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Glycerol /Phthalic Anhydride Novel Nano Composite for Microwave Absorbing Applications

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ABSTRACT

High-efficiency radar wave absorbing material was prepared utilizing nanoparticles poly (glycerol /phthalic anhydride), and the effect of using these nanocomposites with thermal paint on the properties of electromagnetic absorbing was analyzed and investigated. Results show that when the percentage of nano polymers is increased from 1% to 3%, with the presence of 2.5% of iron oxide, the reflectivity loss value rises dramatically. For the reflectivity up to 27.2dB at a frequency of 8.3 GH and a bandwidth of (8.1-8.2GH), there is a clear attenuation of the radar waves, which is confined within a bandwidth of GH (9-9.3GH). This material can be utilized as a high- efficiency radar wave absorbing applications.

1. INTRODUCTION

Research into radar absorption materials (RAMs) has been a hot topic among scientists since the development of military radar systems that can identify defence-based devices. With the fast growth of wireless technologies, digital gadgets, and radar systems, electromagnetic (EM) waves have become significant pollution sources that are hazardous to human physical health and national defense security. Microwave absorbing materials (MAMs) are used to attenuate undesired electromagnetic radiation in order to reduce the danger. Because of its high-resolution imagery and improved target recognition, radar systems of X-band frequency are widely recognized in the military industry [1]. Due to its spectacular and prospective uses in the realm of current stealth technology, radar-absorbent materials (RAMs) have garnered a lot of interest from researchers since the end of World War II [2]. Thin absorbing materials, low weight, have a broad absorption band, and have high absorption efficiency are excellent and may be considered to be an ideal material [3]. The absorbing material must also have oxidation resistance, high-temperature resistance, strong mechanical qualities, and so on, to suit the demands of certain applications. New multi-functional wave absorbing materials and high-performance wave absorbers are being developed across the world as radar wave detecting technology continues to advance [4]. Microwave absorbers have been the subject of a lot of study in the last decade. carbon fibers, carbon coils, porous carbon, graphite, and other dielectric loss materials were the subject of early study [5-10]. According to a survey of the literature, despite the numerous research on microwave absorbing materials, no prior results on the problem of improving the microwave absorbing techniques are accessible to the authors' knowledge. As a result, the goal of this research is to prepare and characterize the ability of the prepared nanocomposite materials to absorb the microwave. The influence of several relevant factors like nanopolymer weight mixing and nano Fe_3O_4 weight mixing will be investigated.

A material's shielding efficiency (SET) may be defined as the percentage of electromagnetic energy it blocks as it travels through it [11-13]. As seen in Figure 1, electromagnetic waves may interact with a variety of different materials. When electromagnetic waves land on the front face of the material, some of the force (PI) (PR) is reflected, while some of the force (PI) (PR) as energy is absorbed and dissipated, and the remaining force (PT) is transferred via the material of shielding. This means that the total attenuation is a result of a combination of three separate processes, namely, multiple internal interactions (SEM), absorption (SEA), and reversion (SER) [11, 14-17].
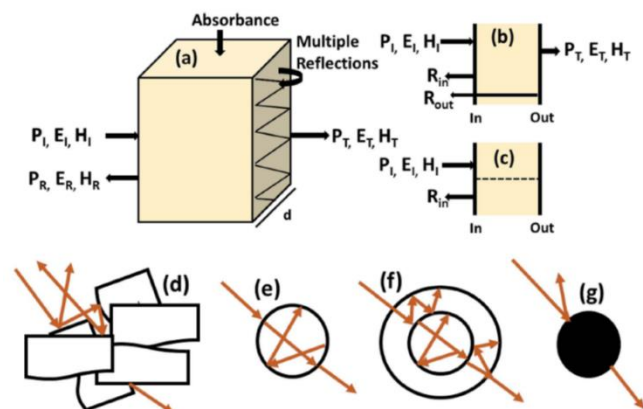
(a) Schematic diagram of electro-magnetic field intensities, transmitted power, reflected, and an incident. (b) and (c) thin sample reflection resources. (d–g) in the instance of a solid spherical, a structural multiple shells, a hollow structure, and a porous structure [18].

Figure 1. Electromagnetic waves interacting with a variety of different materials

$$SET = 10log\frac{p1}{p1} - 20log\frac{E1}{ET} - 20log\frac{HI}{HT} = SER + SEA + SEM \quad (1)$$

where, T is the transmitted, R is the reflected, and I is the incident components, while H, E, and P denote the magnetic field, electric, and energy intensities respectively [19]. Thus, SER stands for net interaction and represents the protection of SEA due to uptake. Note that the SEM can be neglected due to the contributions from secondary interactions (the output interface), then the formula could be written as [20-23]:

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

The basic mechanism of an electromagnetic interference shield (SER) is interaction. The echo loss (SER) refers to the mismatch of relative impedance between the EM waves and the shielding material surface. The value of the reaction loss can be determined by [24]:

$$SER = 20log\frac{z°}{4zin} = 39.5 + 10log\frac{\sigma}{2f\pi\mu}\alpha\frac{\sigma}{\mu} \quad (3)$$

where, μ the relative transmittance, frequency (f), and s is is the overall conductivity. SER is the materials' permeability and conductivity function (μ), i.e. SER f (σ / μ). Thus, for the constants σ and, the SER decrease with frequency. Consequently, materials must contain carriers of mobile charges (holes or electrons) to reflect electromagnetic radiation [25].

A secondary mechanism for protection against electromagnetic interference is absorption loss (SEA). The electromagnetic wave's amplitude reduces dramatically within matter as it travels through it, according to the plane wave hypothesis [26]. So, the absorption loss is caused by heating of the material and ohmic losses owing to generated currents. The absorption loss (SEA) in decibels (dB) may be stated for conductive materials [27]:

$$SEA = 20loge_{a=}^{\alpha}8.7d\sqrt{f\pi\sigma\mu\alpha d\sigma\mu\alpha d} \quad (4)$$

where, a and d are the slab attenuation and thickness constants, respectively. The constant of attenuation determines how much the intensity of an electromagnetic wave decreases as it passes through a material. SEA depends on sample thickness

(d), transmittance (μ), and electrical conductivity (σ) [28]. This SER/SEA dependence on and specifies that in ferromagnetic conductive metals, the domination of shielding is by adsorption rather than interaction. Furthermore [29]:

$$\alpha = \frac{4\pi n}{\lambda 0} \quad (5)$$

where, n is the index of refractive and λ_0 is the wavelength in a vacuum, which in the case of non-magnetic materials is given μ-1. So,

$$\alpha = \frac{4\pi\varepsilon_2^1}{\lambda 0} \quad (6)$$

2. EXPERIMENTAL PART

In this study, a novel nano co-polymer was prepared and used in absorbing materials for electromagnetic radiation for radar, which includes:

2.1 Preparation of the polymeric nano material

The novel nano co-polymer was prepared in ratio (5: 2) as follows: In 250 mL round bottom flask, (740.5 gm, 5.0 mole) of Phthalic anhydride was dissolved in 70 ml DMSO at 110°C when the mixture became a clear solution, (184 gm, 2.0 mole) of Glycerol was then added. Along 15 min, 15 ml of p-Xylene was dropped in batches to eliminate the water molecules which are formed during the esterification reaction, then added cooled deionized water to get the suspension solution. Finally, the solid white precipitated was collected by filtration. These nanoparticles' co-polymer was characterized using a spectrum of FT-IR, H-NMR, AFM and TEM techniques.

2.2 Preparation of different mixtures proportions

Table 1 showed the mixing ratios and weights of the polymeric material and thermal dye. The mixtures were prepared based on what is mentioned in the table and on the mixing ratios and weights of the polymeric material and thermal dye that were used based on (20) g.

Table 1. Mixing ratios and weights of the polymeric material and thermal dye

Mixture No.	Nano Polymer Weight Mixing %	Nano Fe$_3$O$_4$ Weight Mixing %	Nano Polymer Weight (gr.)	Nano Fe$_3$O$_4$ Weight (gr.)	Paint Weight (gr.)	Total Mixture Weight Paint + Nano polymer +Nano Fe$_3$O$_4$ (gr.)
Paint only	---	----	---	----	----	20
1	1%	2.5%	0.2	0.5	19.3	20
2	3%	2.5%	0.6	0.5	18.9	20

2.3 Specimens

Steel pieces with dimensions of (10.5 x 10.5) cm were prepared [30-34]. After that, the surface was polished and smoothed with polishing papers (grade 400) to prepare the surface to receive the mixture, as well as to clean the surface from any sediments, oxides, dust or dirt, just like preparing the surface for car painters [35-39]. The surface was washed with

fresh water, dried for 1 hour, then cleaned with acetone or alcohol, according to what is customary among scientific sources. After the cleaning process, a base layer primer was used to increase the adhesion, as a primer from (model thick primer filler, WURTH, Germany) [40, 41]. The paint (heat resistance paint, model dr ferro, black color, turkey origin) was sprayed with the addition of thinner (Nitro Cellulose thinner, Jordan origin) having a mixing ratio of 1:1, after the process

of mixing the nanomaterial, iron oxide with the thinner to be ready for paint. Figure 2 illustrates the raw materials utilized. Figure 3 illustrates the steel samples after applying the primer coat as shown in Figure 3 (a), and after applying the full mixture coat as shown in Figure 3 (b). the mixture was sprayed using an air compressor gun at 6 Bar of pressure.

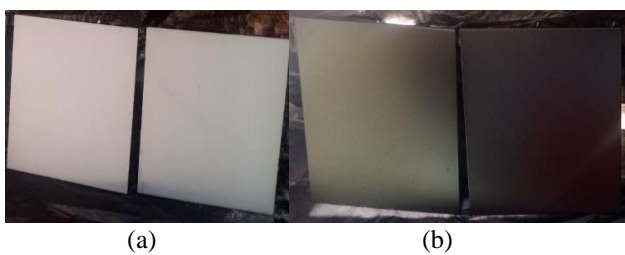

Figure 2. Raw materials used

(a)	(b)

Figure 3. Samples of the steel (a) only a primer coating paint; (b) after applying the mixture paint

A network vector analyzer (VNA) model (VectorStar, Anritsu company, Japan) was utilized to study the reflectivity loss characteristics of the samples as shown in Figure 4.

Figure 4. VNA setup

3. RESULTS AND DISCUSSION

3.1 Characterization of prepared novel nano co-polymer

Figure 5 illustrates the FT-IR diagram, which shows weak broadband at (3055 cm^{-1}) due to the alcoholic (O-H) bond and the hydrogen bond, as it showed a riding band at (3057 cm^{-1}) caused by the aromatic (C-H) bond and the riding bundles at (2871-2992 cm^{-1}) belong to the asymmetric and symmetric (C-H) bond, and a strong riding bundle at (1770-cm^{-1}) appears back to the Isterian (C=O) band, and riding bundles at (1584-1495 cm^{-1}). It refers to the (C=C) aromatic, strong sharp crest at (1069 cm^{-1}) of the esteric (C-O) bond, and the firmness appears at (734 and 897 cm^{-1}) due to the bilateral compensation on the aromatic ring. Figure 6 shows the [1]H-

NMR spectrum, which explains the single signal at 13.12ppm of the characteristic proton in the group of carboxylic acid. However, the multiplier in the region 7.48-7.79 ppm is attributed to all protons in the aromatic ring, the signals at 4.26-4.28 ppm methylene protons in the copolymer structure, and the multiply at 3.68-3.66 ppm of methyl protons, but the aliphatic alcohols signal disappeared indicating that copolymer composition.

Figures 7 (a, b) show the copolymer nanoparticles' outer surface. The copolymer surface roughness modulus was 22 nm and the sqrt was equal to (26.6) nm. This suggests that the nanoparticles dark side plays a significant importance in the surface finish, surface homogeneity and regular crystal system. Also, the average particle height was equal to 106.37 nm. Figure 8 illustrates the particle size distribution of the nano co-polymer.
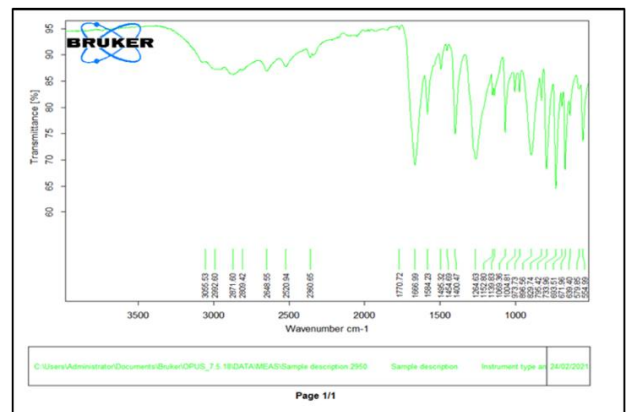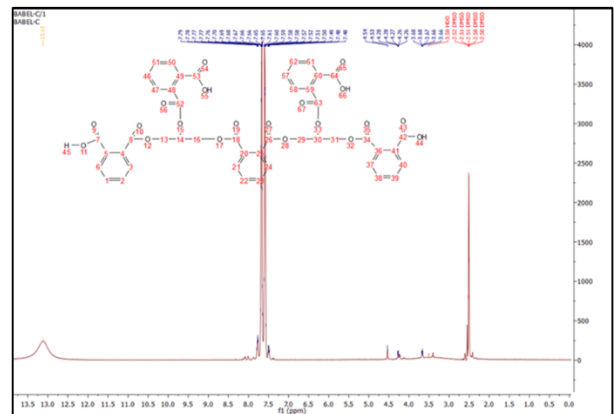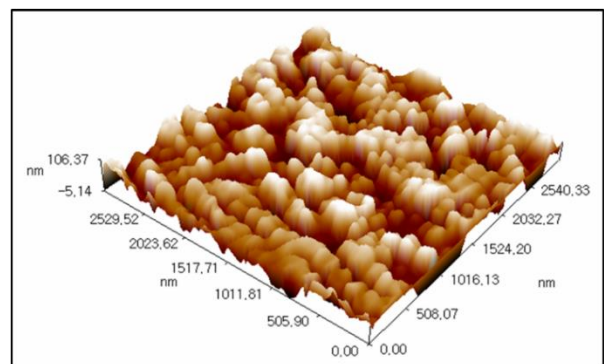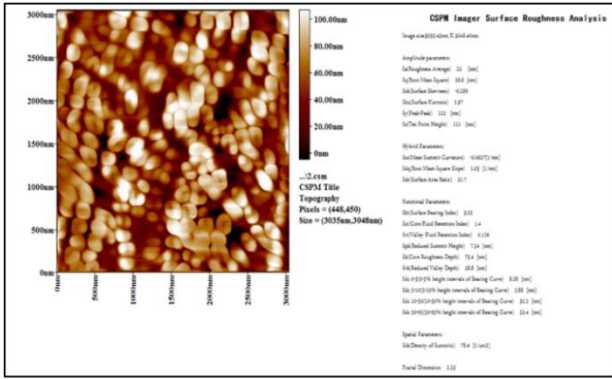

Figure 5. Nano co-polymer FT-IR

Figure 6. ^{1}HNMR Spectrum

(a)

(b)

Figure 7. (a) 3D nano co-polymer AFM Image. (b) 2D nano co-polymer AFM Image
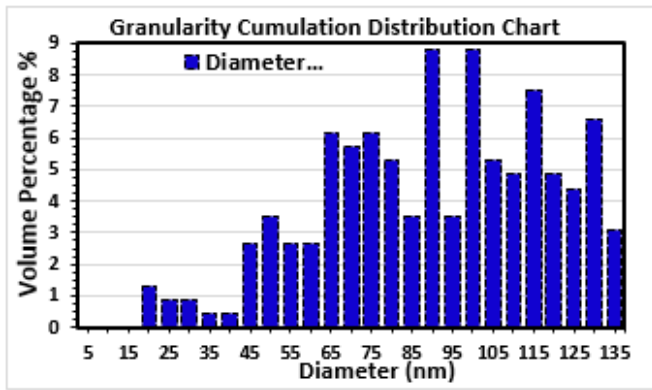

Figure 8. Nano co-polymer particle size distribution

Figure 9 illustrates the nano co-polymer TEM micrographs of nanoparticles containing irregular layer-shaped particles of different sizes and shapes on a semi-spherical shape. It was found that the average size of nanoparticles of the co-polymer is 106.37 nm.
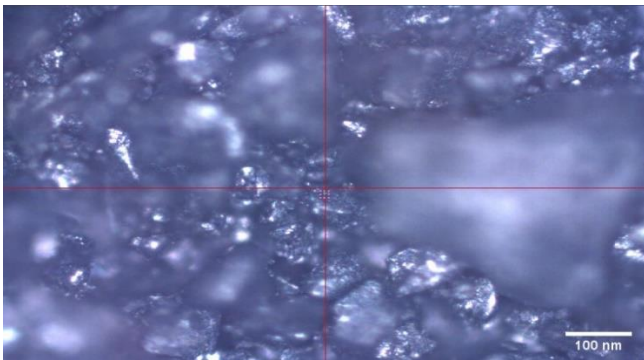

Figure 9. TEM micrographs for the nanoparticles co-polymer

3.2 Reflectivity loss

The reflectivity loss was measured with frequency for the samples that were prepared using the radar wave attenuation device and within the (x-band) 12-8 band. Figures 10 show the relationship between the loss of reflectivity and frequency without coating. When the percentage of nano polymer added

is 1%, that is, 0.2 gm to the coating, as in Figures 11 in the presence of 2.5 nanoparticles of iron oxide, it reaches 25.3 dB - at frequency 9.15 GH with other values for the reflection energy loss at different frequencies. When the percentage of nano polymers is increased to 3%, i.e. 0.4, to the thermal coating with the presence of 2.5% of iron oxide, we find that the value of the reflectivity loss increases significantly. Figures 12 show the reflectivity up to 27.2 dB at a frequency of 8.3 GH and bandwidth of (8.1-8.2 GH), thus, there is a clear attenuation of the radar waves, and we note another reflectivity value of 10.7 dB at the frequency 9.14GH, which is confined within a bandwidth of GH (9-9.3 GH). Through these results, it was found that the nanocomposite material has high properties in absorbing microwave waves and thus helped in the continuous dispersal of radiation and work to weaken it and thus absorb and attenuate the waves coming from the radar [42, 43].
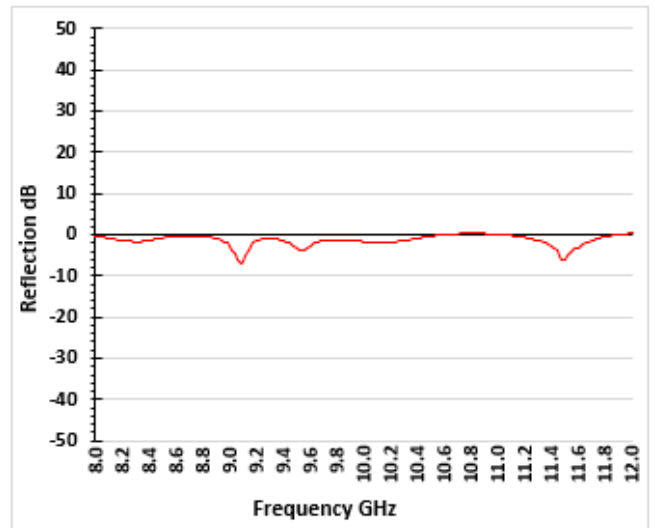

Figure 10. The relationship between reflectivity and frequency without coating metal parts
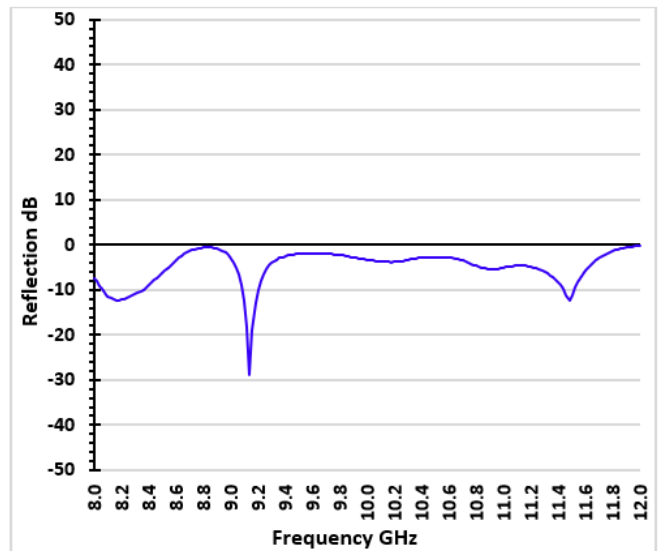

Figure 11. The relationship between reflectivity and frequency in the presence of paint (1%) nano polymer
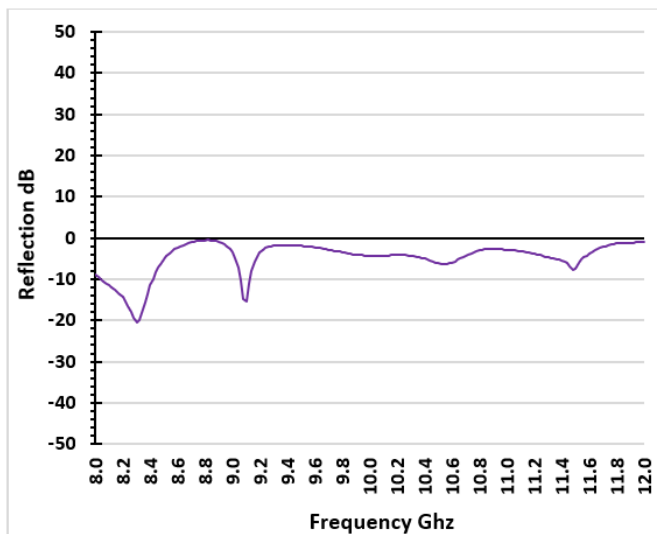
Figure 12. Shows the relationship between reflectivity and frequency in the presence of paint (3%) nano polymer

4. CONCLUSIONS

The nanoparticles copolymer was successfully synthesized using the melting process by condensation polymerization from the reaction of glycerol with phthalic anhydride with the release of water as a by-product. From the nano co-polymer AFM Images, it is clear that the nanoparticles dark side plays a significant importance in the surface finish quality, surface homogeneity and regular crystal system. From the TEM micrographs of copolymer nanoparticles which contain irregular layer-shaped particles of different sizes and shapes on a semi-spherical shape; It was found that the average size of nanoparticles of the copolymer is 106.37 nm. Results show that when the percentage of nano polymers is increased to 3%, with the presence of 2.5% of iron oxide, the value of the reflectivity loss significantly increases. For the reflectivity up to 27.2 dB at a frequency of 8.3 GHz and a bandwidth of (8.1-8.2 GHz), there is a clear attenuation of the radar waves, which is confined within a bandwidth of (9-9.3 GHz). As a consequence of this, the combination of glycerol and phthalic anhydride, with the appropriate proportions of each component, would be an appealing possibility for microwave-absorbing materials.

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