Research Article

A Comparative Study on Structural, Morphological, and Tensile Properties of Binary and Ternary Epoxy Resin-Based Polymer Nanocomposites

Dalia M. T. Mustafa,¹ Sarkawt Rostam ¹ and Shujahadeen B. Aziz ^{2,3}

¹Department of Mechanical Engineering/Production, College of Engineering, Sulaimani Polytechnic University, Sulaimani, Kurdistan, Iraq

²Advanced Polymeric Materials Research Lab., Department of Physics, College of Science, University of Sulaimani, Qlyasan Street, Sulaimani, Kurdistan, Iraq

³Department of Civil Engineering, College of Engineering, Komar University of Science and Technology, Sulaimani 46001, Kurdistan, Iraq

Correspondence should be addressed to Shujahadeen B. Aziz; shujaadeen78@yahoo.com

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In the present work, epoxy resin-based nanocomposites (NCPs) were fabricated with improved mechanical properties. The epoxy resin (EPR) was mixed with differing amounts of calcium titanate (CaTiO_3) nanoparticles (NPs) and glass fiber. The results showed that the ternary system contained glass fiber exhibits low mechanical performance compared to binary [EPR: CaTiO_3] system. The effect of fiber glass and NPs on the epoxy resin mechanical behavior was determined by conducting a tensile test for various specimen sets. From the mechanical characterizations, it was seen that there is a monotonic relationship between the NPs concentration and Young's modulus. Additionally, NCPs samples were brittle in nature and the strain rate significantly decreased upon the addition of CaTiO_3 concentration; while the tensile strength was increased. From the X-ray diffraction evaluation, it can be concluded that the addition of NPs have a great impact on the epoxy structure. Besides, the morphology appearance was in good agreement with structural and mechanical analysis.

1. Introduction

Recently, structural adhesives, coating compounds, composite matrices, coatings, and other technical applications make extensive use of epoxy resins. When it comes to automotive and aerospace industries and similar applications, composite structures are increasingly being manufactured with the help of resin transfer molding [1]. In aerospace military applications weight is a crucial point; thus, composite materials are considerably helpful since they have better specific stiffness (stiffness to weight) and specific strength (strength to weight) compared to conventional materials. Recent structural applications demand novel materials; therefore, nanomaterials and polymer composites have been widely investigated and developed [2]. There is

also an escalating need for materials having high exceptional mechanical properties since polymer and its composites are being greatly used [3]. In terms of the novel composite materials, which are NCPs, they have a constituent whose dimension hails between 1 and 100 nm. Molecular scale interactions give material enhancements to nanocomposite materials, which influences material and physical parameters at scales unreachable with conventional filler materials [1, 4]. Polymers have been getting reinforcements in the form of organic or inorganic fillers for tens of years. Recently, polymeric NCPs are getting increasingly manufactured with the help of NPs as reinforcements. Nanofillers (NFs) will have different properties if mixed with matrix properties. Researchers are conflicted on whether matrix properties are enhanced or there is a negative impact due to

the addition of NPs [5, 6]. As expected, natural or artificial inorganic compounds were present in polymeric materials for many reasons such as reducing its price. Otherwise, its properties such as electrical conductivity and permeability for gases such as oxygen or water vapor are decreased. Alternatively, other properties that include heat resistance, impact resistance, and mechanical strength are improved. Plate-formed particles (such as mica), fibers (such as fiberglass), and materials (such as calcium carbonate) existing in the particle form are known as traditional fillers [5, 7]. Numerous works have been published to enhance the toughness of epoxy composites. Inorganic nanoparticles, thermoplastic materials, rubbers, and other kinds of fillers having varied chemical natures are served as a third component to the epoxy systems as they were mixed. Because of this, the thermal and mechanical properties were enhanced to a great extent [8]. The impacts concerning NPs such as TiO_2 [9, 10], ZnO [11], clay [12], multiwalled carbon nanotube (MWCNT) [13, 14], SiO_2 [15], and Al_2O_3 [16] have been investigated in a number of research studies. Previous studies revealed that various factors affecting the mechanical properties of materials [17–20]. The fatigue crack propagation and statistical distribution of microcracks are well studied by Qian et al., [17, 18]. The development of He implantation-induced imperfections and dissimilar irradiation temperatures are also reported as factors that affect the materials properties [19, 20]. By looking at the literature both intensively and extensively, it can be seen that numerous industrial applications (particularly, electronic applications) have deeply utilized the epoxy resins due to their superior fatigue life and above-average specific strength/stiffness. With that being said, electrical and thermal conductivity, thermal expansion, and mechanical properties, with manifold functions necessitate more enhancements for some high-level applications [21].

As of late, researchers could be seen becoming increasingly interested in polymer nanocomposite creation. Accordingly, this research is aimed at investigating the mechanical properties concerning epoxy resin incorporated with CaTiO_3 nanoparticles and glass fiber.

2. Experimental Methodology

2.1. Materials. The Don Construction Products (DCP) company was consulted for purchasing the quick mast 105 epoxy resin. The purchased epoxy had greater than or equal to 25 MPa tensile strength, greater than or equal to 45 MPa flexural strength, greater than or equal to 70 MPa compressive strength at a week with 25°C temperature, around 50–70 min of pot life with 25°C temperature, VOC smaller than 20 G/liter, 5° lowest application temperature, 3–5 poise viscosity with 25°C temperature while 1–2 poise with 35°C temperature, and 1.1 ± 0.05 density. The CaTiO_3 nanoparticles (purity 99.9%) with a diameter of 30 nm were used with the epoxy resin to prepare polymer nanocomposites. The chopped fiberglass was obtained from commercial sources. The chopped glass fiber was supplied by Dost Kimya (DK) company and with an application temperature limit of -60°C to 650°C , specific gravity 2.60 gr/cc, and fiber diameter of 13–15 microns.

2.2. Equipment. Nanoparticles, fiberglass, hardener, epoxy, and other such materials underwent the weighing process with the help of the digital balance scale (Sartorius-German origin). The epoxy composite acquired homogeneous dispersion with the help of BOECO Germany's magnetic stirrer that could run at 1500 rpm at best. The furnace KAROL type alongside a thermostat was used to heat the specimens. Afterward, the specimens were tested with the help of a Cussons technology universal testing machine of 100 kN maximum load. At room temperature and 5 mm/min crosshead speed, the machine was used for tensile testing according to the ASTM D638 standard (Figure 1).

2.3. Fabrication of Nanocomposites. The filler/epoxy matrix NCPs were fabricated with the help of a facile method. Beakers that had magnetic stirrer bars were used for sample preparation. A 2:1 ratio was considered for fixing the hardener and epoxy resin mixtures. Three sets were prepared: (1) the magnetic stirrer was used to prepare pure epoxy; (2) epoxy with nanoparticles; and (3) nanoparticles, chopped fiberglass, and epoxy. The magnetic stirrer was used for carrying out the mixing method with a speed of 750 rpm and 1500 rpm in the first and final steps, respectively. The diamine curing agent and epoxy resin were kept in the beakers. Afterward, epoxy resin NCPs were prepared by adding CaTiO_3 nanopowder in different amounts. 1, 3, 5, and 7 wt.% were the amounts of the nanocomposites' filler. Then, the epoxy resin lost some of its viscosity through heating so that the agglomerations do not form. A reduced pressure was considered for degassing overnight.

Appropriate molds were used for containing mixtures and ambient conditions were considered for conducting the curing process that lasted a week alongside a postcuring process for 7 hours with 95–100°C temperature. With the utilization of a laser CNC cutting machine, acrylic was used for making the mold. A lubricant was utilized for simply removing the mixture of epoxy from the mold after curing. The research also involves studying the fiber-reinforced epoxy resin polymer NCPs. The epoxy was mixed with the chopped fiberglass with (5–10) min of stirring and heating at (95–100)°C. Afterward, there was the addition of nanoparticles (with 5–10 min of stirring) and hardener (with 2–3 min of stirring). Normally, epoxy-based composites are cured with the help of primary amines. Furthermore, there can be dissimilar properties in epoxy resins upon combining and curing them using different curing agents. The amine's loading amount and type determine the curing speed. Some of the curing agents include aliphatic and aromatic compounds [22–25]. Under the room temperature and 5 mm/min crosshead speed, there was a tensile test for determining the mechanical properties concerning the nanoreinforced epoxy. The ASTM D638 standard was considered for conducting the tests of these specimens. Figure 2 schematically presents the curing procedure. For the binary system which consists of pure epoxy resin and nanocomposites, the samples were coded as EPR 0 and $\text{EPR} + x\text{CaTiO}_3$ ($0.01 \leq x \leq 0.07$). For the ternary samples, the systems were coded as EPRGF0, EPRGF1, EPRGF3, EPRGF5, and

FIGURE 1: Dimensions of tensile specimen according to ASTM D638 standard.

FIGURE 2: Schematic representation of the curing process of nanocomposites.

EPRGF7, and for Epoxy resin (EPR), 0.01 wt.% and 0.02 wt.% fiberglass (GF) incorporated with 0 wt.%, 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.% of CaTiO_3 , respectively.

3. Results and Discussion

3.1. XRD Analysis. The XRD pattern of the pure epoxy resin is shown in Figure 3. According to a variety of reports, the XRD evaluation can help with observing the NPs dispersion and the extent to which they exfoliate/intercalate within the epoxy resin matrix [21]. Earlier research studies are shown that there are wide peaks in pure epoxy resins. Each pattern demonstrated broad diffraction from $5\text{--}80^\circ$ alongside a couple of maxima close to $20\text{--}45^\circ$ as the cured epoxy

network scattered and its amorphous nature revealed [8, 21]. Nevertheless, when the epoxy matrix experienced CaTiO_3 dispersion, the XRD pattern of epoxy NCPs demonstrated the epoxy diffraction peaks with poor intensities. Additionally, the enhanced sharp diffraction peaks of CaTiO_3 with improved intensities were detected, as shown in Figures 3(b) and 3(c). This is not the case with epoxy XRD pattern particularly when the CaTiO_3 nanoparticles are highly concentrated. According to prior research studies, it is true that the attraction between surface groups of nanofiller (NF) based on Coulomb and van der Waals forces is responsible for aggregating NPs with high concentration [24, 25]. The maximum intensity for pure epoxy resin is about 250a. While it reduces to around 100 a.u at 3 wt.% of

FIGURE 3: XRD pattern for (a) pure epoxy resin (EPR0), (b) EPR + 3 wt.% CaTiO_3 , and (c) EPR + 7 wt.% CaTiO_3 .

CaTiO_3 and finally becomes around 80 a.u at 7 wt.% of CaTiO_3 . Some sharp peaks due to CaTiO_3 especially at 7 wt.% of CaTiO_3 NPs have appeared which can be attributable to the high content of NF. This makes the sample more rigid and thus affecting the strain rate as can be seen in a later section. The results of the present work show that it is necessary to conduct the structural study to understand the physicochemical properties of polymer composites which is a multidisciplinary field.

The surface morphology of the samples is shown in Figures 4(a)–4(e). It is displayed that pure epoxy resin (see Figure 4(a)) has many pores. These pores are not desired for mechanical properties. On the other hand, with increasing CaTiO_3 NPs the sizes of pores are reduced and at higher concentration of the filler, most of the pores are disappeared. The mechanical properties as can be seen in the later section may give more information regarding the effect of structure and morphology on mechanical behavior.

3.2. Mechanical Properties. Figure 5 presents the curves that compare tensile stress to tensile strain for the cured epoxy and its NCPs. It is fascinating to note the stress-strain relationship that exists between pure epoxy and epoxy NCPs as there is an increase in the tensile stress as CaTiO_3 nanoparticles are added. In general, NCPs cause the tensile properties to enhance, which is not the case with neat proxy [21]. The literature reports that composites having different fillers have better tensile strengths [12–16]. It is interesting to note that at 7 wt.% of CaTiO_3 NPs the sample exhibits the maximum tensile strength. These results are supported by the XRD results presented in Figure 3 and morphology appearance. Looking at the literature, it can also be said that the dispersion and shape of particles in the matrix, interfacial adhesion, matrix material strength, and other such factors

determine the tensile strength of polymer composites [21]. As this research involves increased tensile strength, the stirring method with heating is indeed novel for NPs dispersion via the host epoxy resin as the viscosity is reduced. Accordingly, heating would be a good option for reducing the epoxy resin viscosity (as we have discussed earlier) as it results in the homogeneous dispersion of the filler particles. Upon loading the matrix, the NPs dispersion can withstand more tensile load of the matrix. Considering this, the composites can have better strength as the elastic deformation region is exceeded by the applied load since the load transfer is improved with the dispersed NPs [26]. There is an extension in the primary epoxy resin peaks with the increase in CaTiO_3 concentration (see the XRD results, Figure 3), i.e., the d-spacing can be increased with the polymer chains' galleries experiencing NPs diffusion [27, 28].

Another part of the current work is mixing NPs with epoxy resin: fiber glass composites as well. Prior research also aimed at showing the influence of silica NPs on the mechanical properties concerning the epoxy: fiber glass composites. In the Zheng et al. [15] study, two different conditions were considered for studying how SiO_2 NPs affect mechanical properties of epoxy resin: (1) mixing with epoxy and (2) mixing with epoxy and fiberglass. The driving force behind it was to show the impact of the NF particles on the mechanical properties. Figures 6 and 7 show the tensile stress-strain curve and morphology appearance for epoxy resin: 0.1 wt% fiberglass incorporated with various concentrations of CaTiO_3 nanoparticles. It can be seen that the tensile strength gets reduced to 16.0812 MPa as the fiberglass was mixed with epoxy. Although, the maximum tensile strength (with 5 wt.% CaTiO_3 and 42.93 MPa amount) was acquired by mixing the NPs having 0.2 wt.% fiberglass with epoxy. To the best of our knowledge, the fiberglass is impeding the homogeneous distribution of CaTiO_3 NPs.

FIGURE 4: The FESEM image for (a) pure epoxy resin (EPR0), (b) EPR + 1 wt.% CaTiO_3 , (c) EPR + 3 wt.% CaTiO_3 , (d) EPR + 5 wt.% CaTiO_3 , and (e) EPR + 7 wt.% CaTiO_3 .

Moreover, in the ternary system, the viscosity may increase and some bubbles may stay through the samples, and thus some pores may appear again. It is clear from Figure 8 that at high CaTiO_3 concentration some pore has appeared especially for 0.1 wt.% fiber glass. There is a strong relationship

between the matrix dispersion and composite's strength since prior research suggests the stress concentrations are introduced with the NPs agglomeration, which reduces the tensile strength for composites [21]. Figures 8 and 9 show the tensile stress-strain curve and morphology appearance for

FIGURE 5: Stress-strain curves for neat epoxy resin (EPR) and epoxy resin nanocomposites.

epoxy resin:0.2 wt.% fiber glass incorporated with various concentrations of CaTiO_3 NPs. In Figure 9, it can be seen that CaTiO_3 NPs are aggregated and some phase separation has appeared. These phenomena are responsible for a wide reduction in strength especially for 0.2 wt.% fiberglass with various concentrations of CaTiO_3 NPs.

The literature revealed that mixing fillers with epoxy resin causes the tensile strength to decrease, which could be due to different potential reasons such as the NCPs having flaws. Particles having poor boundaries and the bubbles trapped when the sample was being prepared are some of these flaws. As the volume fraction of the filler increased, there could be an increment in these flaws. The nonhomogeneity of the network density of the sample is another thing that could happen. The resulting samples would end up having heterogeneity because of this high viscosity [29].

There was a comparison research that dealt with how multiwalled carbon nanotubes (MWCNTs) and graphene NPs influence the physical and mechanical properties concerning epoxy nanocomposites and various loadings of fillers. From the outcomes of the experiment, it was found that epoxy nanocomposites with graphene NPs have slightly lower mechanical properties as opposed to the epoxy nanocomposites with MWCNT [14]. It has also been seen from scratch tests that the tensile tests of TiO_2 particles enable new NPs to be mixed with the epoxy composites through an ultrasonic method for NPs dispersion into the epoxy with 5, 10, 15, and 20 wt.% [10]. Research also showed that the SiC NPs improve the mechanical properties concerning epoxy resin by 20–30% on average with various loading particles of (1.5 to 3.0) wt.% of the resin and 1.5 wt.% of the loading [29]. A dynamic mechanical evaluation was used for arranging and characterizing epoxy/clay NCPs. By adding clay, it was acquired significantly increased storage modulus and modestly increased glass transition temperature [12].

Figures 10 and 11 present the elongations and force at breaks concerning the epoxy resin alongside 1 wt.% and 2

wt.% of fiber glass doped with different CaTiO_3 amounts. As the nanofiller is increased, there is a decrease in the elongation. The system becomes more brittle and the polymer less elongated as there is beyond a limit increase in the filler-polymer ratio [30]. From the engineering viewpoint, the rupture behavior of composite materials can be described with the key parameter that exists within elongation at break. Nevertheless, there is an inverse relationship between filler content and strain at break, as aforementioned [9]. A composite having high stiffness and strength is likely to result in decreasing elongation with increased nanocontent that NCPs demonstrate [31]. It can be seen that NF has a more positive impact on the mechanical properties of epoxy, whereas fiberglass affected the mechanical properties of epoxy negatively as opposed to those seen in the case of NF and epoxy only. The reason for this could be that fiber glasses with epoxy are not able to have a compact structure. According to the review paper by Pinto et al. [9], the dispersion of NFs through the epoxy resin can be achieved with the heating method and constant stirring. There was a significant enhancement in the mechanical properties of an epoxy resin inserted with NF (Figures 10–14). A previous study indicated that polymers and their composites form a very important class of engineering materials and are invariably used in mechanical components and their application in electronics is also wide [32, 33].

Table 1 presents the mechanical properties concerning the NCPs.

Nonetheless, elongation at break peaks with 1 wt.% CaTiO_3 , whereas it gets to the lowest value with 5 wt.% CaTiO_3 and 0.2 wt.% fiberglass, as it can be seen from Figure 8. The maximum force at break was 1893.5 N at 7wt.% of CaTiO_3 while it is reduced upon the addition of fiberglass, as it can be seen in Figure 11. This is since the fiberglass cannot constitute a compact structure with epoxy. From Figure 12, it can be seen that epoxy- CaTiO_3 nanocomposite achieved the highest ultimate tensile strength with 49.271 MPa at 7 wt.% of NPs and the lowest tensile strength with 25.95 MPa at 5 wt.% NPs and 0.2 wt.% fiberglass. The improved mechanical characteristics may have contributed owing to good interaction between the epoxy resin and CaTiO_3 NPs. The good interfacial tension between the epoxy resin and CaTiO_3 may have possibly attributed to a rise in the values of the tensile strength. The maximum Young's modulus of elasticity (i.e., 2622.904 MPa) was achieved at 0.1 wt.% fiberglass and 7 wt.% CaTiO_3 , whereas the lowest one (i.e., 1654.747 MPa) was achieved at 0.2 wt.% fiberglass and 1 wt.% CaTiO_3 , as it can be seen in Figure 13. The robust adhesion between CaTiO_3 nanoparticles and epoxy resin may have resulted in the powerful load-bearing capacity of the films thus raising Young's Modulus while the fiberglass with epoxy is not able to have a compact structure. The fiberglass harms the mechanical properties of epoxy. The strain rate significantly decreased upon the addition of CaTiO_3 concentration. The maximum value of strain at break for NPs was 4.444% at 1 wt.% of CaTiO_3 as it can be seen in Figure 14.

FIGURE 6: Stress-strain curves for epoxy resin: 0.1% fiberglass: $x\text{CaTiO}_3$ ($0.01 \leq x \leq 0.07$) ternary composites.

FIGURE 7: The FESEM images of epoxy resin: 0.1% fiberglass: $x\text{CaTiO}_3$ ($0.01 \leq x \leq 0.07$) for (a) EPRGF 1 (b) EPRGF 2, (c) EPRGF 3, and (d) EPRGF 4.

FIGURE 8: Stress-strain curves for epoxy resin: 0.2% fiberglass: $x\text{CaTiO}_3$ ($0.01 \leq x \leq 0.07$) ternary composites.

(a)

(b)

(c)

(d)

FIGURE 9: The FESEM images of epoxy resin: 0.2% fiberglass: $x\text{CaTiO}_3$ ($0.01 \leq x \leq 0.07$) for (a) EPRGF 1 (b) EPRGF 2, (c) EPRGF 3, and (d) EPRGF 4.

FIGURE 10: Elongation at break versus nanoparticle concentration for all the samples.

FIGURE 11: Force at break versus nanoparticle concentration for all the samples.

TABLE 1: Mechanical property of epoxy-CaTiO₃ NCPs.

Strain at break (%)	Young's modulus (MPa)	Force at break (N)	Elongation at break (mm)	Ultimate tensile strength (MPa)	CaTiO ₃ Content wt (%)	Fiberglass content wt. %
4.444	2248.488	1701.7	2.222	47.564	1	0
3.340	2206.009	1598.0	1.670	42.207	3	0
2.949	2346.616	1778.3	1.474	46.274	5	0
3.246	2610.476	1893.5	1.623	49.271	7	0
3.348	2236.816	1742.7	1.674	45.42	1	0.1
3.586	2305.848	1776.6	1.793	46.41	3	0.1
2.454	2326.548	1610.6	1.227	41.91	5	0.1
2.442	2622.904	1612.7	1.221	41.97	7	0.1
3.811	1654.747	1139.3	1.906	30.52	1	0.2
2.816	2068.546	1572.4	1.408	40.91	3	0.2
1.228	2253.011	997.5	0.614	25.95	5	0.2
3.648	2300.663	1649.1	1.824	42.93	7	0.2

FIGURE 12: Ultimate tensile strength versus NPs concentration for all the samples.

FIGURE 13: Young's modulus versus NPs concentration for all the samples.

4. Conclusions

In the present study, the epoxy resin-based NCPs were prepared with different amounts of NPs and glass fiber. Several sets such as pure epoxy resin, epoxy with 0.1 and 0.2 wt. % fiberglass, and 1, 3, 5, and 7 wt.% of CaTiO₃ nanoparticles were prepared. Tensile test was performed for the different sets of specimens by using ASTM D638 and various parameters, such as Young's modulus, ultimate tensile strength, strain at break, elongation at break, and force at break, were determined. It was presented that highest ultimate tensile strength with 49.271 N/mm² was obtained at 7 wt.% of CaTiO₃. While, the maximum elongation at break was 2.222 mm at 1 wt.% of CaTiO₃. Furthermore, the highest value for Young's modulus of elasticity was 2622.904 MPa at 7 wt.% of CaTiO₃ and 0.1 wt.% of fiberglass. It was also shown that the maximum value of strain at break was 4.444% at 1 wt.% of CaTiO₃. Later, force at break with the highest value of 1893.5 N at 7 wt.% of CaTiO₃ was recorded. XRD

FIGURE 14: Strain at break versus NPs concentration for all the samples.

was also conducted to show how the addition of NPs significantly impacted on the epoxy structure. The FESEM images revealed a wide range of pores with various sizes for pure epoxy resin. The addition of CaTiO₃ filler reduced the number of pores.

Data Availability

All the data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare no conflicts of interest.

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