Enhancement of carbon fiber/epoxy composite electrical, optical and thermal properties by using different types of nano-additives

Enhancement of carbon fiber/epoxy composite electrical, optical and thermal properties by using different types of nano-additives.

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Abstract. Environmental space threats are becoming more critical as they affect the optical, thermal, and electrical properties of the reinforced fiber polymeric-based materials in spacecraft. Three different Nano-particles Alumina (Al_2O_3), Multiwall Carbon Nanotubes (MWCNT), and Reduced Graphene Oxide (RGO) were added to the epoxy matrix and then reinforced by bidirectional carbon fiber plain to form carbon fiber/epoxy by hand lay-up using autoclave curing technique to make three different reinforced materials. In this paper, the electrical, optical, and thermal properties of the carbon fiber/Epoxy Nanocomposite were studied.
Fourier transform infrared (FTIR) was performed to evaluate the structural changes in the newly synthesized materials. The optical, thermal, and electrical properties were tested by UV-visible Spectroscopy, Photo-acoustic spectroscopy (PA), and Keithley 2635A respectively. The results showed an enhancement in the electrical, optical, and thermal properties of the epoxy matrix after the addition of Nano-particles.
The optical test showed that the neat epoxy and epoxy/Nano-particles absorption spectra were in the infrared range. The thermal test indicated that the three thermal parameters diffusivity, effusivity, and conductivity showed the best enhancement after the addition of MWCNTs. The electrical test pointed out that after the addition of Nano-particles, neat epoxy changed from an insulating material to a semi-conductive material.

1. Introduction

Many environmental threats cause the degradation of materials used on the external panels of spacecraft [1]. For this reason, it is important to keep up-to-date and improve the existing materials and establish new materials with enhanced properties for proper use in space applications [2]. Space radiation causes the spacecraft material properties degradation and this degradation affects the spacecraft orbit lifetime. There are two kinds of space radiation, charged particles radiation and electromagnetic radiation [3].

Epoxy resin is the most widely used matrix in polymer-based composites. The reason behind this, the epoxy matrix contains good chemical, electrical, thermal, and mechanical properties [4]. However, the typical thermal conductivity of epoxy ranged between (0.17–0.21 W/mK) and often exhibits a brittle nature [5]. Therefore Nano-reinforcements such as Nano-ceramic, carbon nanotubes, and graphene structures are usually employed to enhance the deficiencies in neat epoxies [3], [4].

In 2016, a study by Tetjana Tomaskova et. al tested the effect of aluminium oxide nanoparticles (Al2O3) on the mechanical and physical properties of epoxy resin. Various weight percentages of 2, 4,

6, 8, and 10 wt. % were added to epoxy resin. The steady-state method, Dielectric strength tests, and tensile tests were applied to study the physical and mechanical properties of epoxy/Al2O3 Nano-composite. The results revealed an enhancement in the thermal and electrical properties of epoxy resin after the addition of Al2O3 nanoparticles. The highest increase in thermal conductivity and dielectric strength was achieved by 8 wt. % of nanoparticles [6].

In 2017, Ganiu B. et. al studied the effect of reduced graphene oxide (RGO) on the thermal and mechanical properties of the epoxy matrix. Different thermal and mechanical behaviors of the epoxy Nano-composites were investigated. The result revealed an outstanding increase in the thermal conductivity when a small amount of RGO was added. An enhancement of 40% compared with that of the unmodified epoxy polymer was obtained by adding a value of 0.06 wt. % of RGO. This value represents one of the highest increases in the thermal conductivity per wt. % when adding RGO ever reported [7].

In 2019, a study was performed by Tagreed M. Al-Saadi et.al to show the use of multi-walled carbon nanotubes (MWCNTs) for the strengthening of epoxy resin with different MWCNTs wt. % (0.0, 0.02, 0.04, and 0.06) to fabricate the epoxy/MWCNTs nanocomposites. This research observed the effect of MWCNTs on the mechanical, electrical, and thermal properties of the epoxy matrix. Hardness, electrical properties such as dielectric constant, dielectric loss factor, dielectric strength, electrical conductivity, and thermal properties such as thermal conductivity were studied. The results revealed an enhancement in hardness, thermal and electrical conductivity, and break down strength with increasing MWCNTs wt. %. However, the dielectric loss factor and dielectric constant are decreased when the concentration of MWCNTs increased [8].

In this work, aluminium oxide (Al_2O_3), multi-wall carbon nanotubes (MWCNTs), and reduced graphene oxide (RGO) were dispersed in an epoxy matrix to study their effect on the physical property of epoxy resin. These three Nano-particles were chosen because they showed an enhancement in the physical and mechanical properties of the epoxy matrix [6] [8].

2. Experimental Work

2.1. Material

Epoxy Nanocomposites samples were made using biresin® two parts matrix; part A CR82 (resin) and part B CR80-6 (hardener). Three different nanoparticle materials were added to the epoxy matrix are listed in Table 1.

Table 1 summarize different types and characteristics of nanoparticle used

Nanoparticles	Characteristics
Aluminum oxide (Al_2O_3) by Sky Spring Nanomaterials Co.	White powder of Gamma- Al_2O_3 diameter range (0.4 to 1.5) Nano-meter.
Multi-walled carbon nanotubes (MWCNT). It was purchased from the Egyptian Petroleum Research Institute.	MWCNT's were formed by a high-yield catalytic process based on chemical vapor deposition (CVD) with an outer mean diameter of (8-10 nm) and inner mean diameter (4nm) and length from (5-10 μm). The purity of neat MWCNTs was greater than 90%.
Reduced graphene oxide (RGO) Three different types of reduced graphene oxide used in this study were (G24N, G33C, and G270). They were purchased from the City of Scientific	**Sample G24N** is an N-doped reduced graphene oxide with 3D structure. Its atomic composition is 83.3% C, 13.9% O, and 2.8% N. It is synthesized by thermal dissociation of polyethylene tetra-phthalate (PET) waste bottles with urea at 800˚C for 5 hr. **Sample G33C**. It was prepared as sample 9C as described in reference [9], [10] but not in the same batch. Its atomic composition is 90.49% C, 2.131% H, and 7.379% O. **Sample G270**. It is an N-doped multi-layered graphene Nano-sheet. Its atomic composition is 89% C, 7.2% O and 3.8% N. The graphene

Research and Technological Applications in Alexandria	Nano-sheets are well exfoliated. The doped N has dominated pyloric conformation followed by pyrdinic and lastly graphitic. It is synthesized by hydrothermal treatment of glucose solution under mild synthesis conditions.

2.2. Specimen Preparation

2.2.1. Sonication process. Epoxy resin (50g) was mixed with the three different types of nanoparticles (Al_2O_3, RGO, and MWCNTs) each type was added at a specified weight percent (1%, 0.3%, and 0.5% respectively) in epoxy resin after stirring for (5 min) at room temperature. The nanoparticles were well dispersed in epoxy resin by using "SONICS VCX750" sonicator for a constant time (1.5hr.) with (9kHz) frequency and (750W) power.

2.2.2. Production of nanocomposites sheet. Firstly, about (27) wt. % of hardener was added to the mixture of (50g) epoxy and nanoparticles. Then using a well-waxed glass sheet with a double-faced tape border; the epoxy/nanoparticle mixture was rolled into a uniform layer. A carbon fiber fabric layer of (0.4mm) thickness was then rolled on the epoxy/ nanoparticle layer. Followed by a second layer of the epoxy/nanoparticle mixture. This process was repeated three times then a layer of foam was added to absorb the excessive resin used in the hand layup process. The whole glass was wrapped in a vacuum bag and sealed. Finally, the whole sheet was vacuumed with (-1) bar at (45℃) for (24hr.). The final sheet obtained contained four layers of carbon fiber fabric with (2.4mm) thickness.

2.3. Experimental methods

2.3.1. Fourier-Transform Infrared Spectroscopy (FTIR). The FTIR spectrometer "JASCO 4100" was used to characterize the neat epoxy and epoxy/nanoparticles. In this technique, two parameters such as the chemical bonds and molecular compositions can be obtained for each specimen. The FTIR showed a number of peaks for the tested specimens that are hard to be recognized. Therefore, All the FTIR analysis was accomplished on the basic constituents of each composite type. The wavelength obtained was in the range of 400 to 4000 cm-1 with an average resolution of 4 cm^{-1}. The peak profile was sharpened by background scans to improve the resolution and remove any spectrum noise. At a room temperature of 23±3°C and relative humidity of 50±10 % according to the ASTM D-3039-06 [11], the investigated materials are prepared.

2.3.2. Optical Test (*Spectrophotometer (JASCO 670)).* A double beam Spectrophotometer (V-670) was used to obtain the optical behavior of the samples by measuring the amount of electromagnetic radiation transferred through a sample as a function of wavelength. The wavelength ranged from 200-2700nm. All samples were extracted from the fabricated sheet in a square of dimension (10 mm x10 mm).

2.3.3. Thermal Test (Photoacoustic spectroscopy (PA)). Thermophysical properties (Thermal diffusivity, Thermal effusivity, and Thermal conductivity) of each sample were determined by a laser photoacoustic spectroscopy (Stabilite 2017 model 2550) as Figure 1. Moreover, the output beam was subjected to a sample dimension (10x10) mm that was attentively mounted inside PA cell.

2.3.4. Thermal diffusivity (α). It is a thermos-physical parameter that determines how adequately photons transfer heat through the sample. Also, it indicates the rate of heat distribution inside the material. This rate depends mainly on the thermal conductivity (k) of the material and the energy stored inside the material [12];

$$\alpha = {}^{k}/_{\rho c} \quad (1)$$

Where: (c) is the specific heat capacity and (ρ) is the density of the sample.

Thermal diffusivity is experimentally determined by two commonly used methods [13]; the first method is known as the transient heat-flow method [14] and the second method is known as the periodic heat-flow method [15]. The PA method used in this research is related to the second method. Moreover, to determine the thermal diffusivity (α), the PA signal is obtained for various chopping frequencies.

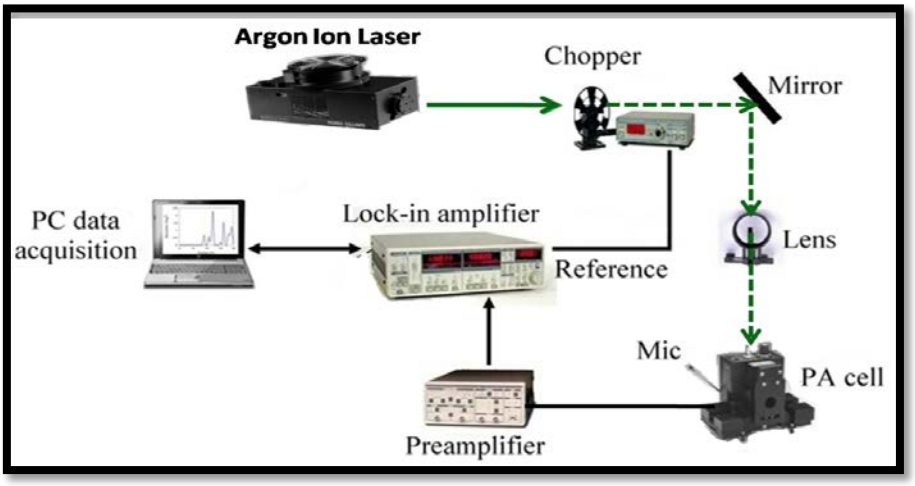

Figure 1 Photoacoustic spectroscopy

2.3.5. Characteristic frequency (fc). It is defined as the critical frequency at which the sample passes from the thermally thin region to the thermally thick region [16]. The thermal diffusivity is computed from the characteristic frequency. The PA signal varies with the thickness and the optical absorption of the sample. The characteristic frequency is obtained by plotting (ln PA) amplitude versus (ln f) for neat epoxy and epoxy/nanoparticles. As an example, Epoxy/G33C Nano-composite is drawn in Figure 2.

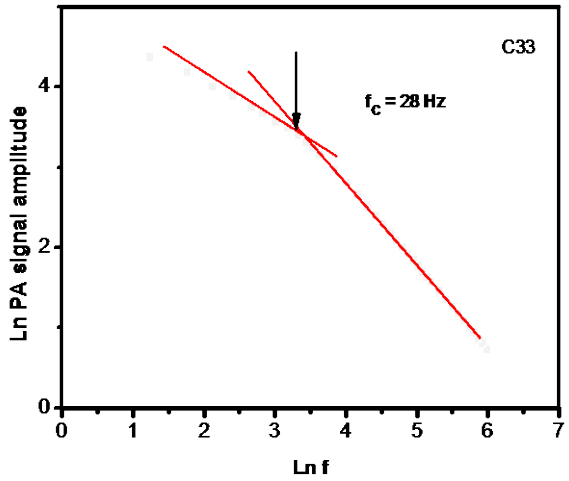

Figure 2 Characteristic frequency (f_c) of Epoxy/ G33C nanocomposites

The marked change in the slope is where the characteristic frequency (f_c) crossover takes place at which the sample changes from being thermally thin to thermally thick. The thermal diffusivity (α) was then estimated by using this equation [17].

$$\alpha = f_c l^2 \quad (2)$$

Where: (f_c) is the characteristic frequency and (l) is the sample thickness. The values of thermal diffusivity (α) are calculated and written in Table 2 for the neat epoxy and epoxy Nano-composites samples. From Table 2, the diffusivity (α) value for epoxy/carbon fiber (neat epoxy) is (2.4×10^{-7}) m^2s^{-1} which is much greater than pure epoxy of (0.5×10^{-7}) m^2s^{-1} [18]. Also, the highest thermal diffusivity is obtained by epoxy/MWCNTs of (3.1×10^{-7}) m^2s^{-1}.

2.3.6. Thermal effusivity (e). It is an important thermal parameter that indicates the surface heating of the substances. The amplitude of the PA signal "q" in case of thermally thick and optically opaque samples is given by:

$$\mathbf{q} = {}^{\mathbf{B}}/_{\mathbf{ef}} \tag{3}$$

$$\mathbf{B} = \frac{\mathbf{I_o}}{\mathbf{2}} \frac{\boldsymbol{\gamma} \mathbf{P_o} \boldsymbol{\alpha}_{\mathbf{g}}^{1/2}}{\mathbf{2\pi l_g T_o}} \tag{4}$$

Where: (I_o) is the incident light intensity, (γ) is the ratio of specific heats, (P_0) is the ambient pressure, (α_g) is the gas thermal diffusivity, (l_g) is the length of the gas column, (T_o) is the ambient temperature.

The thermal effusivity is obtained by Equation 5 of the sample and the modulation frequency (f) [19].

$$\mathbf{e} = \sqrt{\mathbf{k\rho c}} \tag{5}$$

The different values of the thermal effusivity of neat epoxy and epoxy/Nano-particles are obtained by linear fitting the relation between PA amplitude (q) and (1/f) as shown in Figure 3.

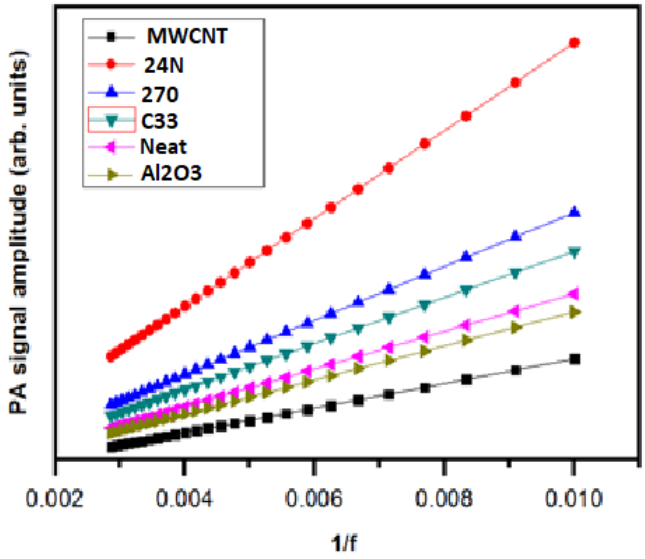

Figure 3 PA spectra for epoxy and epoxy/nanoparticles

2.3.7. Electrical Test. The samples' electrical resistances were measured by (Keithley 2635A) System Source Meter. Each sample was cut into a small sheet of (10x10) mm as per ASTM D-7264-15. The samples were subjected to a current of (1mA) and a voltage of (20V). The clamps were connected to positive and negative electrical current. The samples were placed between the clamps that contain a subjected circular area of (12.57mm^2). The electrical conductivity was calculated by Equation 6

$$\boldsymbol{\sigma} = \frac{\mathbf{L}}{\mathbf{R} * \mathbf{A}} \tag{6}$$

Where (σ) is the electrical conductivity in S/cm, (L) is the thickness in cm, (R) is the resistance in ohms, and (A) is the subjected area in cm2.

2.4. Results and Discussion.

2.4.1. Fourier Transform Infra-Red (FTIR) results. The FTIR analysis was used to investigate the chemical compositions and the functional groups obtained after the addition of nanoparticles to neat epoxy. The infra-red radiation causes stretching and bending of different bonds. These Bending bonds require high energies such as the part of the fingerprint region (1000-500) cm^{-1}. The other functional groups are characterized by their stretching frequencies. For example, C=O shows a peak at 1700 cm^{-1}

but for a C-H sp3 displays a peak at 3000 cm^{-1} [20]. The FTIR results depend on two parameters the wavenumber and the absorption intensity variation (abs).

From Figure 4 and Figure 5, FTIR results revealed that after the addition of nanoparticles, there was no change in the epoxy fingerprint region which means that the chemical structure of the epoxy remained the same. Moreover, in the epoxy matrix, there was no loss of functional groups, even after the addition of nanoparticles. This proves that the reactive sites of the nanocomposites are preserved for future manipulation. In addition, new bonds of (N-H) and (NH_2) appeared for epoxy/G24N and epoxy/ G270 nanocomposites as a consequence of using nitrogen doping to reduce the oxygen inside the graphene. Finally, it can be stated that there was a physical change not a chemical change after the addition of nanoparticles.

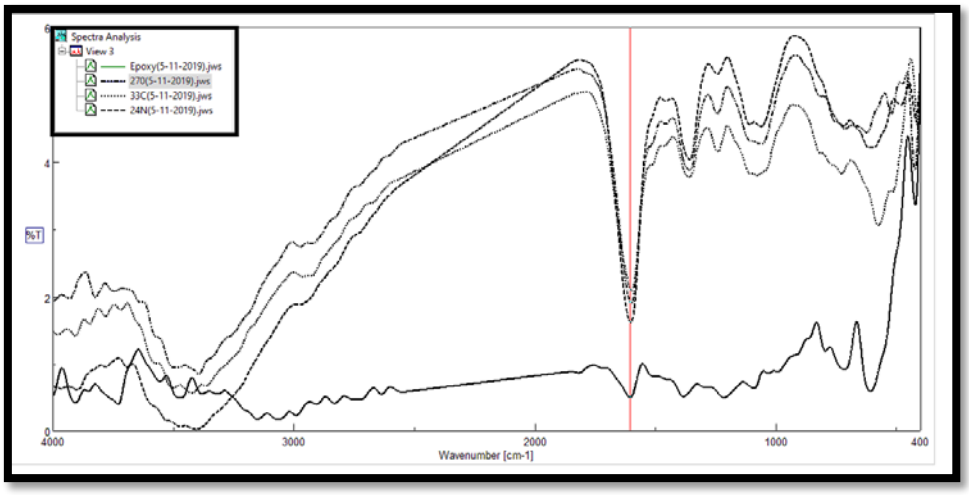

Figure 4 FTIR Results neat epoxy vs epoxy/RGO

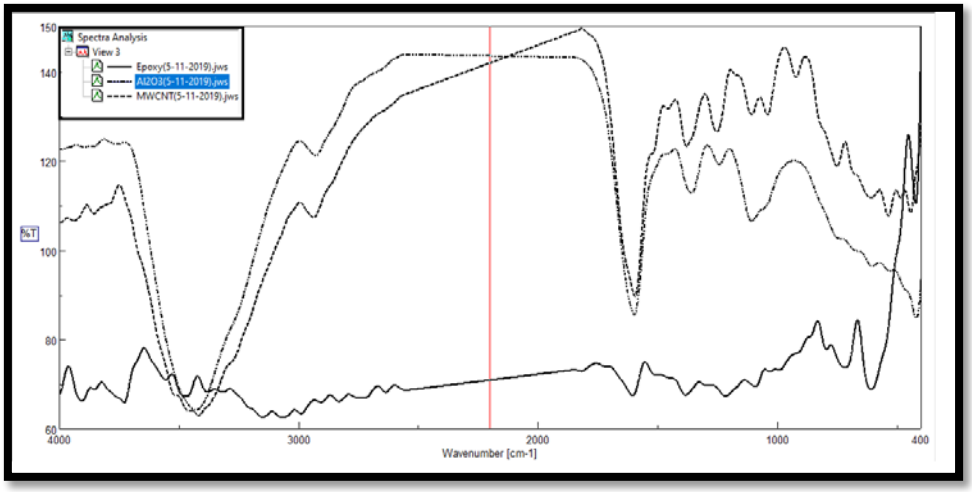

Figure 5 FTIR Results neat epoxy vs epoxy/MWCNT and epoxy/Al_2O_3

2.4.2. Optical test results. The optical absorption spectra of neat epoxy and epoxy/nanoparticles were obtained by regular UV-Vis. The range of wavelength was from (200-2700) nm to show the different types of light absorbed by each sample such as Ultraviolet (200-400) nm, visible light (400-700) nm, near-infrared (700-1100) nm, Infrared light (1100-2700) nm. As shown in Figure 6, it is easily observed that the epoxy matrix and all nanocomposite samples absorption ranges were in the infrared wavelength range. This is because of the opaque nature of the epoxy matrix. The absorption range for neat epoxy, epoxy/Al_2O_3, and epoxy/G270 Nano-composites laid at an absorption

peak rang of (1800-2700) nm. This is because of the presence of oxygen and nitrogen that causes polarity charges on the sample surface which facilitate the movement of the infrared light [23], [24]. But epoxy/G33C and epoxy/MWCNTs showed a higher wavelength peak at (2200) nm because the charges on the surface are equivalent. The highest wavelength peak was obtained by G24N where it was observed at (2350) nm. This could be due to the small amount of doped Nitrogen used to reduce the graphene oxide as mention in **Table 1**.

Table 2 FTIR peaks indicating bonds for all samples

Bond	Wavelength (cm^{-1}) [21], [22]	Samples					
		Epoxy	MWCNT	Al2O3	G33C	G24N	G270
Finger print	~<1000	421.37 - 715.461	442.583 - 921.807	419.442 - 603.61	410.763 - 727.996	419.442 - 718.354	424.263 - 622.895
sp^3 C-O	1025-1200	-	1040.41	1106.94	1077.05	1061.62	1060.66
sp^2 C-O	1200	1215.9	1253.5	1241.93	1240.97	1242.9	1242.9
Sp^3 C-H	2850-3000	-	2937.06	2929.34	-	-	-
Sp^2 C-H	3000-3100	3151.11	-	-	-	-	-
C=C-H	1607- 1510	1605.45	1594.84	1598.7	1598.7	-	-
CH2-CH2	1450-1470	-	-1457.92	-	-	-	-
CH2-CH3	1360-1390	-	1378.85	1359.57	1359.57	1357.64	1357.64
NH2	1590-1650	-	-	-	-	1601.59	1599.66
N-H	3350-3500	-	-	-	-	3406.64	3394.1
O-H	3200-3900	3728.69	3417.24 - 3938.89	3428.81 - 3895.5	3423.99 - 3907.07	-	3824.15

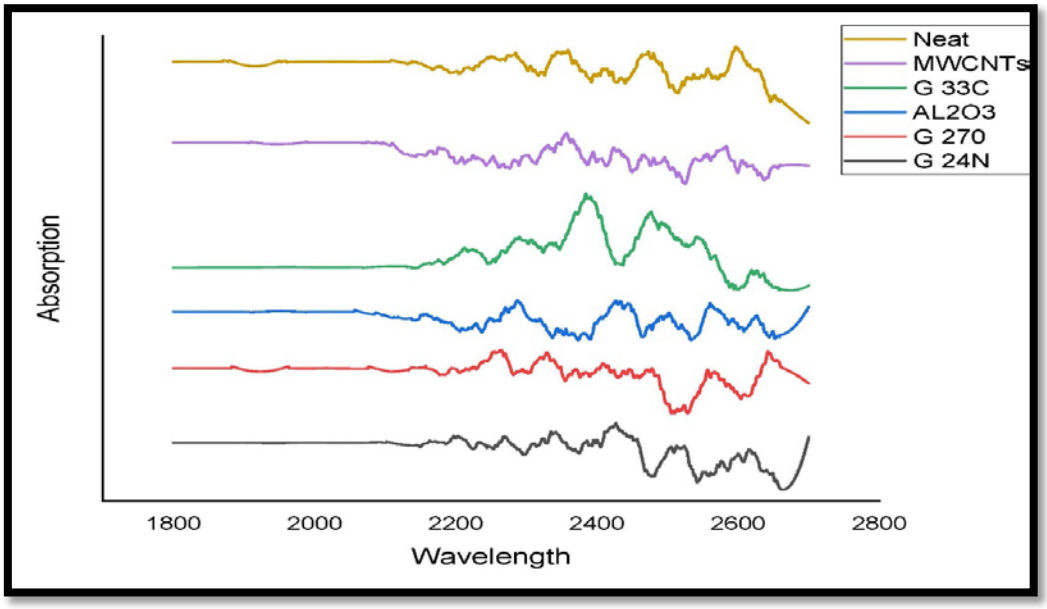

Figure 6 optical test for neat epoxy and epoxy nanocomposite

The absorption spectra shown in Figure 6 varies as a function of the additive type in the wavelength discussed above. It is clear that the neat epoxy absorption value is greater than the remaining nanocomposite samples. In addition, it is noticed that the epoxy/MWCNT nanocomposite has the greatest value among nanocomposites. While the epoxy/G33C has the highest value among the different RGO samples, which indicate about 50% of the neat epoxy absorption value. The absorption values of the remaining RGO samples (epoxy/G270, epoxy/G24) were decreased to approximately (25%, 10%)

of the neat epoxy, respectively. Moreover, the epoxy/AL2O3 nanocomposite shows a low absorption value around 40% of the neat epoxy. Thus, it may reveal that the absorption value varies from the value of neat epoxy regarding the additive chemical structure.

2.5. Thermal test results

The thermal properties of the neat epoxy and epoxy nanocomposites have been investigated by Photo Acoustic (PA) technique, which is of great importance for space application [25]. The sample's dimension (10x10) mm was mounted carefully inside the PA cell. Based on the depth profile analysis, the PA signal amplitude was obtained at various chopping frequencies (f) for each sample. The values of thermal properties are summarized in Table 3.

Table 3 Thermal properties of Neat epoxy, Epoxy/nanoparticles

Samples	Thermal diffusivity (α) ($10^{-7}m^2/s$)	STD	Thermal effusivity (e) ($Ws^{1/2}m^{-2}K^{-1}$)	STD	Thermal Conductivity (k) (W/m.k)	STD
Neat	2.4	±0.12	1080.8	±54	0.52	±0.03
Al2O3	2.85	±0.14	950.60	±48	0.50	±0.025
MWCNT	3.1	±0.15	1141.89	±57	0.63	±0.03
G24N	1.1	±0.06	581.35	±29	0.19	±0.01
G270	1.28	±0.06	700.37	±35	0.25	±0.012
G 33C	1.3	±0.06	793	±40	0.28	±0.014

As presented in Table 3, the thermal effusivity of neat epoxy is increased by the addition of MWCNTs nanoparticles. However, it decreased by Al2O3 was added and reduced graphene oxide nanoparticles (G270, G33C, and G24N)

2.5.1. Thermal conductivity (k). It is a thermal parameter that can indicate the ability of the material to conduct heat. It is obtained by Equation 7 that relates to the thermal effusivity and diffusivity [17].

$$k = e\sqrt{\alpha} \tag{7}$$

Thermal conductivity was calculated by using the determined values from (e), (α) and listed in Table 3. Thermal conductivity of CF/epoxy (neat epoxy) was increased to 0.52 W m-1 K-1 compared to the thermal conductivity of the epoxy matrix of 0.12 Wm-1K-1. Moreover, as expected [8], the highest increase in thermal conductivity was obtained by epoxy/MWCNTs nanocomposite by 83% compared to neat epoxy. This is because of the presence of the volume of the new phase with high thermal conductivity, which increases the heat flow [26]. However, for reduced graphene oxide (G33C, G270, G24N) nanoparticles the thermal conductivity decreased by (54%, 48% 37%) compared with neat epoxy. As known in epoxy/RGO nanocomposite, the thermal conductivity of a material depends on the interfacial resistance between the RGO graphene and epoxy matrix. When this interfacial resistance is low, the epoxy matrix creates an interfacial layer with nanoparticles. This layer reduces the heat flow inside the epoxy matrix causing the reduction of thermal conductivity [27], [28]. Moreover, the thermal conductivity decreased for epoxy/Al2O3, which could be a result of small agglomeration that reduces the heat flow.

2.6. Electrical test results

Electrical resistivity is a measurement of the resistance of a given size of a specific material to electrical conductivity. Electrical resistivity for each sample was measured by (Keithley 2635A) system source meter. Table 4 shows the electrical conductivity calculated by

$$\boldsymbol{\sigma = \frac{L}{R*A}} \tag{8}$$

Where (σ) is the electrical conductivity S/cm, (L) is the thickness in cm, (R) is the resistivity in ohms, and (A) subjected area = 0.125664 cm^2.

Table 4 Electrical conductivity of Neat epoxy, Epoxy/nanoparticles

Sample	Resistivity Ω	Electrical conductivity S/cm
Neat	$0.2076x10^{9}$	$8.69x10^{-9}$
MWCNT	7433	$2.57x10^{-4}$
Al2O3	633	$3x10^{-3}$
G-270	669	$3.09x10^{-3}$
G-24N	1047	$1.84x10^{-3}$
G-33C	957	$2.08x10^{-3}$

The electric conductivity of the epoxy matrix increased after nanoparticles were added and changed from an insulating material into a semi-conductive material [29]. The greatest increase in electrical conductivity was obtained by epoxy/G-270 nanocomposites relative to neat epoxy. The reason behind this improvement is the reduction of the graphene oxide because as the oxygen decreases the electrical conductivity increases [30]. In addition, as mentioned in FTIR results, new bond such as (N-H) was formed at different peaks at 3394.1 cm^{-1} and 3429.78 cm^{-1}. This new bond increases the flexibility of the epoxy matrix and facilitates the movement of electrons inside the epoxy matrix.

3. Conclusion

In this article, the electrical, optical, and thermal properties of carbon fiber/epoxy composite were enhanced by using various types of Nano-additives. The effect of adding nanoparticles in carbon fiber/epoxy (neat epoxy) composite has been investigated to prove the ability of the materials to sustain the space working conditions.

The optical test showed that the neat epoxy and epoxy/nanoparticles absorption spectra were in the infrared wavelength range of (1100-2700) nm. The highest absorption peak was obtained by Epoxy/G24N at 2350 nm.

The thermal test indicated that the three thermos-physical parameters; diffusivity, effusivity, and thermal conductivity were enhanced after the addition of MWCNTs. Moreover, thermal conductivity of carbon fiber/epoxy (neat epoxy) was enhanced to 0.52 $Wm^{-1}K^{-1}$ in compared with 0.12 $Wm^{-1}K^{-1}$ of neat epoxy. However, adding of Reduce Graphene Oxide (RGO) decreased the thermal conductivity of neat epoxy.

The electrical test revealed that after the addition of nanoparticles neat epoxy changed from an insulating material to a semi-conductive material. The highest increase in electrical conductivity was achieved by Epoxy/G270.

This means that adding nanoparticles increases the physical and electrical properties of carbon fiber/epoxy. In addition, the improvement was more precise for its optical and electrical properties than its thermal property where more research in this field should be taken into consideration.

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