0.002 wt%, as listed in Table 2. This shows that mixing with graphene slightly decreased the hydrophobicity of the nanocomposites surface. This was assured by the surface roughness, Sa, as shown in Fig. 3 [26, 27].

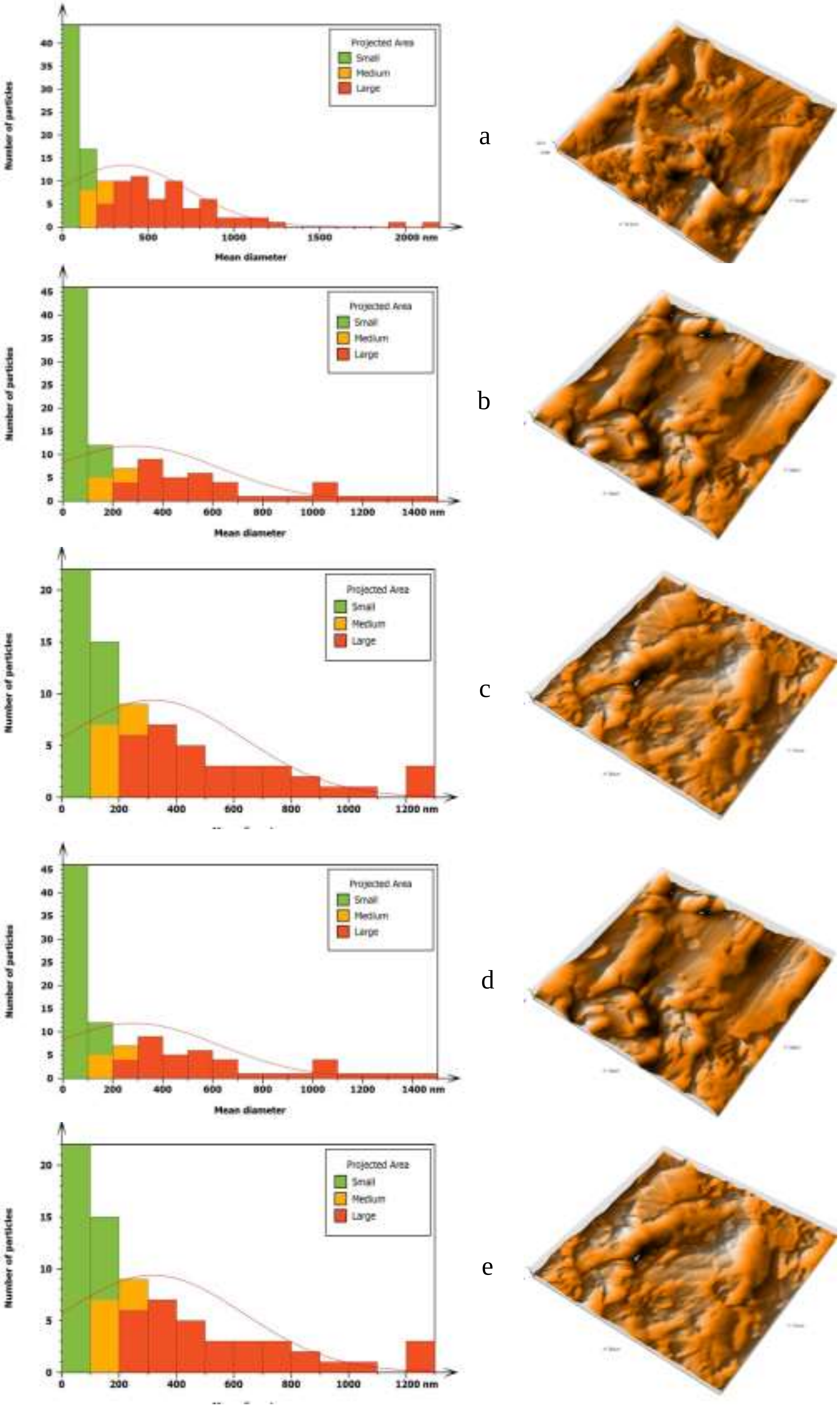

Figure 3: AFM images of (a) A2, (b)A3, (c) A4, (d) A5, and (e)A6.

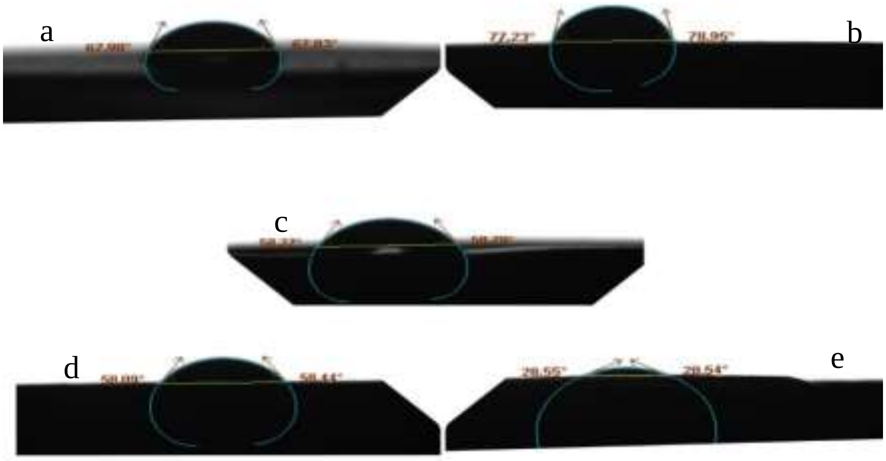

Figure 4: Contact angle of (a) A2, (b) A3, (c) A4, (d) A5, and (e)A6.

Table 2: Surface roughness and contact angle of epoxy and epoxy-graphene nanocomposites.

Symbols	Contact angle	Sa (nm)
A2	67.98	244.2
A3	78.95	491
A4	58	370.2
A5	28	253.3
A6	58.79	33.0

4. Conclusions

The graphene incorporation into the epoxy matrix was chosen for its potential as high-performance nanocomposites. This work studied mixing different concentrations of graphene (0.002, 0.004, 0.006, and 0.008) in an epoxy matrix. The epoxy-graphene nanocomposites were synthesized using the combined action of the magnetic stirrer and sonication technique to reduce the aggregation and agglomeration of graphene. The FTIR verified the prepared samples by the presence of main, distinguishing peaks associated with vibrational groups. SEM images showed homogeneously dispersed graphene by exhibiting less agglomeration and aggregation. The AFM and surface roughness showed that the incorporation of graphene into the epoxy increased the hydrophobicity of the epoxy-graphene nanocomposite and decreased its surface roughness.

References

1. Y.-R. Zhang, S. Gu, Y.-Z. Wang, and L. Chen, Sust. Mat. Tech. **40**, e00883 (2024). DOI: 10.1016/j.susmat.2024.e00883.
2. X. Lu and X. Gu, Int. J. Bio.Macromole. **229**, 778 (2023). DOI: 10.1016/j.ijbiomac.2022.12.322.
3. M. Y. Khalid, A. Kamal, A. Otabil, O. Mamoun, and K. Liao, Chem. Eng. J. Advan. **16**, 100537 (2023). DOI: 10.1016/j.ceja.2023.100537.
4. F. P. Lodh and R. V. I. Gadhave, Open J. Comp. Mat. **14**, 60 (2023). DOI: 10.4236/ojcm.2024.141005.
5. A. R. Urade, I. Lahiri, and K. S. Suresh, JOM **75**, 614 (2023). DOI: 10.1007/s11837-022-05505-8.
6. P. Sayfo, D. Z. Pirityi, and K. Pölöskei, Mat. Today Chem. **29**, 101397 (2023). DOI: 10.1016/j.mtchem.2023.101397.
7. J. Kim, F. Kim, and J. Huang, Mat. Today **13**, 28 (2010). DOI: 10.1016/S1369-7021(10)70031-6.
8. A. K. Geim and K. S. Novoselov, Nat. Mat. **6**, 183 (2007). DOI: 10.1038/nmat1849.

9. H. Hiura, H. Miyazaki, and K. Tsukagoshi, Appl. Phys. Exp. **3**, 095101 (2010). DOI: 10.1143/APEX.3.095101.
10. V. B. Mbayachi, E. Ndayiragije, T. Sammani, S. Taj, E. R. Mbuta, and A. U. Khan, Res. Chem. **3**, 100163 (2021). DOI: 10.1016/j.rechem.2021.100163.
11. S. M. Omran, E. T. Abdullah, and O. A. Al-Zuhairi, Iraqi J. Sci. **63**, 3719 (2022). DOI: 10.24996/ijs.2022.63.9.6.
12. S. J. Lee, S. J. Yoon, and I.-Y. Jeon, Polymers **14**, 4733 (2022). DOI: 10.3390/polym14214733.
13. A. M. Díez-Pascual, Polymers **14**, 2102 (2022). DOI: 10.3390/polym14102102.
14. E. Toto, S. Laurenzi, and M. G. Santonicola, Polymers **14**, 1030 (2022). DOI: 10.3390/polym14051030.
15. X. Sun, C. Huang, L. Wang, L. Liang, Y. Cheng, W. Fei, and Y. Li, Adv. Mater. **33**, 2001105 (2021). DOI: 10.1002/adma.202001105.
16. X. Wang, X. Qi, Z. Lin, and D. Battocchi, Nanomaterials **8**, 1005 (2018). DOI: 10.3390/nano8121005.
17. S. Bellucci, J. Compos. Sci. **6**, 39 (2022). DOI: 10.3390/jcs6020039.
18. S. I. Hussein, Nano Hyb. Comp. **22**, 23 (2018). DOI: 10.4028/www.scientific.net/NHC.22.23.
19. N. Liu, Q. Tang, B. Huang, and Y. Wang, Crystals **12**, 25 (2022). DOI: 10.3390/cryst12010025.
20. M. R. Nobile, L. Guadagno, C. Naddeo, L. Vertuccio, and M. Raimondo, Mat. Today Proce. **34**, 160 (2021). DOI: 10.1016/j.matpr.2020.02.139.
21. W. Sun, T. Wu, L. Wang, Z. Yang, T. Zhu, C. Dong, and G. Liu, Comp. P. B Eng. **173**, 106916 (2019). DOI: 10.1016/j.compositesb.2019.106916.
22. M. S. Sahan and E. T. Abdullah, Basrah J. Sci. **40**, 128 (2022). DOI: 10.29072/basjs.20220107.
23. S. M. Kabeba, A. Hassanb, Z. Mohamadb, Z. Sharerb, M. Mokhtarb, and F. Ahmadc, Chem. Eng. J. **72**, (2019).
24. X. Wang, X. Qi, Z. Lin, and D. Battocchi, Nanomaterials **8**, 1005 (2018). DOI: 10.3390/nano8121005.
25. A. S. Zamil and A. N. Naje, J. Opt., (2023). DOI: 10.1007/s12596-023-01481-7.
26. A. N. Ali, M. A. Abd-Elnaiem, I. S. Hussein, S. A. Khalil, R. H. Alamri, and S. H. Assaedi, Current Nanosci. **17**, 494 (2021). DOI: 10.2174/1573413716999200820145518.
27. M. R. Ali, M. A. Chowdhury, M. Shahin, M. M. Rahman, M. O. Ali, and M. A. Gafur, Arabian J. Chem. **17**, 105424 (2024). DOI: 10.1016/j.arabjc.2023.105424.

توصيف مركبات الجرافين/الايبوكسي النانوية

مالك رشيد خشيت[1] وإستبرق طالب عبد الله[1]
[1]قسم الفيزياء، كلية العلوم، جامعة بغداد، العراق

الخلاصة

حظيت الصفائح النانوية من الجرافين باهتمام واسع النطاق من المجتمع العلمي في القرن الحادي والعشرين بسبب بنيتها المستوية ثنائية الأبعاد، ومساحة سطحها العالية، وقدراتها الوظيفية، وخصائصها الكهربائية والميكانيكية والحرارية الرائعة. في هذا العمل، تم تصنيع صفائح الجرافين / المركبات النانوية الإيبوكسي، بنسب وزنية (0.002، 0.004، 0.006، و0.008)، من خلال العمل المشترك لتقنيات الطرد المركزي والصوتنة لتقليل تجميع وتكتل الجرافين. يتحقق التحليل الطيفي للأشعة تحت الحمراء (FTIR) بمحول فورييه من العينات المحضرة من خلال وجود قمم رئيسية مميزة مرتبطة بالمجموعات الاهتزازية. يُظهر المجهر الإلكتروني الماسح (SEM) تشتتًا جيدًا للجرافين في الإيبوكسي، حيث يتم تفريقه بشكل متجانس من خلال إظهار تكتل وتجميع أقل. يُظهر الفحص المجهري للقوة الذرية (AFM) أن متوسط قيمة Ra لراتنجات الإيبوكسي كانت 29 نانومتر. بالمقارنة، كانت المركبات القائمة على الجرافين أقل من 5 نانومتر، مما يقلل بشكل كبير من خشونة السطح عن طريق إضافة كمية صغيرة من الحشوات النانوية. تظهر خشونة السطح أن الايبوكسي أكثر خشونة من المركبات النانوية.

الكلمات المفتاحية: كرافين، ايبوكسي، الموجات فوق الصوتية، زاوية الاتصال، مجهر القوة الذرية.