

SYNTHESIS AND CHARACTERIZATION OF A NOVEL NANO-COMPOSITE MATERIAL FOR MICROWAVE ABSORPTION

B.A.R. Zaki¹ F.L. Rashid¹ M.N. Al Baiati²

*1. Department of Chemistry, College of Education for Pure Sciences, University of Karbala, Karbala, Iraq
 batool.abdulrasool@s.uokerbala.edu.iq, farhan.lefta@uokerbala.edu.iq*

*2. Department of Petroleum Engineering, College of Engineering, University of Karbala, Karbala, Iraq
 mohammad.nadhum@uokerbala.edu.iq*

Abstract- Using poly (glycerol/phthalic anhydride) nanoparticles, a radar wave-absorbing material having high-efficiency was fabricated, and the impact of combining thermal paint with these nanocomposites on the electromagnetic absorption capabilities was examined. findings reveal that when the nano polymers proportion is raised from 2 percent to 4% in the presence of iron oxide at 2.5%, an increase in the value of reflectivity is considered, as seen by the results. For reflectivity of up to 38.0 dB at a bandwidth of (8.1-8.4 GHz) and frequency of 8.8 GHz, there is a radar waves distinct attenuation, which is restricted to a (9-9.3 GHz) bandwidth; The nanocomposite material was discovered to have strong qualities in microwave absorbing, which aided in the continual dissemination of work and radiation to weaken it, allowing the waves from the radar to be absorbed and attenuated. It can be used to absorb high-frequency radar waves with great efficiency.

Keywords: Nanocomposite, Electromagnetic Applications, Radar Absorbing Structures, Microwave Absorbing Material.

1. INTRODUCTION

Since the military radar systems introduction that can detect defense-based devices, scientists have been researching radar absorption materials (RAMs). The military sector recognizes X-band frequency radar systems for their high-resolution imaging and better target recognition [1-3]. Since the conclusion of WWII, radar-absorbent materials (RAMs) have piqued researchers' attention because to their amazing and potential applications in the field of modern stealth technologies [4].

Thin absorbent materials with high absorption efficiency, a wide absorption band, and a low weight may be called ideal and are outstanding [5]. To meet the needs of particular applications, the material having absorbing excellent properties must also have strong mechanical properties, resistance at high temperature, oxidation resistance and so on. As radar wave detecting technology advances, high-performance wave absorbers and new multi-functional wave absorbing materials are being developed across the world [6].

In the recent decade, microwave absorbers have gotten a lot of attention. Early investigations concentrated on other materials of dielectric loss, graphite, (porous, coils, and fibers) of carbon [7-12]. The proportion of electromagnetic radiation that a material blocks as it passes through it is known as its shielding efficiency (*SET*). A wide range of materials may be affected by electromagnetic radiation.

A component of the force, denoted by *PR* and *PI*, is reflected, while another force portion, denoted by *PR* and *PI*, is dissipated and absorbed by the materials' front face, leaving the remaining force (*PT*) to be transmitted through the shielding material [13-17]. This suggests that the entire attenuation is the product of three distinct processes: numerous internal interactions (*SEM*), absorption (*SEA*), and reversal (*SER*) [18, 19].

$$SET = 10 \log \frac{P_1}{P_2} - 20 \log \frac{E_1}{ET} - 20 \log \frac{H_1}{HT} \quad (1)$$

$$SET = SER + SEA + SEM \quad (2)$$

where, *T* stands for transmitted, *R* for reflected, and *I* for incident, while *P*, *E*, and *H* stand for energy intensity, electric field, and magnetic field respectively [18]. As a result, *SER* standing for net interaction and reflects *SEA*'s protection against uptake. Because of the influence of the output interface (secondary interactions), the *SEM* may be ignored, and the formula can be expressed as [18]:

$$SET = SER + SEA \quad (3)$$

A shields of electromagnetic interference primary mechanism is interaction (*SER*). Echo loss is the relative impedance mismatch between the shielding material surface and the EM waves. The response loss may be calculated using [20, 21]:

$$SER = 20 \log \frac{z^o}{4z_{in}} = 39.5 + 10 \log \frac{\sigma}{2f\pi\mu} \alpha \frac{\sigma}{\mu} \quad (4)$$

As z^o refers to the overall conductivity, *f* is frequency, and μ is the relative transmittance. *SER* is the material's conductivity and permeability function (μ), i.e., $SER f(\sigma / \mu)$. Therefore; constants μ and σ , the *SER* lowers with frequency. As a result, materials that reflect electromagnetic radiation must include mobile charges carriers (electrons or holes) [22].

Absorption loss is a supplementary technique for electromagnetic interference prevention (SEA). According to the plane wave theory [21], the electromagnetic wave amplitude decreases considerably when it moves through the matter. As a result, absorption losses are created by material heating and losses of ohmic caused by the currents generated. For materials having conductivity nature, the loss of absorption (SEA) in decibels (dB), is expressed as follows [18]:

$$SEA = 20 \log e_a^\alpha = 8.7d \sqrt{df \pi \sigma \mu \alpha} \quad (5)$$

As constants d and a are for thickness and slab attenuation, respectively. The amount by which the strength of an electromagnetic wave is weakened when it travels across a material is determined by a constant called the constant of attenuation. SEA depends on electrical conductivity (σ), transmittance (μ), and sample thickness (d), [23]. When shielding is accomplished via the process of adsorption rather than interaction, this reliance on and indicates that shielding in metals that are conductive ferromagnetic is done. Furthermore [24]:

$$\alpha = \frac{4\pi n}{\lambda_0} \quad (6)$$

where, λ_0 is the length of wave in vacuum and n refers to the index of refractive, and $\mu-1$ is given for non-magnetic materials. thus [18],

$$\alpha = \frac{4\pi \epsilon_2^1}{\lambda_0} \quad (7)$$

2. EXPERIMENTAL PART

A unique nano co-polymer was employed and developed as an electromagnetic radiation absorbing materials for radar applications. In this work, which included:

2.1. Polymeric Nano Material Preparation

The following steps were used to prepare the nano co-polymer in the ratio of 5:2: at a temperature of 110 degrees Celsius, phthalic anhydride of 740.5 gr, or 5.0 moles were dissolved in 65 ml of DMSO. When the combination turned into a transparent solution, glycerol of 184 grammes, or 2.0 moles were added to the round-bottom flask with a capacity of 250 ml. For the suspension solution, p-Xylene of 15 ml was dropped in batches over 15 minutes to remove any water molecules generated during the esterification process. Filtration was used to capture the solid white precipitation. A wide range of TEM, AFM, ¹H-NMR, and FTIR methods were used to study the co-polymer of these nanoparticles [25-27].

2.2. Different Mixtures Proportions Preparation

Heat resistance paint and polymeric material weights and ratios are shown in Table 1. The mixes were made according to the information listed in the table, as well as the mixing weights and ratios of the thermal dye and polymeric material that were utilized based on 20 grams.

2.3. Specimens

It was decided to make steel pieces that were (10.5×10.5) centimeters in size. The surface is cleaned, smoothed, and polished using emery papers to get it ready for the pigment, as well as to remove any dirt, dust, oxides, or sediments that could have been on it. This process was similar to how vehicle painters prepare the surface before painting it. In accordance with the standard protocol followed by scientific sources, the surface was cleaned using either acetone or alcohol. After the cleaning procedure, a primer from (Würth, Germany) was used as a base layer primer to boost the adhesion. This was done after the washing process. Following the step of combining the nanomaterial with the thermal dye in order to dilute the paint, the paint was sprayed with the addition of thinner before being applied.

Table 1. Heat resistance paint and polymeric material weights and mixing ratios

Mixture No.	Nano Polymer Weight %	Nano Fe ₃ O ₄ Weight %	Nano Polymer Weight (gr.)	Nano Fe ₃ O ₄ Weight (gr.)	Paint Weight (gr.)	Total Mixture Weight (gr.)
Paint only	-	-	-	-	-	20
1	2%	2.5%	0.4	0.5	19.1	20
2	4%	2.5%	0.8	0.5	18.7	20

3. RESULTS AND DISCUSSION

3.1. Nano Co-Polymer Characterization

Figure 1 reveals the diagram of FT-IR, at 3055 cm⁻¹ weak broadband is shown caused by the bond of hydrogen and because of the compensation on the aromatic ring that occurs on both sides, the firmness emerges at 897 and 734 cm⁻¹. The esteric (C-O) bond having a strong sharp crest at 1069 cm⁻¹. The riding bundles at 2992-2871 cm⁻¹ belongs to the symmetric and asymmetric (C-H) bond and the alcoholic (O-H) bond as it revealed a riding band at (3057 cm⁻¹) as a result of the aromatic (C-H) bond, and a strong riding bundle at (1770 cm⁻¹) emerges back to the Isterian (C=O) band, and riding bundles at 1495-1584 cm⁻¹. It corresponds to the (C=C) aromatic. At 13.12 ppm, to understand the single signal of the distinctive proton in carboxylic acid group, see Figure 2 (¹H-NMR spectrum).

In the region of 7.48-7.79 ppm, the multiply is a result of all aromatic ring protons, while the signals at 3.68-3.66 ppm and 4.26-4.28 ppm of methyl protons are attributed to the copolymer's methyl protons, respectively. The signal of aliphatic alcohols', on the other hand, has vanished, suggesting that this is a copolymer. The exterior surface of the copolymer nanoparticles is illustrated by the AFM images (Figures 3a and 3b). The surface roughness modulus of the copolymer was measured to be 22 nm, and its square root was equivalent to 26.6 nm. This lends credence to the idea that the black side of nanoparticles plays a key role in the regular crystal structure, surface uniformity, and surface quality. In addition, the typical height of the particles was 106.37 nm on average.

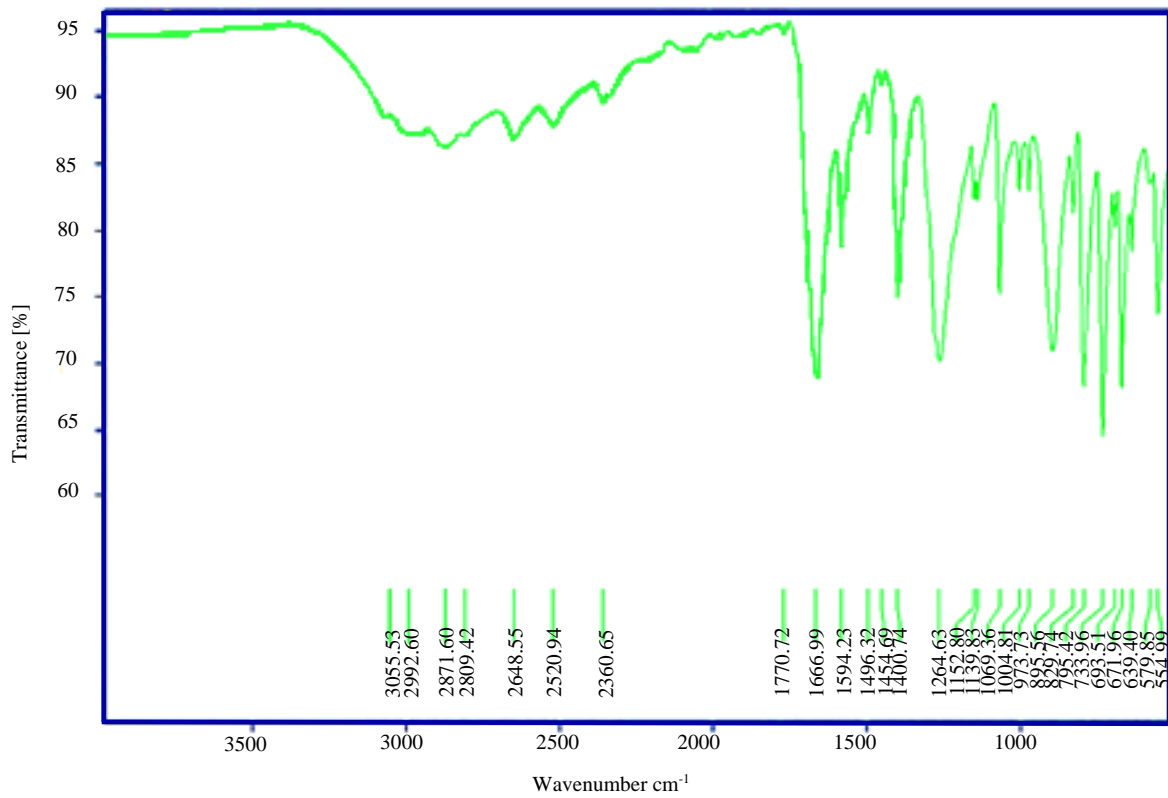


Figure 1. Nano co-polymer FT-IR

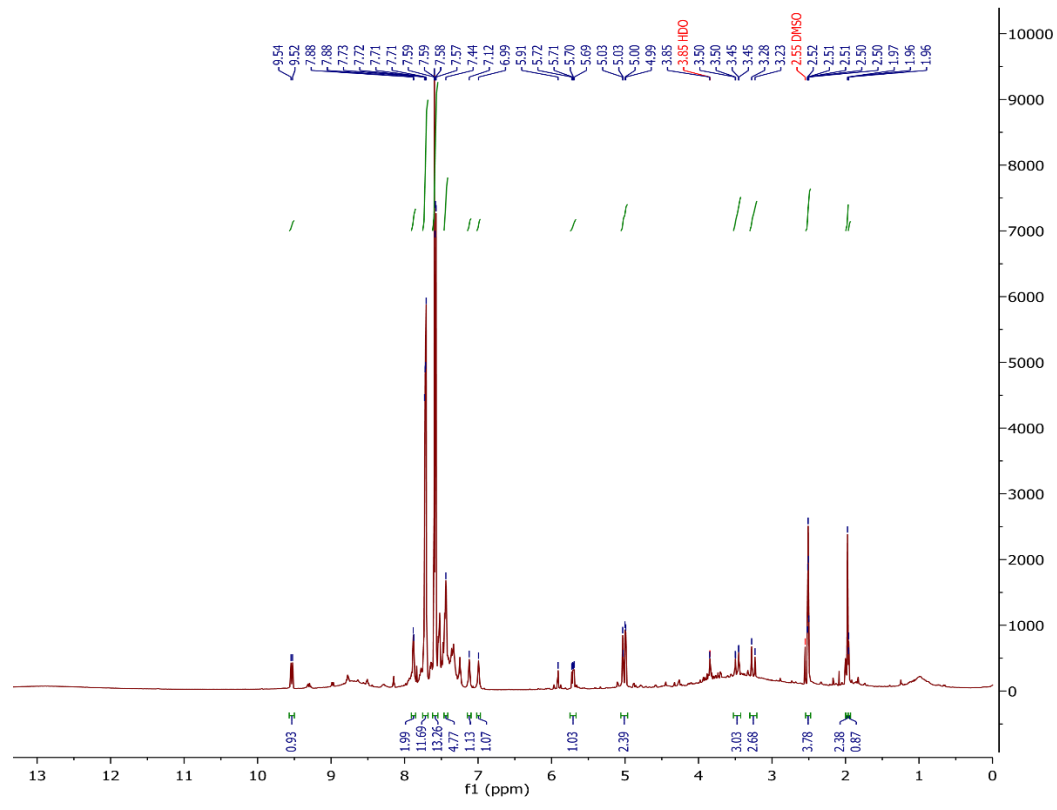


Figure 2. ¹H-NMR Spectrum

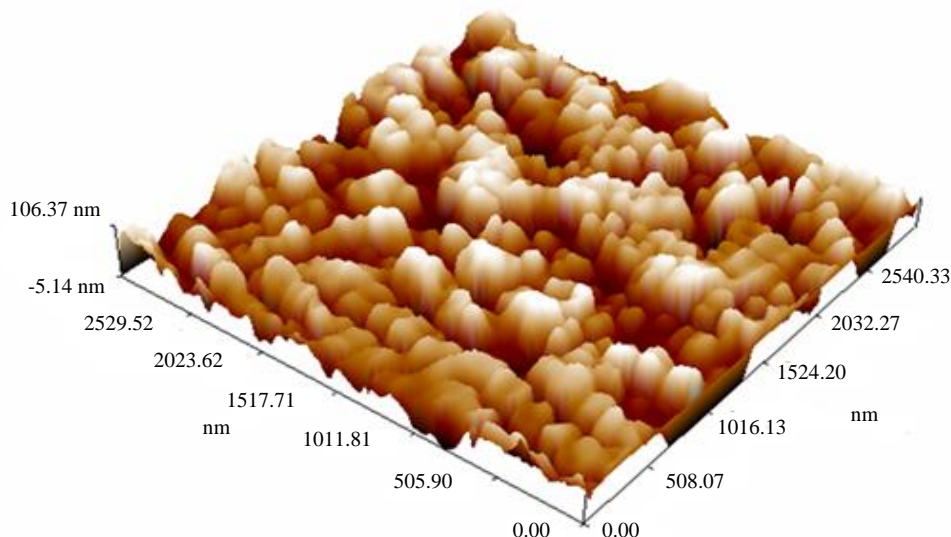


Figure 3. (a) 3D AFM Image

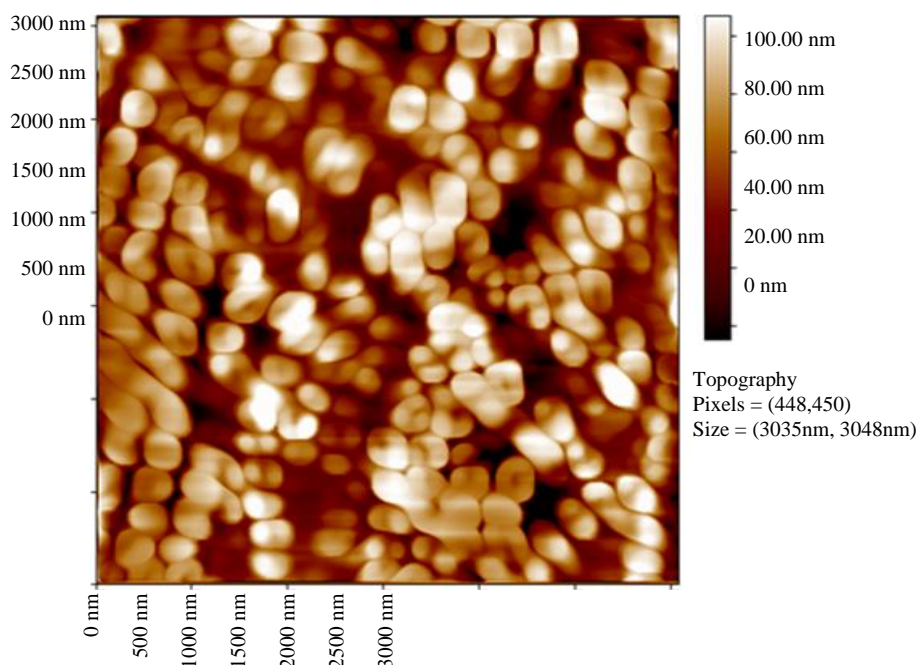


Figure 3. (b) 2D AFM Image

The particle size distribution of nano co-polymer is shown in Figure 4. The nanoparticles TEM micrographs of nano co-polymer having uneven layer-shaped particles of varied shapes and sizes on a shape of semi-sphere are shown in Figure 5. It was discovered that the nanoparticles of the co-polymer had an average size of 106.37 nm [28].

3.2. Reflectivity Loss

For samples manufactured with the device of radar wave attenuation and tested inside at 8-12 GHz (x-band), the frequency was evaluated with reflectivity loss. Figure 6 illustrates the relationship without coating between frequency and the reflectivity loss. When the nano polymer added to the coating is 2 percent, it reaches a value of 24.64 dB at a frequency of 8.3 GHz, which is equal to 0.4 gram,

as shown in Figure 7, when there are 2.5% iron oxide nanoparticles present.

At different frequencies, other magnitudes of the energy loss reflection can be found. When the nano polymers percentage is raised to 4%, i.e. 0.8 gram, with the presence of iron oxide at 2.5% to the thermal coating (heat resistance paint), a dramatic rise in the loss magnitude of reflectivity is observed.

Figure 8 illustrates the reflectivity up to 43.44 dB at a bandwidth of (8.1-8.4 GHz). As a consequence of these findings, it was discovered that the nanocomposite material had strong absorbing microwave waves properties in, which assisted in the continuous radiation dissemination and worked to weakening it, also helped attenuate and absorb the waves of radar [29, 30].

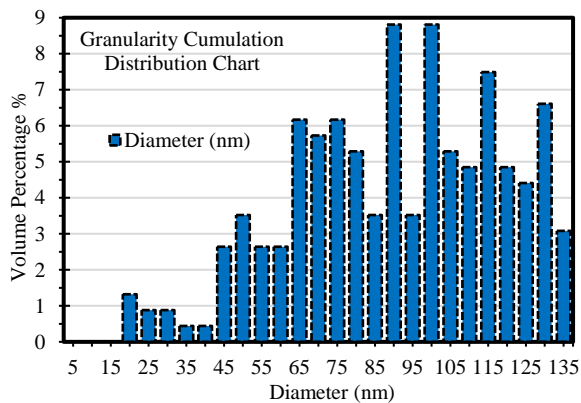


Figure 4. particle size distribution

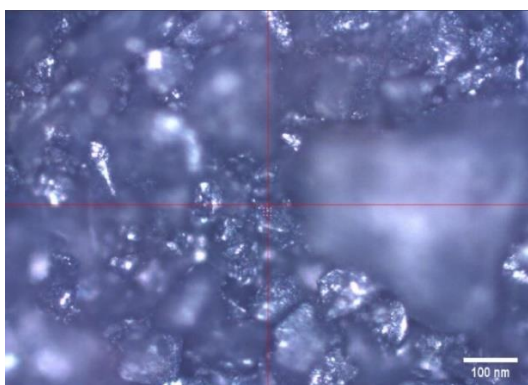


Figure 5. Nanoparticles co-polymer TEM micrographs

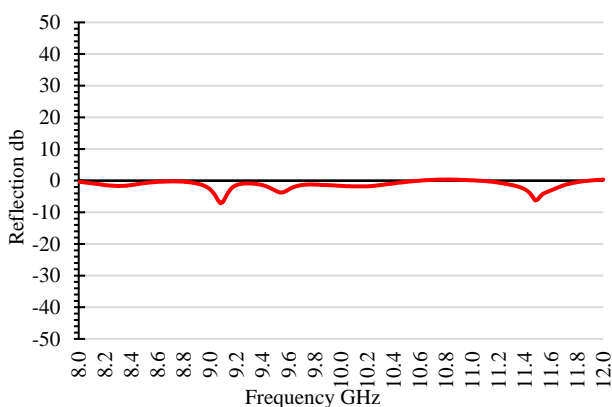


Figure 6. Frequency vis reflectivity at (0%) nano polymer

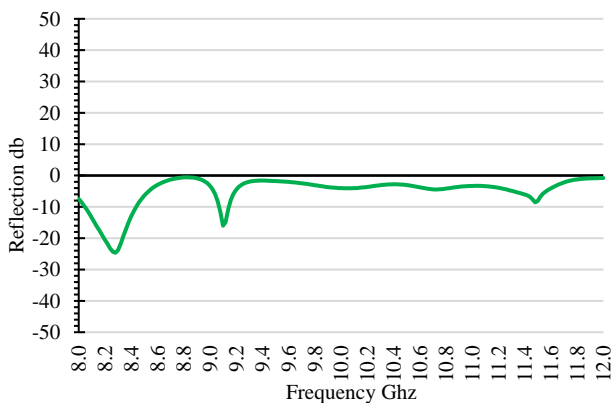


Figure 7. Frequency vis reflectivity at (2%) nanopolymer

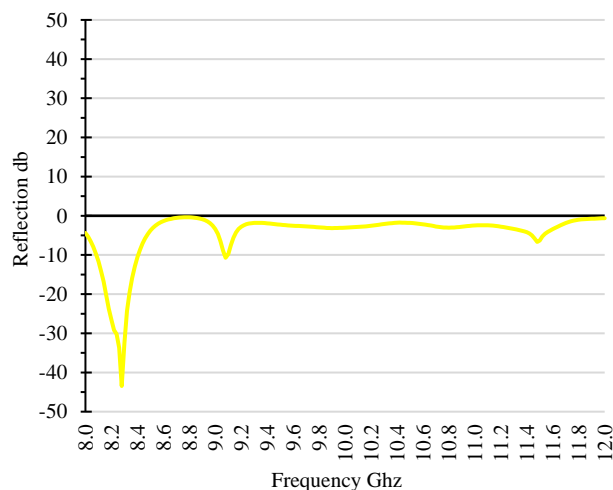


Figure 8. Frequency vis reflectivity at (4%) nano polymer

4. CONCLUSIONS

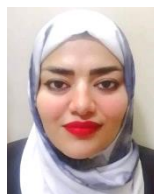
Condensation polymerization of glycerol and phthalic anhydride yielded a nanoparticles copolymer that was effectively melted utilizing the melting procedure by condensation polymerization. The dark side of nanoparticles clearly plays an important role in a regular crystal system, surface homogeneity, and surface finish quality as shown by the AFM images of nano co-polymers. These copolymer nanoparticles have an average particle size of 106.37 nm, which was determined from the images of the TEM micrographs of the uneven layer-shaped particles. The reflectivity loss value increases dramatically when the nano polymers proportion is increased to 4% and the iron oxide content is 2.5%. Researchers discovered that the nanocomposite material has excellent microwave absorption qualities, which helped to keep the radiation dispersed and weaken it, allowing it to be more effectively used to absorb and attenuate radar waves.

REFERENCES

- [1] A.H. Naheda Humood, E.H. Abdullah, S.M. Abu Alais, F.L. Rashid, A. Hadi, "Polymer-CoFe₂O₄ Nanocomposites as Flexible Microwave-Radiation Absorbing and High Corrosion Resisting Coating Materials for Biological Applications", Nano systems, Nanomaterials, Nanotechnologies, Vol. 19, No. 3, pp. 689-695, Ukraine, 2021.
- [2] R. Panwar, V. Agarwala, D. Singh, "A Cost-Effective Solution for Development of Broadband Radar Absorbing Material using Electronic Waste", Ceram. Int., Vol. 41, No. 2, Part B, pp. 2923-2930, 2015.
- [3] F.L. Rashid, "Gamma and X-Rays' Shielding of New Nanomaterials for Biomedical Applications", Nano systems, Nanomaterials, Nanotechnologies, Vol. 19, No. 3, pp. 697-705, Ukraine, 2021.
- [4] Z. Zhang, et al., "Cellulose-Chitosan Framework/ Polyaniline Hybrid Aerogel Toward Thermal Insulation and Microwave Absorbing Application", Chem. Eng. J., Vol. 395, p. 125190, 2020.
- [5] X. Lin, et al., "Targeted Design and Analysis of Microwave Absorbing Properties in Iron-Doped

- SiCN/Si₃N₄ Composite Ceramics", *Ceram. Int.*, Vol. 47, No. 4, pp. 4521-4530, 2021.
- [6] A. Ling, et al., "Broadband Microwave Absorbing Materials Based on MWCNTs' Electromagnetic Wave Filtering Effect", *Compos. Part B Eng.*, Vol. 171, pp. 214-221, 2019.
- [7] Y. Cui, et al., "Three Dimensional Porous MXene/CNTs Microspheres: Preparation, Characterization and Microwave Absorbing Properties", *Compos. Part A Appl. Sci. Manuf.*, Vol. 145, p. 106378, 2021.
- [8] Z. Qu, Y. Wang, W. Wang, D. Yu, "Hierarchical FeCoNiOx-PDA-rGO/WPU Layers Constructed on the Polyimide Fabric by Screen Printing with High Microwave Absorption Performance", *Appl. Surf. Sci.*, Vol. 562, p. 150190, 2021.
- [9] B.S. Kwak, G.W. Jeong, W.H. Choi, Y.W. Nam, "Microwave-Absorbing Honeycomb Core Structure with Nickel-Coated Glass Fabric Prepared by Electroless Plating", *Compos. Struct.*, Vol. 256, p. 113148, 2021.
- [10] Y. Liu, et al., "Microwave Absorbing Performance of Polymer-Derived SiCN (Ni) Ceramics Prepared from Different Nickel Sources", *J. Alloys Compd.*, Vol. 749, pp. 620-627, 2018.
- [11] M. Zhang, C. Ma, H. Liu, Q. Liu, "Controllable Magnetic Properties and Enhanced Microwave Absorption of Ba₂Mg₂Fe₁₂O₂₂@Ni_{0.5}Zn_{0.5}Fe₂O₄ Multi-Walled Carbon Nanotubes Composites", *J. Alloys Compd.*, Vol. 861, p. 158624, 2021.
- [12] X. Zhuang et al., "Boosted Microwave Absorbing Performance of Ce₂Fe₁₇N₃₋₈@SiO₂ Composite with Broad Bandwidth and Low Thickness", *J. Alloys Compd.*, Vol. 883, p. 160835, 2021.
- [13] S.J. Mezher, M.O. Dawood, O.M. Abdulmunem, M.K. Mejbel, "Copper Doped Nickel Oxide Gas Sensor", *Vacuum*, Vol. 172, p. 109074, February 2020.
- [14] S.J. Mezher, M.O. Dawood, A.A. Beddai, M.K. Mejbel, "NiO Nanostructure by RF Sputtering for Gas Sensing Applications", *Mater. Technol.*, Vol. 35, No. 1, pp. 60-68, January 2020.
- [15] S.J. Mezher, K.J. Kadhim, O.M. Abdulmunem, M.K. Mejbel, "Microwave Properties of Mg-Zn Ferrite Deposited by the Thermal Evaporation Technique", *Vacuum*, Vol. 173, p. 109114, March 2020.
- [16] H. Mikhilif, M. Dawood, O. Abdulmunem, M.K. Mejbel, "Preparation of High-Performance Room Temperature ZnO Nanostructures Gas Sensor", *ACTA Phys. Pol. A*, Vol. 140, No. 4, pp. 320-326, 2021.
- [17] W.J. Aziz, M.A. Abid, D.A. Kadhim, M.K. Mejbel, "Synthesis of Iron Oxide (β-Fe₂O₃) Nanoparticles from Iraqi Grapes Extract and its Biomedical Application", *IOP Conf. Ser. Mater. Sci. Eng.*, Vol. 881, p. 12099, 2020.
- [18] D. Jiang, et al., "Electromagnetic Interference Shielding Polymers and Nanocomposites-a Review," *Polym. Rev.*, Vol. 59, No. 2, pp. 280-337, 2019.
- [19] V. Shukla, "Review of Electromagnetic Interference Shielding Materials Fabricated by Iron Ingredients", *Nanoscale Adv.*, Vol. 1, No. 5, pp. 1640-1671, 2019.
- [20] F. Meng, et al., "Graphene-Based Microwave Absorbing Composites: A Review and Prospective", *Compos., Part B Eng.*, Vol. 137, pp. 260-277, 2018.
- [21] M. Sharma, M. P. Singh, C. Srivastava, G. Madras, S. Bose, "Poly (Vinylidene Fluoride)-Based Flexible and Lightweight Materials for Attenuating Microwave Radiations", *ACS Appl. Mater. Interfaces*, Vol. 6, No. 23, pp. 21151-21160, 2014.
- [22] Z. Liu, et al., "Microwave Absorption of Single-Walled Carbon Nanotubes/Soluble Cross-Linked Polyurethane Composites", *J. Phys. Chem. C*, Vol. 111, No. 37, pp. 13696-13700, 2007.
- [23] Y. Cheng, et al., "An Unusual Route to Grow Carbon Shell on Fe₃O₄ Microspheres with Enhanced Microwave Absorption", *J. Alloys Compd.*, Vol. 762, pp. 463-472, 2018.
- [24] F. Ren, H. Yu, L. Wang, M. Saleem, Z. Tian, P. Ren, "Current Progress on the Modification of Carbon Nanotubes and Their Application in Electromagnetic Wave Absorption", *RSC Adv.*, Vol. 4, No. 28, pp. 14419-14431, 2014.
- [25] T.R. Mehdiyev, A.M. Hashimov, S.N. Aliyeva, I.F. Yusibova, A.A. Sadigova, "Optic Radiation of Thin-Film Structure of FD-Resistor with Active Ni_{0.4}Zn_{0.6}Fe₂O₄ Layer", *International Journal on Technical and Physical Problems of Engineering (IJTPE)*, Issue 50, Vol. 14, No. 1, pp. 161-165, March 2022.
- [26] N.N. Mursakulov, S.G. Nuriyeva, N.N. Abdulzade, K.M. Guliyeva, "Cu₂ZnSnSe₄ Thin Films for Solar Cell Applications", *International Journal on Technical and Physical Problems of Engineering (IJTPE)*, Issue 50, Vol. 14, No. 1, pp. 156-160, March 2022.
- [27] M.K. Allawi, M.K. Mejbel, M.H. Oudah, "Iraqi Gasoline Performance at Low Engine Speeds", *IOP Conf. Ser. Mater. Sci. Eng.*, Vol. 881, p. 12065, 2020.
- [28] A.K. Ghazi, A.A. Muhmmmed, N.K. Taieh, M.K. Mejbel, "Tribological and Mechanical Performance of Epoxy Reinforced by Fish Scales Powder", *Rev. des Compos. of the Materials Advances*, Vol. 32, No. 3, pp. 149-155, France 2022.
- [29] M.M. Lu, et al., "Multi-Wall Carbon Nanotubes Decorated with ZnO Nanocrystals: Mild Solution-Process Synthesis and Highly Efficient Microwave Absorption Properties at Elevated Temperature", *J. Mater. Chem. A*, Vol. 2, No. 27, pp. 10540-10547, 2014.
- [30] S.W. Phang, T. Hino, M.H. Abdullah, N. Kuramoto, "Applications of Polyaniline Doubly Doped with p-Toluene Sulphonic Acid and Dichloroacetic Acid as Microwave Absorbing and Shielding Materials", *Mater. Chem. Phys.*, Vol. 104, No. 2-3, pp. 327-335, 2007.

BIOGRAPHIES



Batool Abdul Rasool Zaki was born in Babylon, Iraq on October 11, 1990. She received her B.Sc. degree from Department of Chemistry, College of Education for Pure Sciences, University of Karbala, Karbala, Iraq in 2012. She is now the M.Sc. student at the same university. She has published one paper in nanotechnology.



Farhan Lafta Rashid was born in Babylon, Iraq on August 1, 1970. He completed the B.Sc. on Nuclear Engineering from University of Baghdad, Baghdad, Iraq in 1994 and the M.Sc. and the Ph.D. on Mechanical Engineering/ Thermo-Fluid Mechanics from University of Technology, Baghdad, Iraq in 2002 and 2015, respectively. He started as a researcher in the Iraqi Atomic Energy Commission. He later moved on to University of Kerbala as a lecturer of Fluid Mechanics and Thermodynamics in Petroleum Engineering Department, at the same time lecturer of Theory and Engineering of Nuclear Reactor in Energy Engineering Department, University of Babylon, Babylon, Iraq. His research interest in analysis of radiation shielding, attenuation, energy, CFD, modeling and simulation. He has more than 154 published papers in respected journals. He has five patents.



Mohammad N. Al Baiati was born in Baghdad-Iraq on April 20, 1970. He completed the B.Sc., M.Sc. and Ph.D. in Chemical Science from University of Baghdad, Baghdad, Iraq in 2003, 2005 and 2008, respectively. He is now a Professor in Department of Chemistry, College of Education for Pure Sciences, University of Kerbala, Kerbala, Iraq. His current interest is in chemical engineering, nanotechnology, waste water, polymers, materials, characterization, physical properties of different polymers. He also works now as a Dean Assistant for scientific affairs of Kerbala University. He has over 35 published papers in the field of chemical science and nanotechnology. He has seven patents.