

Hybridizing MWCNT with nano metal oxides and TiO₂ in epoxy composites: Influence on mechanical and thermal performances

Masoomeh Gazderazi, Masoud Jamshidi

Polymer Research Lab, School of Chemical Engineering, Iran University of Science and Technology (IUST), Tehran, Iran

Correspondence to: M. Jamshidi (E-mail: mjamshidi@iust.ac.ir)

ABSTRACT: In this study, the effects of multi-walled carbon nanotubes (MWCNT), and its hybrids with iron oxide (Fe₂O₃) and copper oxide (CuO) nanoparticles on mechanical characteristics and thermal properties of epoxy binder was evaluated. Furthermore, simultaneous effects of using MWCNT with TiO₂ as pigment and CaCO₃ as filler for epoxy composites were determined. To investigate effects of nano- and micro-particles on epoxy matrix, the samples were evaluated by TGA and DTA. It was found that the hybrid of MWCNT with nano metal oxides caused considerable increment in the tensile and flexural properties of epoxy samples in comparison to the single MWCNT containing samples at the same filler contents. Significant improvement in the thermal conductivity of epoxy samples was obtained by using TiO₂ pigment along with MWCNT. The TiO₂ pigment also caused considerable improvement in mechanical properties of the epoxy matrix and the MWCNT containing nanocomposite. The best mechanical and thermal properties of epoxy nanocomposites were obtained at 1.5 wt % of MWCNT and 7 wt % of TiO₂ that it should be attributed to particle network forming of the particles which cause better nano/micro dispersion and properties. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 2016, 133, 43834.

KEYWORDS: composites; graphene and fullerenes; mechanical properties; nanotubes; thermal properties

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INTRODUCTION

The concept of incorporating nanoparticles into polymeric matrixes as polymer nanocomposites in order to improve their properties has received considerable attention, especially in the context of thermal and mechanical performances.^{1–4} Nanocomposites showed much better behavior and characteristics in comparison to conventional composites.^{5,6} Recently, Dianatdar and Jamshidi⁷ used nano and micro TiO₂ particles to improve mechanical properties of water-based acrylic polymers. They found that TiO₂ nanoparticles and its hybrid with TiO₂ microparticles have more significant effect on mechanical properties.

Polymers are usually used as insulator because of their high thermal resistance against heat transfer.⁸ Normally carbon nanotubes (CNT), metals, and metals oxide nanoparticles have been added to polymers to increase their thermal conductivity. However, these particles influence mechanical properties of polymer composites.^{9–13}

Nowadays, many researches have been done on the mechanical and physical characteristics of CNT-based nanocomposites.^{14,15} There are some researches on the effect of CNT, its functionalization, and dispersion on thermal conduction of polymers.^{16–19}

Mechanical characteristics of nanocomposites are generally dependent on the dispersion of nano- and microparticles in polymer matrix. So that when there is good dispersion of particles in polymer matrix, agglomerates formation are reduced and mechanical characteristics are enhanced. However, by increasing in nano- and microparticles in polymer, the agglomerates are produced and mechanical characteristics are decreased.^{20–22} Mechanical properties of epoxy nanocomposites reinforced with nanoexpanded graphene showed that the mechanical characteristics (fracture toughness, hardness, and Young's modulus) first increase and then decrease.²³

Mechanical characteristics of polyethylene composites reinforced with tungsten disulfide showed that increasing in reinforcement content caused increase in tensile modulus and yield strength and decrease in fracture strain and toughness of nanocomposites.²⁴

Roy *et al.*²⁵ studied mechanical and thermal properties of polyurethane nanocomposites reinforced with MWCNT and graphene oxide as hybrid. It was found that nanocomposites exhibit significant improvement in thermal stability and storage modulus.

In this research, different conductive nanoparticles (i.e., MWCNT, Fe₂O₃, and CuO) were used in single and hybrid

Table I. Physical Characteristics of the Used Nanoparticles

Nanoparticles	Diameter (nm)	Length (μm)	Thermal conductivity (W/mk)	Surface area (m^2/g)
CNT	Outer: 50–80	10–30	2000	200
	Inner: 10–30			
Fe_2O_3	20–30	—	5	30
CuO	50	—	20	80

forms as reinforcement of epoxy matrix. The mechanical and thermal behavior of the nanocomposites was studied using tensile, flexural, compression, and thermal conductivity tests. Effects of TiO_2 pigment and CaCO_3 filler on performance of epoxy matrix and epoxy nanocomposites were evaluated. Scanning electron microscopy (SEM) and thermal gravimetric analysis (TGA) were used to characterize microstructure and thermal stability of the nanocomposites, respectively.

EXPERIMENTAL

Materials

Nanoparticles. The nanomaterials used in this study were multi walled carbon nanotube (MWCNT), iron oxide (Fe_2O_3), and CuO nanoparticle. They were supplied from the NEUTRINO Company (China). The physical characteristics of the nanoparticles are shown in Table I. Nanoparticles were used in epoxy resin in single and hybrid forms.

Epoxy Resin and Additives. The utilized epoxy resin was reaction product of diglycidyl ether of Bisphenol-A which used aromatic di-amine as curing agent. Chemical composition of resin and hardener are shown in Figure 1. FTIR analysis of epoxy resin is shown in Figure 2.

Pigment and Extender. Titanium dioxide micro powder (TiO_2) was used as pigment in this research which was obtained from KRONOS Company (Germany). TiO_2 had an average particle size of 150–200 nm. Calcium carbonate powder (CaCO_3) was used as extender which was purchased from Polymer Concrete Company (Tehran, Iran). CaCO_3 had an average particle size less than 20 μm .

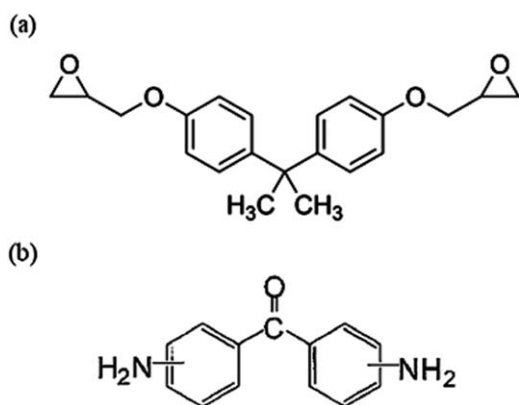


Figure 1. Chemical compositions, (a) diglycidyl ether of Bisphenol-A and (b) aromatic di-amine.

Sample Preparation

To produce epoxy nanocomposites, epoxy resin was diluted by xylene solvent. The nanoparticles at different contents were dispersed in diluted epoxy resin by sonication power of 80–120 W for 40–70 min. The suspension was placed in ice/water bath to decrease temperature during sonication. For evaporation of the solvent and bubble removing, the mixture was kept in a vacuum oven at 60 °C for 8 h. Thereafter, the mixture remained at room temperature for 3 days. Then, the hardener and antifoaming agent were added to the mixture and stirred at room temperature for 30–40 min (i.e., 100 parts epoxy resin, 50 parts curing agent, and 1 part defoaming by weight).

TiO_2 pigment was added to diluted resin at different contents of 3, 7, and 10 wt % and grinded in a fast mill at speeds of 150–350 rpm at room temperature for 45 min. To produce CaCO_3 containing samples, TiO_2 and CaCO_3 were added to epoxy resin and grinded by fast mill at speed of 150–350 rpm in the same way. The samples produced in this research are listed in Table II.

Mechanical Tests

Static mechanical tests were performed on all specimens by SANTAM Universal Machine (STM-150). Five to ten specimens were tested to evaluate accuracy of each test.

Tensile Test. For tensile test, the specimen was made into absciss form. They were prepared according to ASTM D638.²⁶ The specimen size was 63.5 mm \times 9.5 mm \times 3 mm (i.e., length, width, and thickness) with the 3.18 mm width of the narrow section. The test was performed at cross-head speed of 2 mm/min. The maximum point of stress–strain curve was considered as tensile strength [σ_{max} (MPa)] of the specimen. Ductility (ϵ_f) was obtained by calculating the strain of fracture stress in stress–strain curve. Tensile modulus [E (MPa)] was obtained from tangent of slope of the straight-line portion in

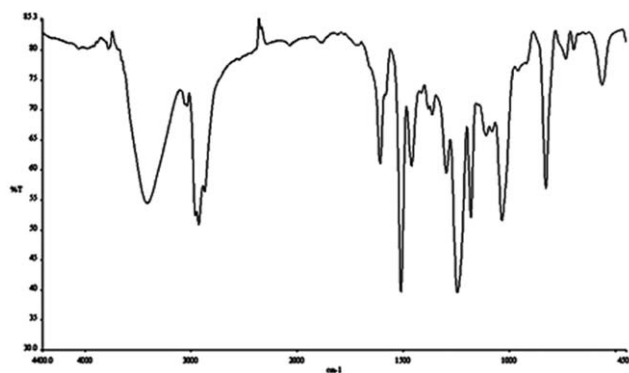


Figure 2. FTIR analysis of the used epoxy resin.

Table II. Chemical Composition of the Prepared Samples

Sample code	Epoxy binder	CNT (wt %)	Nano-Fe ₂ O ₃ (wt %)	Nano-CuO (wt %)	CaCO ₃ (wt %)	TiO ₂ (wt %)
Control (Epoxy)	√	—	—	—	—	—
E-CNT(1.5)	√	1.5	—	—	—	—
E-CNT(3)	√	3	—	—	—	—
E-CNT/FeO	√	1.5	1.5	—	—	—
E-CNT/CuO	√	1.5	—	1.5	—	—
E-CNT/FeO/CuO	√	1	1	1	—	—
E-TiO ₂ (3)	√	—	—	—	—	3
E-TiO ₂ (7)	√	—	—	—	—	7
E-TiO ₂ (10)	√	—	—	—	—	10
E-TiO ₂ -CaCO ₃ (3)	√	—	—	—	3	7
E-TiO ₂ -CaCO ₃ (7)	√	—	—	—	7	7
E-CNT-TiO ₂ (7)	√	1.5	—	—	—	7

stress–strain curve. Toughness [T (MJ/m³)] was determined as surface area under stress–strain curve.

Flexural Test. Flexural test was performed as three-point bending test procedure. The specimens were prepared according to ASTM D790.²⁷ For flexural test, the specimen sizes were 127 mm × 12.7 mm × 3 mm (i.e., length, width, and thickness). The test was performed at cross-head speed of 3 mm/min and length span of 48 mm. According to standard test method, flexural characteristics were determined to strain of 0.05%, because no break has occurred in the specimens. Flexural

strength and toughness in flexural test were obtained such as tensile test. A yield strength [σ_0 (MPa)] was defined as the stress at which a sample begins to deform plastically. Flexural modulus was calculated by drawing a tangent to the steepest initial straight-line portion of the load deflection curve.

Compressive Test. To determine compressive properties, the specimens were prepared in the form of a cylinder on the basis of ASTM D695.²⁸ Specimen sizes were 10 mm in diameter and 25.4 mm in length. The test specimens were evaluated at cross-head speed of 2 mm/min.

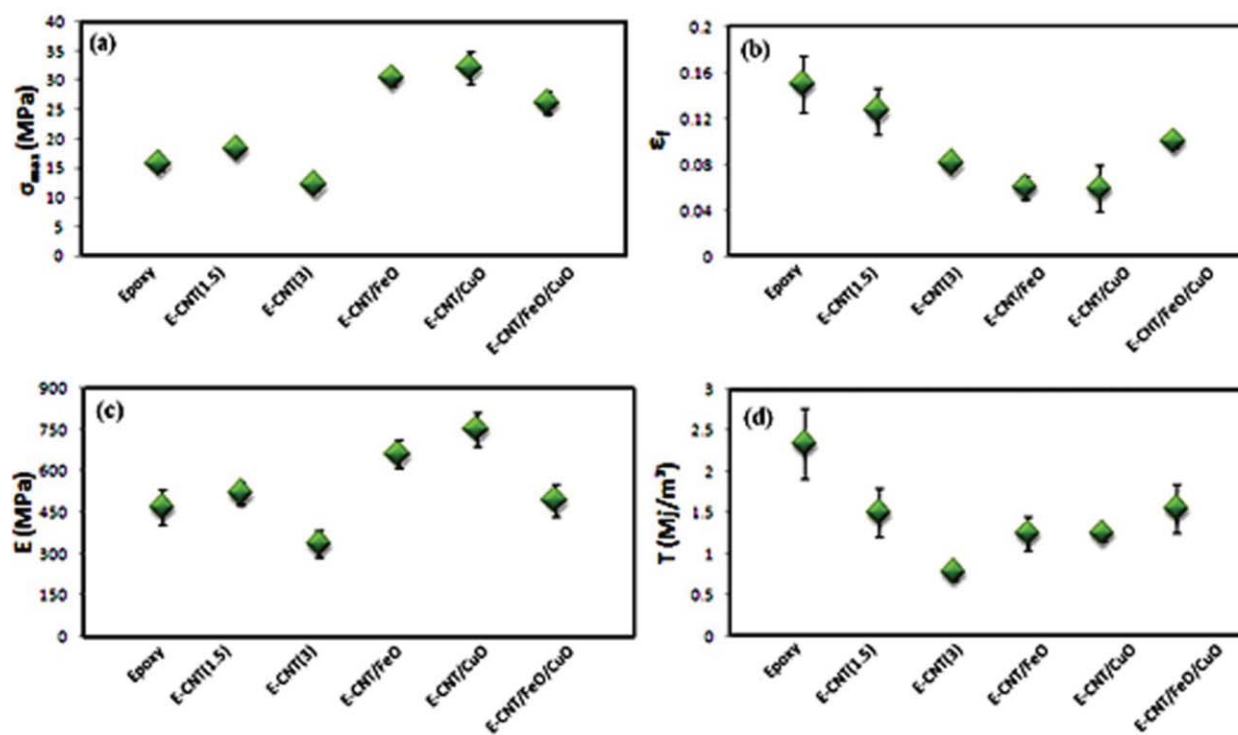


Figure 3. Tensile characteristics of epoxy nanocomposites, (a) tensile strength, (b) ductility, (c) tensile modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 4. SEM micrographs of epoxy nanocomposites containing MWCNT and Fe_2O_3 hybrid nanofillers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Thermal Behavior

Thermal degradation and stability of samples were studied by thermal gravimetric analysis (TGA) and derivative thermal degradation (DTG). TGA data were obtained on a thermal gravimetric analyzer; model LQMS-F36-0. TGA were performed at heating rate of $10^\circ\text{C}/\text{min}$ from room temperature up to 600°C under argon atmosphere.

Thermal conduction of the specimens was tested with a one-dimensional thermal conductivity test machine. The Fourier's law was used to measure thermal conductivity coefficient.

RESULTS AND DISCUSSION

Effect of Hybridizing Nanoparticles

Tensile testing was performed on epoxy composites and MWCNT containing epoxy nanocomposites (i.e., single MWCNT and/or its hybrid with nano metal oxides). Figure 3 shows the results. It can be seen that application of 1.5 wt % of MWCNT had negligible effect on the increment of tensile strength and modulus. However, at 3 wt % loading of MWCNT a clear decrease was observed in mechanical properties. It was attributed to agglomeration of the particles in the epoxy matrix.

It is also evident that by adding $\text{Fe}(\text{OH})_2$ and CuO nanoparticles to MWCNT containing epoxy nanocomposites, tensile strength and modulus increased but ductility and toughness decreased. This means that these nanoparticles increased stiffness of MWCNT containing epoxy nanocomposite. It was attributed to net forming effect of nanoparticles in the presence of other particles. In this case they produce particle network which help in better dispersion. This phenomenon caused improvement in tensile properties.

To evaluate nanoparticles dispersion, SEM analysis was performed on cross-section of the samples. First, very thin films from nanocomposites samples were prepared, then films were broken in liquid nitrogen and SEM analysis was performed on the films cross-sections. Figure 3 shows SEM micrograph of epoxy nanocomposites containing 1.5 wt % of MWCNT and Fe_2O_3 hybrid nanofillers. Large agglomerates of nanoparticles in the epoxy matrix were observed.

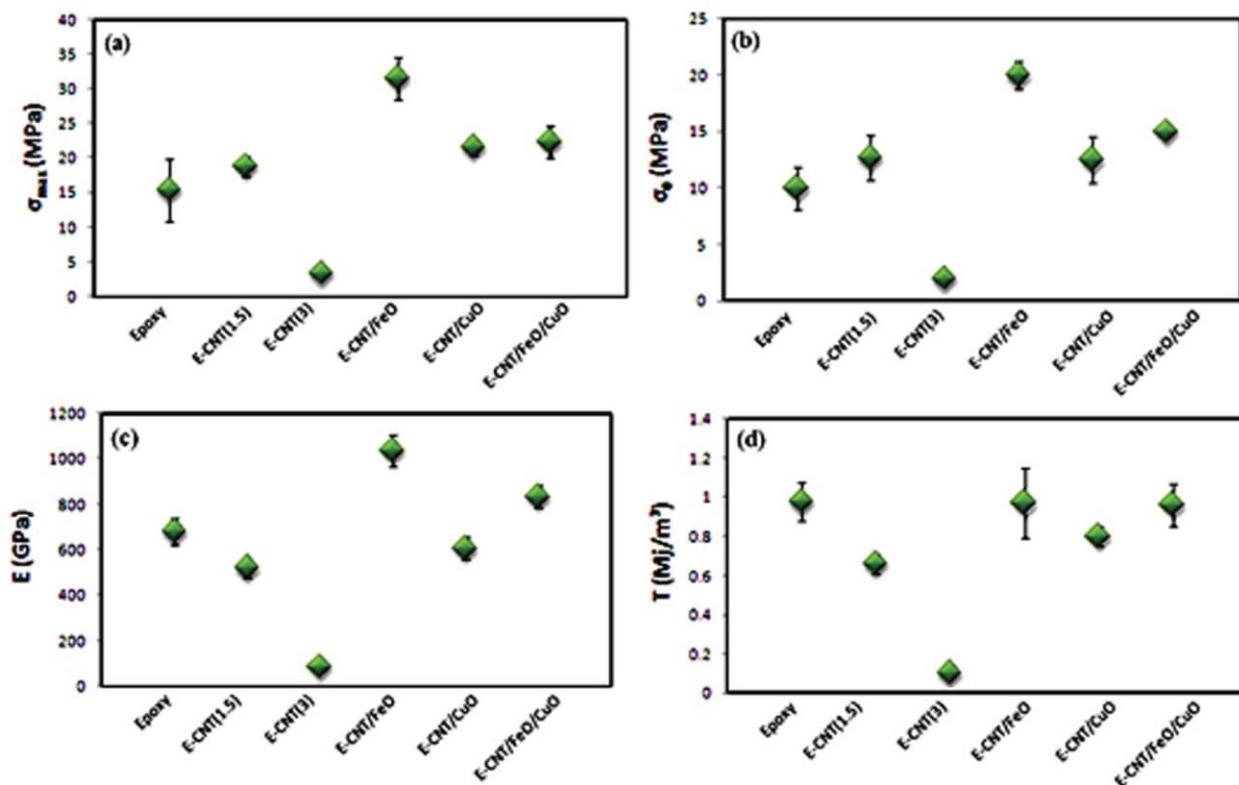


Figure 5. Flexural characteristics of epoxy composites, (a) flexural strength, (b) yield strength, (c) flexural modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

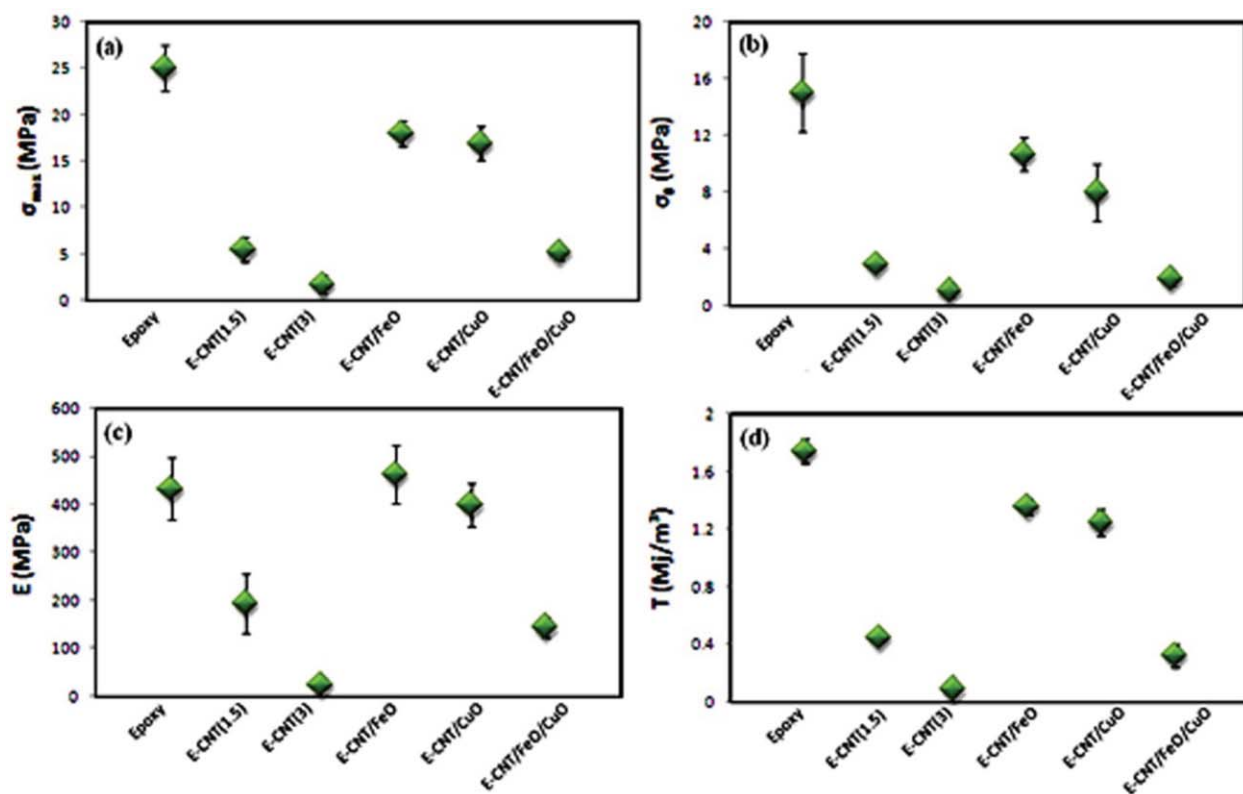


Figure 6. Compressive characteristics of epoxy composites, (a) compressive strength, (b) yield strength, (c) compressive modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

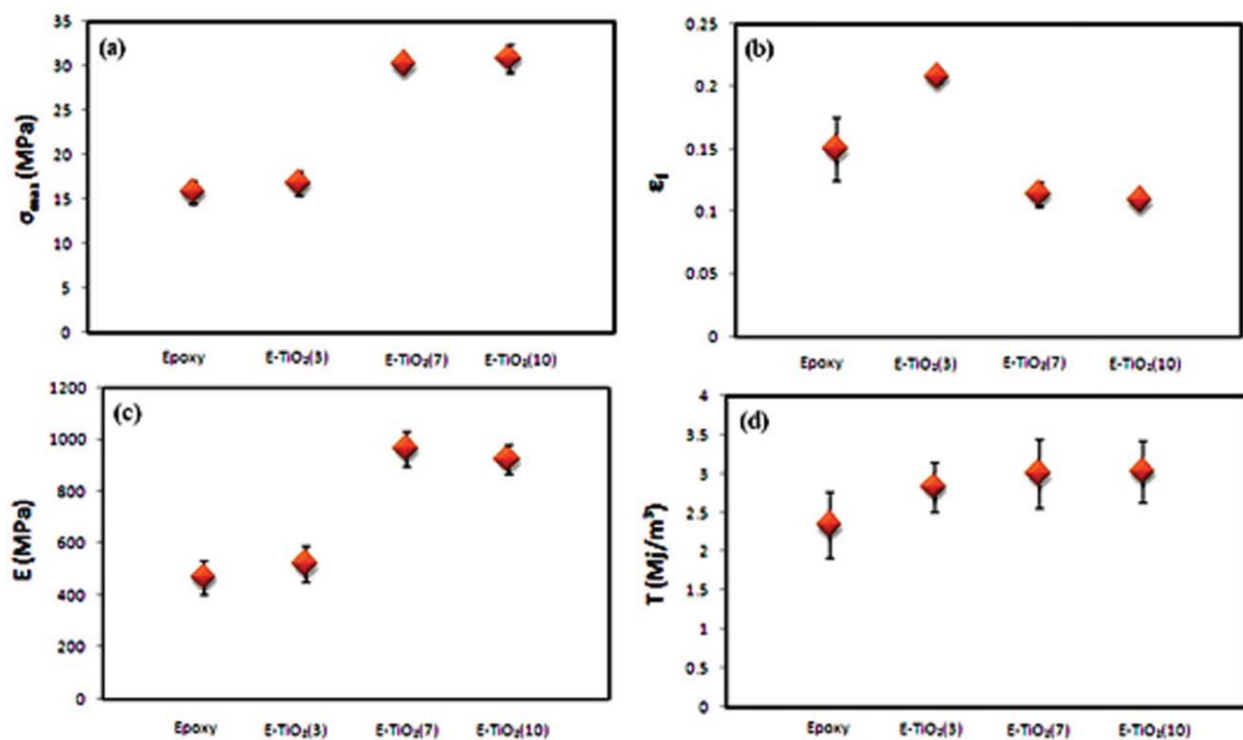


Figure 7. Tensile characteristics of epoxy sample containing TiO₂, (a) tensile strength, (b) ductility, (c) tensile modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

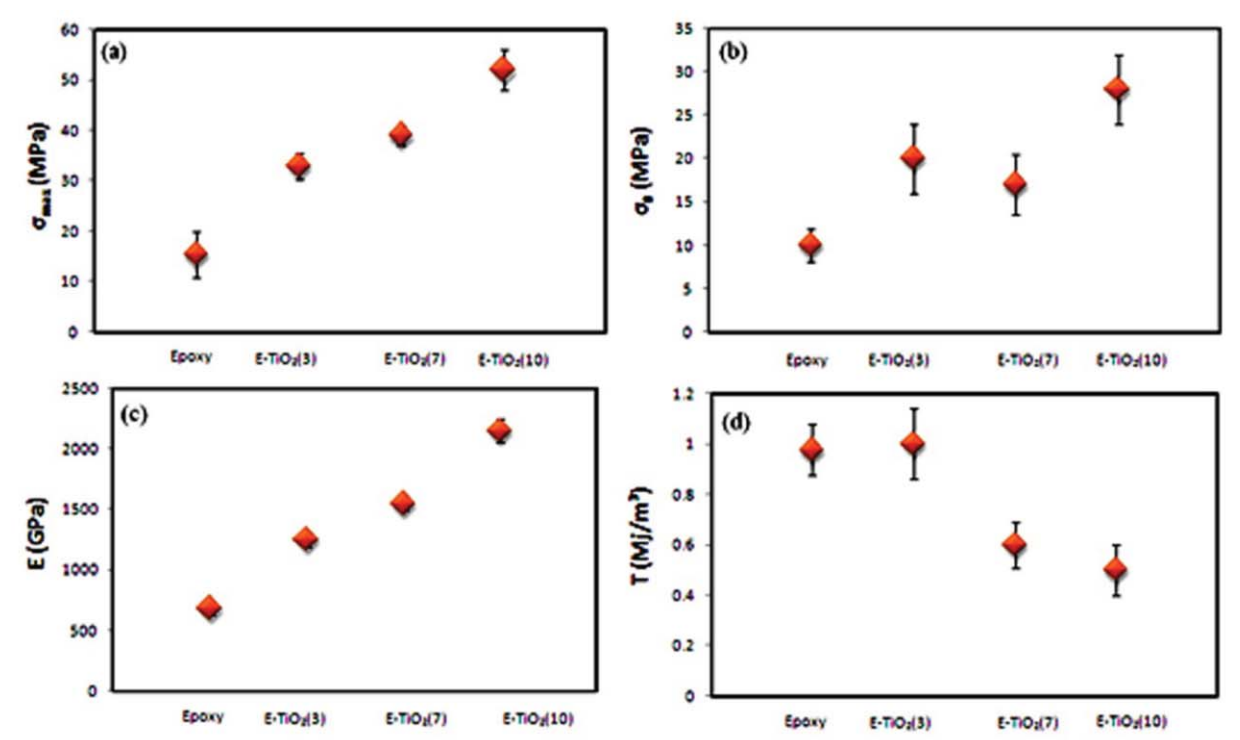


Figure 8. Flexural characteristics of epoxy sample containing TiO₂, (a) flexural strength, (b) yield strength, (c) flexural modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Indeed high specific surface area of MWCNT prevented suitable dispersion of other nanoparticles in the matrix and nanoparticles created agglomerates. So that large agglomerates caused decrease in tensile properties. This was issued by other researchers.¹⁶ Agglomerates of nanoparticles (containing MWCNT and Fe₂O₃) and resin rich area have been marked in Figure 4.

Figure 5 shows flexural behavior of the nanocomposites. It is observed that MWCNT has negligible effects on flexural behavior of epoxy binder at 1.5 wt % of loading. The sample containing 3 wt % of MWCNT showed dramatic decrease in flexural properties as tensile test results. This result confirms our suggestion on agglomeration of the MWCNTs.

Hybridizing nanoparticles with MWCNT caused increase in flexural behavior of the epoxy samples, except for toughness. This was also observed for tensile test samples which was attributed to net forming effect of metal nanoparticles on improving dispersion of MWCNTs.

The most significant improvement of flexural characteristics was obtained in E-CNT/FeO sample which showed 105% increment in flexural strength. Flexural results show that iron oxide nanoparticles are more effective than copper oxide nanoparticles in improvement of the mechanical properties.

Figure 6 shows compressive properties of the nanocomposites. It is seen that the compressive characteristics are reduced by increasing in various hybrid nanoparticles and single carbon nanotube. So that the most decrements of compressive characteristics were obtained in E-CNT/FeO/CuO and E-CNT(3) samples. In compression, all nanocomposites showed

decline in characteristics. This can be attributed to decrease in stiff resin content in the samples and softening effect of the particles in the matrix. The decreasing content was worse in the case of using MWCNT due to fibrous shape of the nanofiller and agglomeration of these nanoparticles in epoxy resin which impact load bearing ability of the samples.

Effect of TiO₂ Pigment

To investigate the effect of TiO₂ particles on mechanical properties of epoxy resin, different amounts of TiO₂ (i.e., 3, 7, and 10 wt %) were introduced to epoxy resin and mechanical characteristics of the samples were measured. Figures 7 and 8 show the results of tensile and flexural properties, respectively. It is clearly seen that by increasing TiO₂ content up to 10 wt %, tensile and flexural characteristics increased (except tensile ductility and flexural toughness). It should be attributed to good interaction of TiO₂ pigment to epoxy binder and its reinforcing effect on the resin.

Figure 9 shows compressive characteristics of TiO₂ containing epoxy samples. It was found that just by adding 10 wt % of TiO₂ pigment to epoxy resin, compressive strength and modulus increased. It is evident that at loading lower contents of TiO₂, compressive strength remained constant. These results were in good correlation to previous studies.^{20,29}

Effect of Micro- and Nanoparticles

Samples containing 7 wt % of TiO₂ showed higher tensile behavior, so, this pigment value was chosen as the optimum content for the next samples. Tensile characteristics of prepared pseudo-coatings containing pigment and filler are shown in

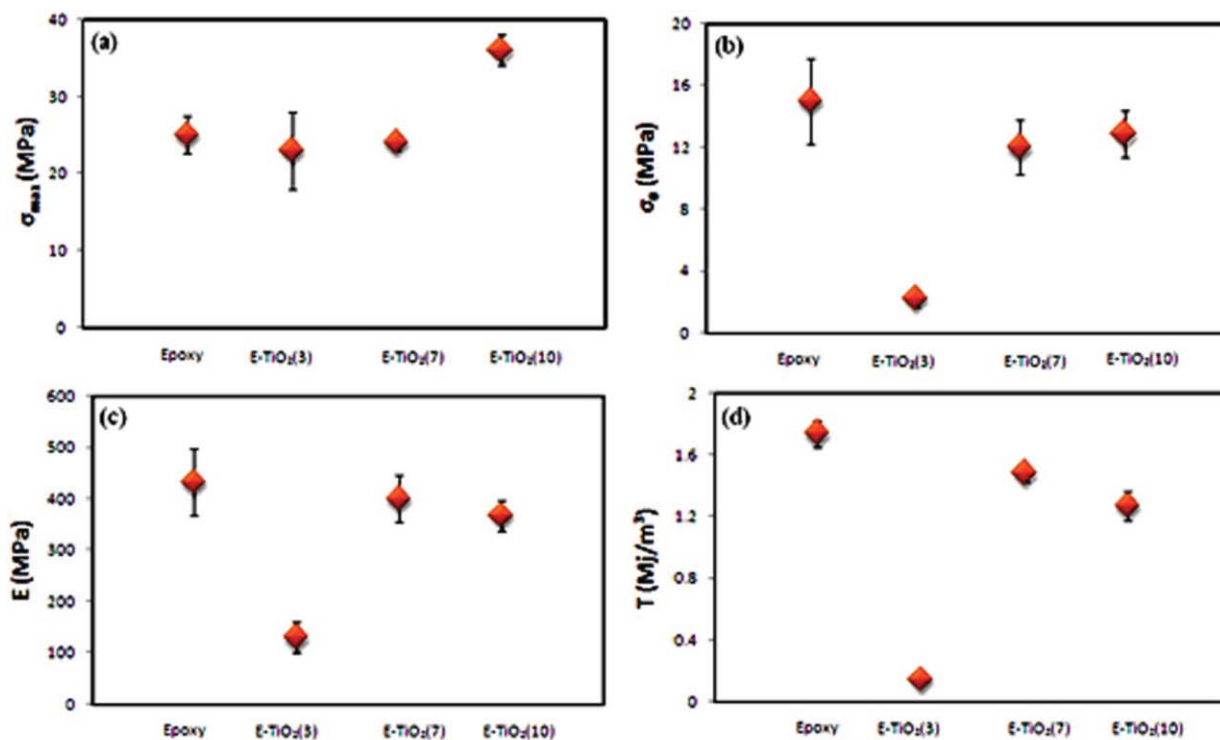


Figure 9. Compressive characteristics of epoxy sample containing TiO₂, (a) compressive strength, (b) yield strength, (c) compressive modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 10. Tensile strength and modulus of the sample increased, by increase in CaCO₃ content. Flexural characteristics of the sample showed slight improvement (see Figure 11).

Figure 12 shows compressive characteristics of this composite. It is seen that all compressive properties showed considerable increase and E-TiO₂-CaCO₃(7) sample showed the highest compressive strength.

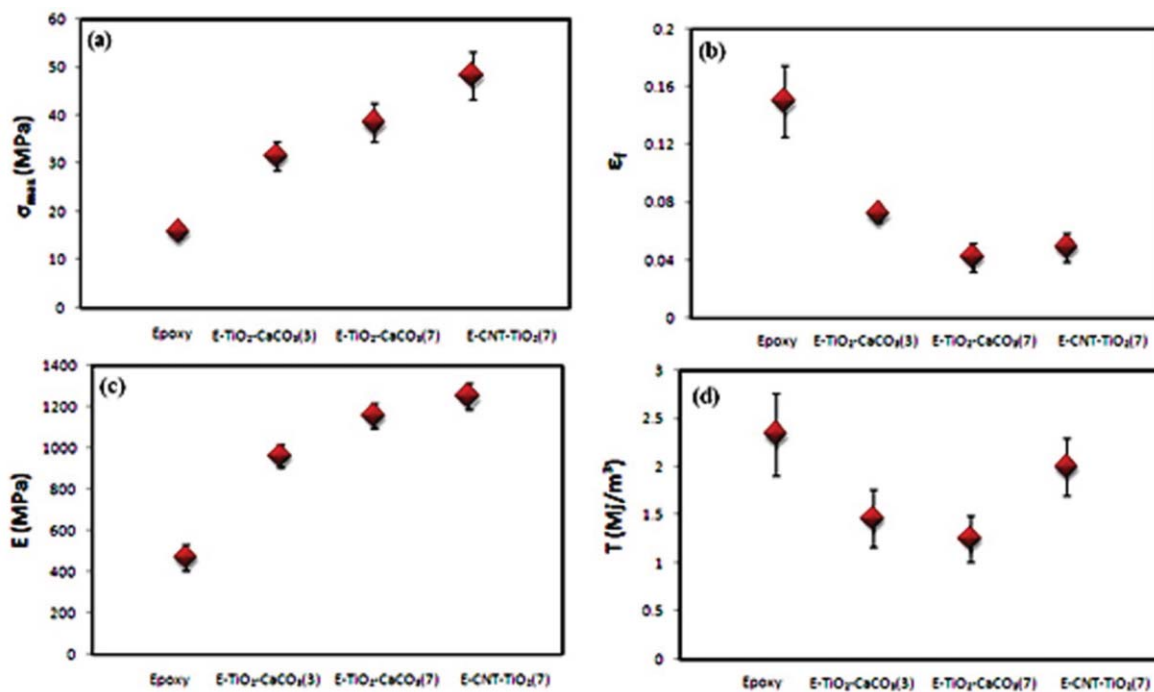


Figure 10. Tensile characteristics of epoxy pseudo-coating, (a) tensile strength, (b) ductility, (c) tensile modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

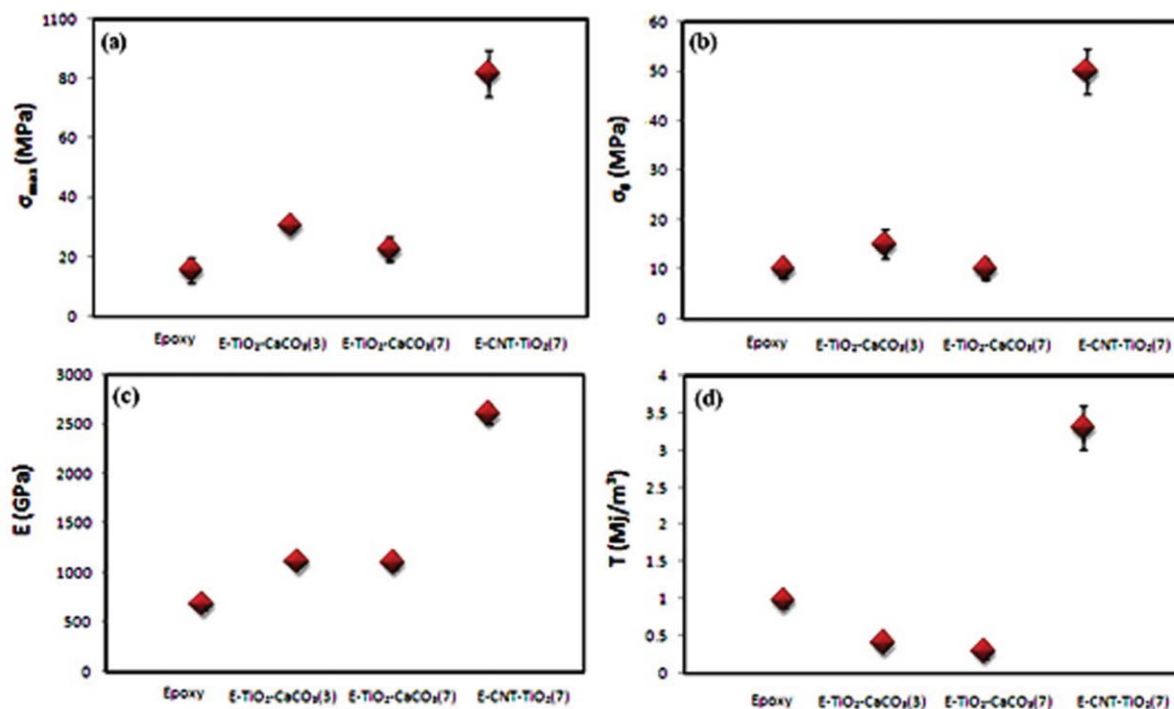


Figure 11. Flexural characteristics of epoxy pseudo-coating, (a) flexural strength, (b) yield strength, (c) flexural modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

In all mechanical tests, E-CNT-TiO₂(7) sample showed the best mechanical properties (except in compressive toughness). It means that the presence of MWCNT along with TiO₂

microparticles had a great effect on mechanical performances. On this basis, using MWCNT not only improves thermal conduction of epoxy composites but increases mechanical

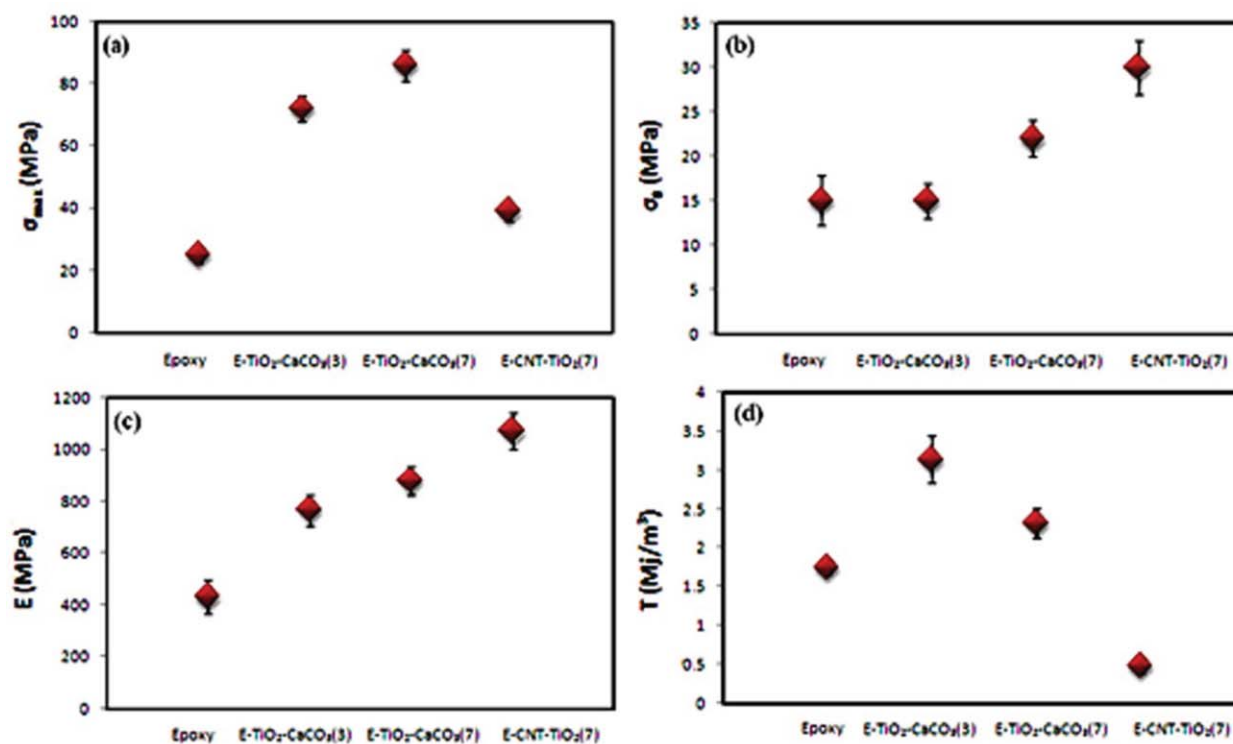
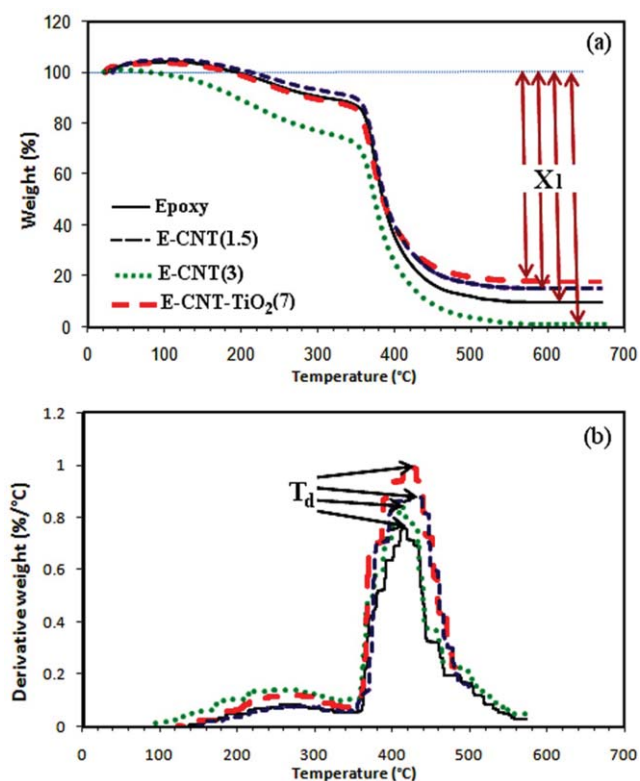


Figure 12. Compressive characteristics of epoxy pseudo-coating, (a) compressive strength, (b) yield strength, (c) compressive modulus, and (d) toughness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table III. Increase/Decrease Percent of Mechanical Properties of Epoxy Composites with Nano and Microparticles Compared to Pure Epoxy

Sample code	Tensile characteristics (%)						Flexural characteristics (%)						Compressive characteristics (%)					
	Strength	Modulus	Toughness	Ductility	Strength	Modulus	Toughness	Yield strength	Strength	Modulus	Toughness	Yield strength	Strength	Modulus	Toughness	Yield strength		
E-CNT(1.5)	16	10	-36	-15	22	-23	-32	27	-78	-55	-74	-80	-78	-55	-74	-80		
E-CNT(3)	-23	21	-67	-46	-78	-87	-89	-80	-93	-94	-95	-93	-93	-94	-95	-93		
E-CNT/FeO	93	41	-47	-60	105	52	-1	100	-28	7	-22	-29	-28	7	-22	-29		
E-CNT/CuO	103	60	-47	-61	40	-11	-18	25	-32	-7	-28	-47	-32	-7	-28	-47		
E-CNT/FeO/CuO	66	5	-34	-33	45	22	-2	50	-79	-67	-81	-87	-79	-67	-81	-87		
E-TiO ₂ (3)	6	11	21	39	114	84	2	100	-8	-70	-91	-85	-8	-70	-91	-85		
E-TiO ₂ (7)	100	106	28	-24	153	127	-39	70	-4	-7.4	-15	-20	-4	-7.4	-15	-20		
E-TiO ₂ (10)	95	97	30	-27	238	216	-49	180	44	-15	-27	-14	44	-15	-27	-14		
E-TiO ₂ -CaCO ₃ (3)	99	105	-38	-52	98	63	-59	50	188	77	80	0	188	77	80	0		
E-TiO ₂ -CaCO ₃ (7)	144	147	-47	-72	46	62	-70	0	244	104	33	47	244	104	33	47		
E-CNT-TiO ₂ (7)	206	167	-15	-67	431	282	238	400	56	148	-72	100	56	148	-72	100		

**Figure 13.** Thermal stability of epoxy, E-CNT-TiO₂(7) and E-CNT(3) samples, (a) TGA and (b) DTG. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

properties, simultaneously. It should be mentioned that loading 1.5 wt % of single MWCNT has no significant influence on epoxy mechanical properties (see Figures (3 and 5) and 6).

The mechanical properties of tensile, flexural, and compressive tests for all of the samples are presented in Table III. Data is calculated on the increase/decrease percent of mechanical properties compared to pure epoxy. E-CNT/FeO/CuO compared to other hybrid nanoparticles has the lowest compressive properties which shows 79% reduction in compressive strength. By adding CaCO₃ up to 7 wt % in pseudo coating composites, strength and modulus increase in tensile and flexural tests. Generally, the most significant improvement in modulus was at obtained at E-CNT-TiO₂(7) sample.

Thermal Gravimetric Analysis

Due to obtaining the best results for E-CNT-TiO₂(7) sample, it was used in TGA and DTG to be compared to epoxy control sample, E-CNT(1.5) and E-CNT(3) samples. Results are shown

Table IV. TGA and DTG Data for Epoxy Samples

Sample code	T ₅ (°C)	T ₅₀ (°C)	T ₈₀ (°C)	T _d (°C)	X _d (%)	X _i (%)
Epoxy	250	390	435	415	72	92
E-CNT(1.5)	275	390	462	430	75	85
E-CNT(3)	160	375	410	405	77	100
E-CNT-TiO ₂ (7)	250	390	500	423	72	82

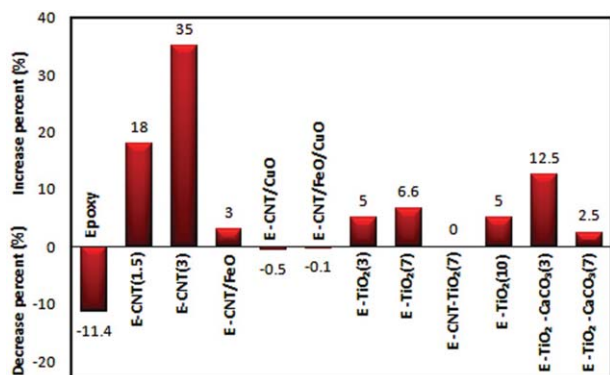


Figure 14. Increase/decrease percent of thermal conductivity of epoxy composites with nano and micro particles. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

in Figure 13. Table IV shows details of curves. T_5 , T_{50} , and T_{80} are defined as temperature at which 5, 50, and 80% of the mass is volatilized, respectively, and T_d is the decomposition temperature. X_d and X_l are decomposition conversion and last conversion respectively.

Results of TGA and DTG analysis showed that samples follow the same decomposition behavior at elevated temperatures. This means that all samples have one peak of decomposition temperature and the graphs have similar trend. A considerable effect on decomposition temperature was seen by adding MWCNT at lower contents, but by increasing MWCNT content up to 3 wt % there were no significant influences on decomposition temperature. By increasing MWCNT content up to 3 wt %, decomposition conversion and last conversion decreased. It was attributed to weak dispersion and agglomeration of MWCNTs at higher loading contents.

Adding TiO₂ to MWCNT containing epoxy composites caused decline in decomposition temperature and increase in thermal stability. The decrease in T_d was attributed to lower loading (i.e., 1.5 wt %) of MWCNT in this sample. Improvement in thermal stability of the sample was attributed to the presence of thermal stable inorganic TiO₂ pigment. These results were in good agreement with previous studies.^{2,30,31}

Thermal Conduction

Effects of different micro- and nanoparticles on thermal conduction of epoxy matrix as increase/decrease percent compared to E-CNT-TiO₂(7) sample are illustrated in Figure 14. It is evident that all particles had positive effect on thermal conduction of epoxy polymer. However, MWCNT at different contents of loading showed the most improving effect. Application of TiO₂ pigment improved thermal conduction. The effect was considerable especially when low contents of CaCO₃ used along with TiO₂.

The interesting results was obtained in E-CNT-TiO₂(7) sample. It seems that the presence of both conductive materials in epoxy binder caused interference of conduction mechanisms. It should be attributed to this fact that TiO₂ probably cut thermal conduction paths of MWCNT. In the same way, MWCNT restrict thermal conduction through TiO₂ particles.

CONCLUSIONS

In this study, the experimental results of mechanical characteristics, thermal degradation, and thermal conductivity of epoxy composites containing different nano- and microparticles were investigated. On the basis of the data the following results was concluded:

- The MWCNT particles had negligible effect on tensile and flexural properties of epoxy matrix at 1.5 wt % of loading while at 3 wt % of loading it caused significant decrease in the properties (i.e., 23 and 78% decrease, respectively) due to agglomeration because of lack of surface treatment.
- Hybridizing the MWCNT with the nanoparticles caused more than 100 and 300% improvement in tensile and compressive properties, respectively. Fe(OH)₂ nanoparticles caused 100% improvement in flexural properties but CuO showed lower increments (i.e., 50%).
- Adding the TiO₂ pigment up to 10 wt % to the epoxy resin caused increment in flexural and tensile strengths between 114 to 238% and 6 to 100%, respectively. This was attributed to reinforcing effect of TiO₂ pigment on the epoxy resin and good interaction of the resin to the pigment.
- Incorporating the MWCNT and the TiO₂ pigment into the epoxy matrix caused 206 and 431% improvement in tensile and flexural strengths, respectively. It was observed that using both particles individually improve mechanical properties, but simultaneous usage of them showed synergistic effect and improved considerably the mechanical properties.
- Using the MWCNT and the TiO₂ influenced thermal stability of the epoxy composite but TiO₂ showed higher improvement, Because of higher thermal stability of the inorganic TiO₂ pigment.
- Adding lower contents of the CaCO₃ (i.e., 3 wt %) in the presence of 7 wt % of the TiO₂, despite of its non-thermal conductive nature showed considerable improvement in the thermal conduction of epoxy composites. It means that CaCO₃ and TiO₂ can also use as micro conductive pigments in epoxy composites.

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