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RESEARCH PAPER

Investigation of Mechanical Properties of Polyester/Polyethylene Glycol/TiO_2 Nanocomposites

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ABSTRACT

In this paper, the effects of synthesized titanium dioxide (TiO_2) nanoparticles and Polyethylene glycol (PEG) on mechanical, morphological, and thermal properties of unsaturated polyester (UPE) based nanocomposites were studied. The TiO_2 nanoparticles were synthesized by sol–gel (SG) method. The UPE/PEG/TiO_2 nanocomposites were prepared at various concentrations of synthesized TiO_2 nanoparticles and PEG by direct mechanical mixing technique. The synthesized TiO_2 nanoparticles were mixed with UPE resin through ultra-sonication in different weight fractions (wt%), namely, 0%, 0.5%, 0.75%, and 1%. Polyethylene glycol (PEG) is considered in different wt% fractions such as 5wt%, 10wt%, 15wt% for preparing UPE/PEG/TiO_2 nanocomposite. Consequently, chemical structure of nanocomposite was investigated with FT-IR analyses. Also, the TiO_2 nanoparticles and optimized samples were characterized by TGA, SEM and XRD analyses. The results obtained by TGA, FT-IR, SEM and XRD analyses exhibited an improvement of thermal and mechanical properties of the nanocomposites containing synthesized TiO_2 nanoparticles (0.5%) and PEG (10%) compared to pristine polyester.

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INTRODUCTION

Improvements in product performance depend on the ability to make new materials that exhibit enhanced properties, such as strength, impact resistance, fracture toughness, durability, etc. Nanocomposites are a new class of materials to meet this challenge, as various researches have shown that they have high thermal, physical and mechanical properties in contrast to conventional composites, because of the much stronger interfacial interactions involving the dispersed nanoparticles and the matrix (1-7).

Recently, polymer nanocomposites have been established as an interesting class of polymers that has been reinforced with small quantity (less than 5% by weight) of nano-sized materials (1). The advancement of polymer nanocomposites can produce unique materials with upgraded properties. The reinforcing materials or fillers are nano scaled and dispersed into the polymeric matrix, with their main function being the improvement of the barrier properties to liquids and gases (8), physical-chemical (9), mechanical and thermal properties (10), among others. It is worth pointing out, that the degree of enhancement of these properties is strongly dependent on the extent of filler adhesion to the matrix, the filler/matrix type of system used, the

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shape and size of filler and the level of the filler dispersion throughout the matrix and aggregate size.

In preparing a polymer nanocomposite, the interaction between fillers and the polymer matrix depends on the structure and the nature of the filler used. Among the most studied, the aluminum silicates , clays and metal oxides with different kinds of forms are filler materials (11, 12).

The use of inorganic fillers in polymer nanocomposite production has been a common practice in the plastic industry to enhance the mechanical and thermal properties of thermosets and thermoplastics, such as hardness, heat distortion temperature, stiffness, toughness and mold shrinkage. In general, the physical properties, such as barrier properties and surface smoothness, cannot be performed using convectional micron-sized particles. Furthermore, the thermal and mechanical properties of the composites filled with micron-sized particles are not as good as the ones packed with nanoparticles of the exact same filler (13).

In materials studies, polymer nanocomposites appear in the numerous multidisciplinary sciences with wide applications that have many advantages for industry. Polymer resins have been broadly used as matrix materials for many high-performance components in the building construction, automotive area, water pipes, architectural panels, boat hulls, panels in stackable chairs and aircraft due to their high chemical resistance, easy processing ability, and comparatively low charge (14, 15).

Highly cross-linked thermosetting polymeric materials, such as the unsaturated polyester (UPE) resin used as the polymer matrix in composites. Unsaturated polyester is really a long-chain polymer that is chemically made of an ester, a terephthalic acid and a dihydric alcohol, formed by the esterification condensation of acids and polyfunctional alcohols. Unsaturated polyesters refer to that category of thermosetting polyesters with alkyl resins as the backbone. They are basically used in reinforced plastics used in the transportation and marine industries.

Unsaturated polyester resin offers many benefits in comparison to other thermosetting resins such as excellent mechanical and thermal properties and transparency, room temperature cure capability, low pressure molding capabilities which will make it particularly useful for large component manufacture at somewhat minimal cost (16). In contrast to other thermosetting resins, no by-product is made during the curing reaction. Hence resins is often cast, molded at low temperature and pressure(17).

Although unsaturated polyester resins are very brittle because of their covalently bonded network structure, and therefore are poor inhibitors of crack initiation and propagation. Nonetheless, researchers have been able to enhance their toughness with the addition of soft materials, as well as rigid fillers, although it is essential to remember that the increase in toughness thus obtained is frequently along with a concomitant reduction in modulus and strength (18-21).

In this study, unsaturated polyester resin is used. It is actually pre-promoted for room temperature cure with the addition of Methyl Ethyl Ketone Peroxide (MEKP) as the catalyst. The unsaturated polyester resin is a highly cross-linked thermosetting polymer that is rigid, transparent and extremely brittle below the glass transition temperature (Tg) which is in the range of 350-360 $^{\circ}$C; therefore, it is an ideal candidate for the study of the effect(s) of many kinds of fillers on fracture behavior.

Polyethylene glycol (PEG) solubility in water shows biocompatibility and hydrophilicity and plasticizers is generally used, to improve the process-ability of polymers, flexibility and ductility of glassy polymers and reduce the glass transition temperature of the polymer, that means the plasticizer and polymer need to be miscible (1).

Numerous techniques are already useful to synthesize polymer nanocomposites: hot pressing of powders, sol–gel, and melt intercalation (22-25). The production of polymer nanocomposites using the direct incorporation of nano-scale building blocks into polymer solution (or) melt definitely seems to be finer quality than other methods. However, the manufacturing of polymer based nanocomposite, is not as easy task by the use of processing techniques common to conventional plastics, as nanoparticles tend to form agglomerates, or clusters, containing several nanoparticles, frequently negating the sought after "*nano effect*" (26).

The literature has described several studies on the preparation of unsaturated polyester nanocomposites with different inorganic fillers (TiO_2, SiO_2, Fe_2O_3, $CaCO_3$, Al_2O_3 and ZnO) (27). In this work, synthesized TiO_2 nanoparticles and

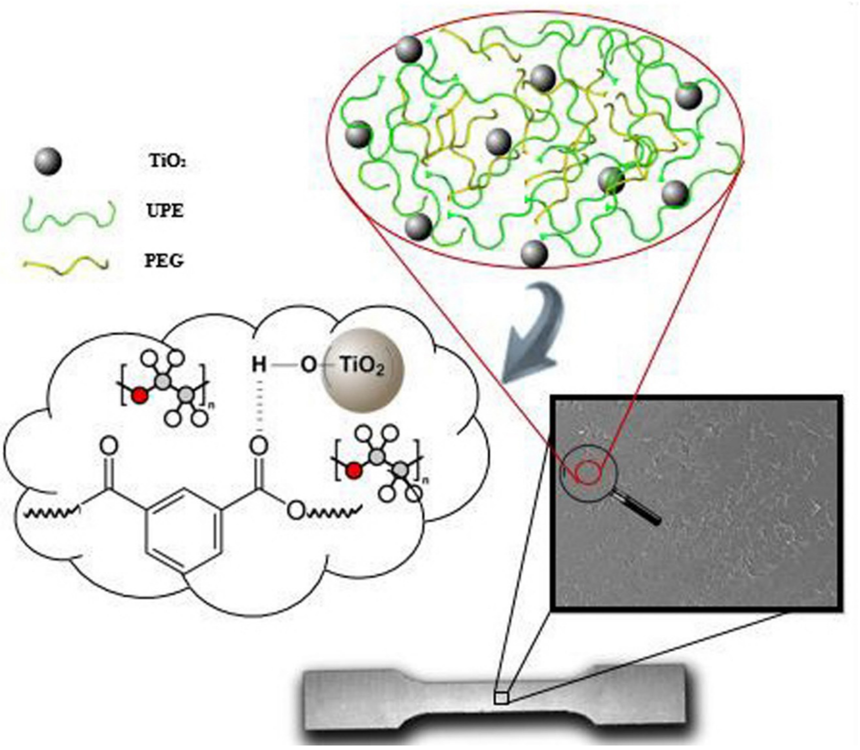

Fig. 1. Representation of the interaction fillers-polymer matrix.

polyethylene glycol are used to prepare polymer nanocomposites. Titanium dioxide nanoparticles with 36 nm average diameter, were selected as the filler component because of their availability, spherical morphology and high modulus. The interaction between the OH groups on the surface of TiO_2 nanoparticle and the ester group of the unsaturated polyester matrix is shown in Fig. 1. The addition of TiO_2 nanoparticles and PEG in the polymeric matrix can improve mechanical and thermal properties of the polymer nanocomposite.

MATERIALS AND METHODS

Materials and chemicals

Unsaturated polyester (UPE) was purchased from Iran ketone company (Iran) and used as polymer matrix of composite in addition of methyl ethyl ketone peroxide (MEKPO) and cobalt naphthenate as catalytic reaction. Polyethylene glycol (PEG), Titanium tetraisopropoxide (TTIP), glacial acetic acid, and ethanol were from Merck.

Preparation of TiO_2 Nanoparticles.

In this study, titanium dioxide nanoparticles were synthesized by sol–gel (SG) method using TTIP, ethanol and glacial acetic acid as precursors, based on the our previous work according to reference (28). Finally, prepared nanoparticles were annealed at 550 °C for 2h.

Polymer Nanocomposite Fabrication

The preparation of UPR/PEG/nanotitania composite was carried out by direct mechanical mixing technique. UPR/PEG/ TiO_2 nanocomposites were prepared by mixing nanotitania and PEG as filler with UPE as a matrix in addition to Methyl ethyl ketone peroxide catalyst (MEKPO) and cobalt naphthenate accelerator. The process is carried in different wt % of PEG such as 0wt%, 5 wt%, 10% wt%, 15% wt% and different wt % of synthesized TiO_2 nano particles such as 0wt%, 0.5 wt%, 0.75 wt% and 1wt% to study the influence of PEG and TiO_2 nanoparticles. In the first step, TiO_2 nanoparticles and PEG were thoroughly mixed with 300 mL of UPE taken in a container equipped with the mechanical stirrer. The solution mixture was stirred well for 30 min followed by ultrasonication at room temperature to achieve uniform distribution of TiO_2 within the matrix. The suspension was then mixed with a hardener

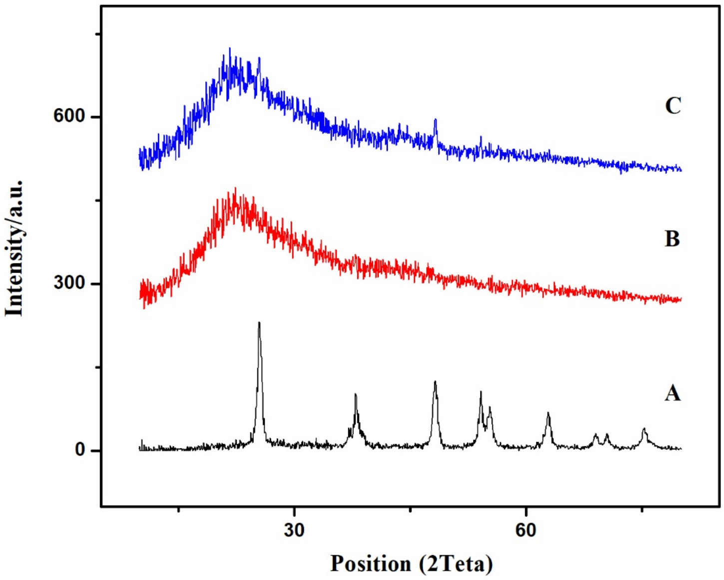

Fig. 2. XRD patterns of (A) TiO_2 nanoparticles, (B) unsaturated polyester and (C) UPE/PEG/TiO_2 nanocomposite

for 10 min by stirring. MEKPO catalyst (3 mL) and cobalt naphthenate accelerator (1 mL) were added to the above mixture to initiate the curing reaction. It was then transferred to a clean mold and allowed to cure at room temperature for 48 h. The fabricated specimens were then dried and stored in a desiccator for further use.

Mechanical Characterization

Mechanical properties of the polymer nanocomposite specimens were performed by a universal testing machine model TB1 according to ASTM D-638 at room temperature. For each nanocomposite, five specimens were tested and the average value was taken. The tensile strength and elongation were directly obtained from stress-strain curves. Prior to tensile test, dumbbell-shaped specimens were prepared for each sample according to (ASTM D638**)**

Morphological Characterization

Morphological surface of cross-sectional fracture of nanocomposite was analyzed by scanning electron microscope (SEM), (SEM, model S-4160, Hitachi**,** Japan) and Energy-dispersive X-ray spectroscopy (EDX) analysis (Peronis 2100, Japan). Further characterization, i.e. thermal behavior of composite was carried out by using Thermogravimetric and Differential thermal analysis (TG- DTA), (STA 503, Germany). This analysis was performed by heating 5.0 – 5.7 mg of sample from 50 to 800 °C with scanning rate of 20 °C/min in nitrogen gas atmosphere. Differences in functional group between the polymer nanocomposites and pure UPR as a control were registered by Fourier transform infrared (FT-IR) spectrometer, (Nicolet Magna IR 550, USA). The crystal phase of the synthesized polymer nanocomposites and TiO_2 nanoparticles was detected by X-ray diffraction (XRD) patterns (Philips X'pert pro MPD model X-ray diffractometer using Cu Kα radiation as the X-ray source, USA).

RESULTS AND DISCUSSION

Crystalline structure

Experimental X-ray diffraction patterns were obtained for the synthesized TiO_2 nanoparticles, unsaturated polyester and TiO_2 conjugated with UPE/PEG as shown in Fig. 2. The phase characterization of the synthesized TiO_2 nanoparticles were investigated by XRD and results are shown in Fig. 2(A). The TiO_2 contains anatase and rutile phase, with the average crystallite size of 36 nm, as obtained by the XRD. The findings demonstrated that TiO_2 anatase appears to have peaks at 2θ of 25.21°, 37.76°, 48.02°, 54.05°, 55.03°, 62.80°, 68.85°, 70.19°, and 75.07°; The TiO_2 rutile structure appears to have peaks at 28°, 31° and 62°. The crystallite size can be estimated from the widths of the X-ray diffraction peaks (the most

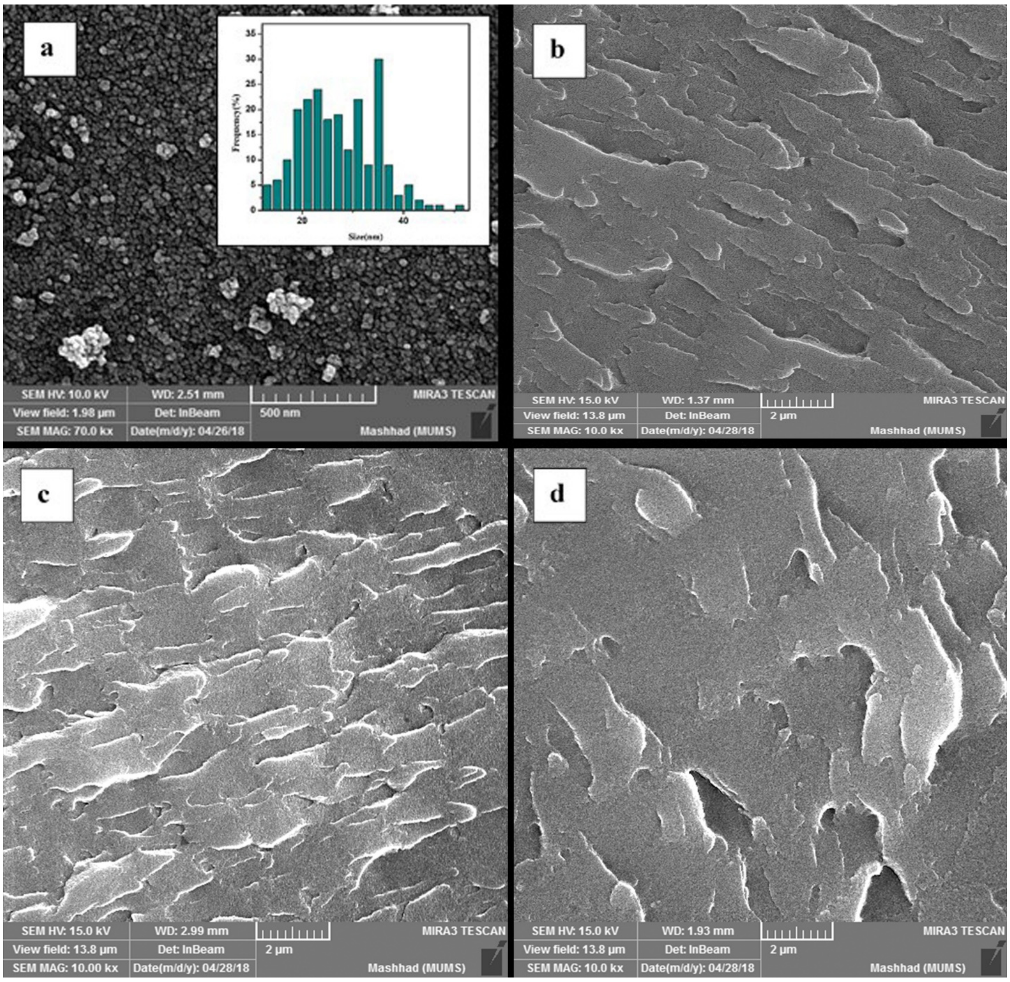

Fig. 3. SEM micrographs of fracture surface of (a) TiO_2, (b) pure UPE, (c) UPE/PEG and (d) UPE/PEG/TiO_2

intense peaks for each sample) using Scherrer's equation. Inserting the experimental data for a pronounced peak: $2\theta=25.45^{\circ}$, the average crystallite size of d=36 nm was measured.

The XRD measurement was used mainly to confirm the successful conjugation of TiO_2 to the UPE/PEG. As shown in Fig. 2, a typical pattern for synthesized TiO_2 [spectrum (A)] was observed and the structure was confirmed based on the Joint Committee on Powder Diffraction Standards (JCPDS) Card file No. 00-001-0562. This pattern confirmed the crystallinity of the nanoparticles. In spectrum (B), a broad peak around 2θ = 23 was observed for pure UPE due to its amorphous nature. In the XRD spectrum (C), diffraction peaks can be seen for both the TiO_2 along with a broad peak of UPE. The spectrum of the TiO_2–UPE/PEG composite showed a combination of these peaks, which proves that the process resulted in the formation of the TiO_2/PEG/UPE nanocomposite.

Morphological and elemental analysis

Fig. 3 (a, b, c and d) shows the SEM pattern of synthesized TiO_2 nanoparticles, pure UPE, UPE/PEG and UPE/PEG/TiO_2, respectively. It is evident that nano-sized TiO_2 particles have been prepared by the sol–gel method. As is apparent in Fig. 3a, the nanoparticles are global, uniform and slightly agglomerated. Inset shows the corresponding particle size distributions as measured from SEM micrographs. Fig. 3 (b, c and d) shows the micrographs of the pure UPE, UPE/PEG and the UPE/PEG/TiO_2. With 10% of PEG and 0.5% TiO_2 in the UPE matrix, a homogeneous distribution of the filler can be observed, indicating a good compatibility with the polymeric matrix.

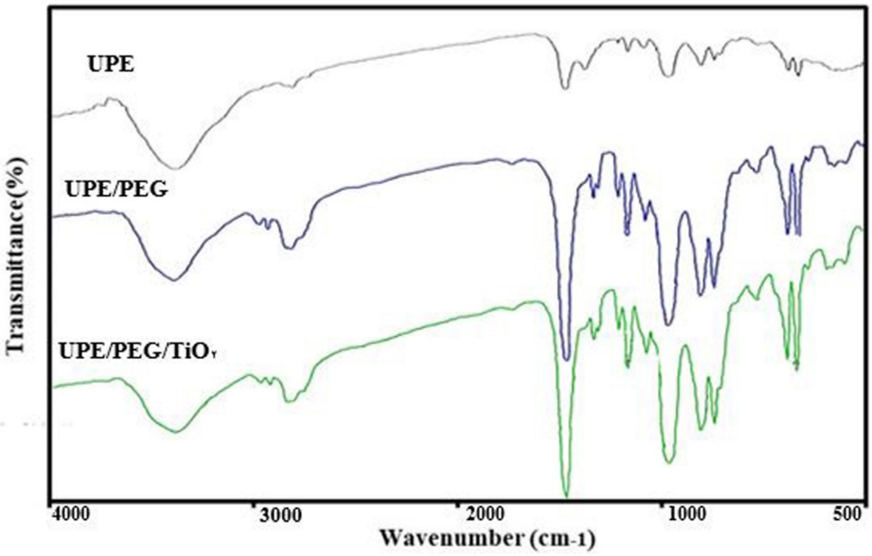

Fig. 4. FT-IR spectra of TiO_2, UPE, UPE/PEG and UPE/PEG/TiO_2 nanocomposites.

Chemical structure

Fig. 4 shows the FT-IR spectra of UPE, UPE/PEG and UPE/PEG/TiO_2 nanocomposites. The FT-IR of pure UPR shows the vibration band to be at wave number of 2931.27 cm^{-1} as aliphatic hydrocarbon (CH stretching), unsaturated bonding (C=C) at 1070 cm^{-1}, aromatic ring at wave number of 1596 cm^{-1}, and carbonyl group (C=O) at 1735.62 cm^{-1}. For the UPE/PEG composite, the absorption bands appear at 3250 cm^{-1} , which was due to the O–H stretching band, 1442, 1374 and 1339 cm^{-1} which were due to C-H bending vibrations, 2903 cm^{-1} which was due to the aliphatic C–H stretching, In addition, the combination band of C–O–H and O–C–H deformation is calculated to be from 1443 to 1340 cm^{-1}. In the area from 1145 to 554 cm^{-1}, the C–O and C–C groups vibration modes can be found and also the carbohydrates commonly show their characteristic bands.

The peaks at 3350–3450 cm^{-1} and 1620–1635 cm^{-1} can be assigned to the stretching vibration of the O–H bond and bending vibration of adsorbed water molecules, respectively. FT-IR spectrum shows a broad intense peak below 1200 cm^{-1} (indication of the Ti–O–Ti vibration) and several peaks at 653–550 cm−1and 495–436 cm^{-1} which are due to the absorption bands of Ti–O and O–Ti–O flexion vibration, respectively**.** UPE/PEG/TiO_2 nanocomposite shows almost the same absorption peaks as pristine UPE.

Tensile Strength

Tensile strength test was conducted for various weight percentages (wt%) of polymer/polyethylene glycol such as 0 wt%, 5 wt%, 10 wt%, and 15 wt% for the PEG as polymer reinforcements, by introducing TiO_2 nanoparticles in different percentages as 0 wt %, 0.5wt%, 0.75wt% and 1wt%. Fig. 5 (a, b, c and d) shows the tensile strength properties of the nanocomposite compared with UPE as a control.

From Fig. 5 (a) it is observable that for the initial value of UPE, the tensile strength is 17.12 MPa. Increasing the TiO_2 nanoparticles concentration up to 1 wt% causes the tensile strength to decrease to 8.85 MPa. Decreasing the tensile strength of nanocomposite on the concentration range of 0.5-1 wt% was due to brittleness of pure UPE. Since, the unsaturated polyesters are very rigid and brittle; we should improve their toughness with the addition of soft materials.

Fig. 5 b, c and d shows the variation of tensile strength and the influence of TiO_2 nanoparticles, when PEG is used in three different concentrations (5wt%, 10wt% and 15wt%). From Fig. 5 (b), it is clear that the addition of 5wt% PEG causes the tensile strength of UPE to increase 23.81 MPa. In addition, the findings showed that the tensile strength improved from 23.81 to 25.86 by reinforcing it with 0.5 wt% TiO_2 nanoparticles. Further addition of TiO_2 concentration over 0.5

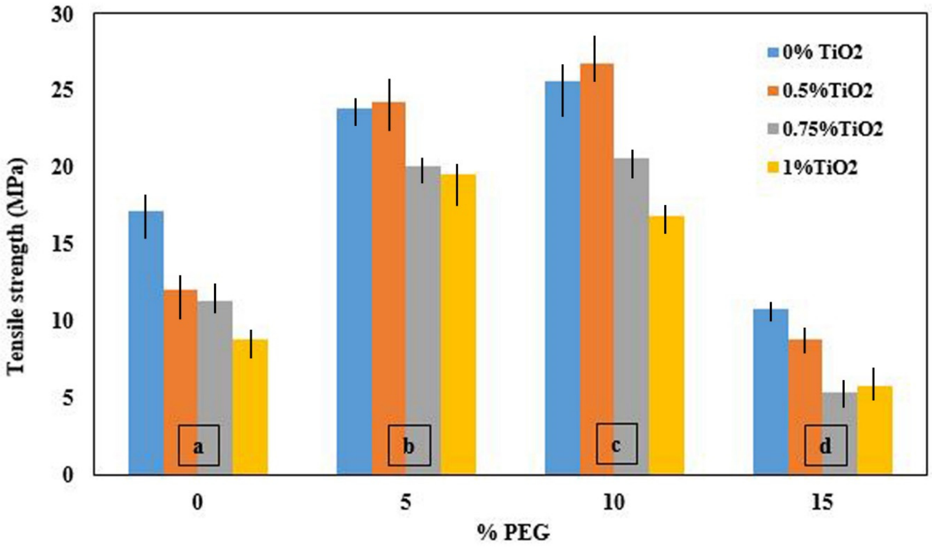

Fig. 5. Comparison of different wt. percentages between tensile strength and concentrations of nano TiO_2 and PEG as Reinforcement.

wt% causes the tensile strength to decrease to 11.3 and 8.85 MPa. The increment in the mechanical properties of the nanocomposite with the concentration of 0.5 wt% was due to the easiness of homogeneous dispersion of the TiO_2 nanoparticles in the polymer matrix. On thy other hand, the decrease with further addition of TiO_2 (over 0.5 wt%) is most likely caused by the aggregation of TiO_2 nanoparticles.

Fig. 5 (c) shows a similar version of variation of tensile strength for 10 wt% PEG and different concentrations of TiO_2 nanocomposite. It is apparent that the trend for the tensile strength variation at different concentrations of the TiO_2 nanoparticles is similar to the trends discussed in the above. It is worth noting, that the addition of 10% PEG increased tensile strength of pure UPE to 25.67 MPa and with 0.5% TiO_2, this value increases up to 26.74 MPa. Further addition of PEG concentration over 10wt% causes the tensile strength to reduce excessively. The result can be seen in Fig. 5 (d) The unsaturated polymer is very brittle and we have been able to improve its toughness with the addition of PEG (5% and 10%), but it is important to note that the increment obtained in toughness is often accompanied by a concomitant decrease in modulus and strength (15% PEG). Fig. 5 illustrates the synchronization of the graphical representations and the conclusions are drawn. The results show that the 10 wt% PEG and 0.5 wt% TiO_2 nanoparticles yield higher tensile strength due to good adhesion between fillers and polymer matrix which can be confirmed by the SEM image shown in Fig. 3.

Fig. 6 confirms these results. As shown in Fig. 6, the fracture toughness values of nanocomposites reinforced by PEG (10 wt%) are significantly larger than that of the other nanocomposite.

Fig. 7 shows the elongation of composite in the variation of TiO_2 and PEG concentrations. These results show that the polyester nanocomposites with 0.5 wt% TiO_2 nanoparticles and 10% PEG have a larger influence on the mechanical properties than other nanocomposites. The reason for the increase in mechanical properties can be attributed to the improved dispersion of TiO_2 nanoparticles within the polymer matrix. In addition, the existence of PEG in the matrix may have promoted a strong chemical interaction between the PEG and the polymer matrix.

TGA

The thermal degradation curves of pure UPE, UPE/PEG and UPE/PEG/TiO_2 were analyzed by TGA (Fig. 8). The results show a single stage of degradation, both for the pure polymer and for polymer nanocomposites. In addition, slight variations in start and end regions of degradation were observed, which indicates that there was an interaction between the TiO_2 and PEG with the polymeric matrix. The results proved that the thermal stability of the pure UPE was enhanced by

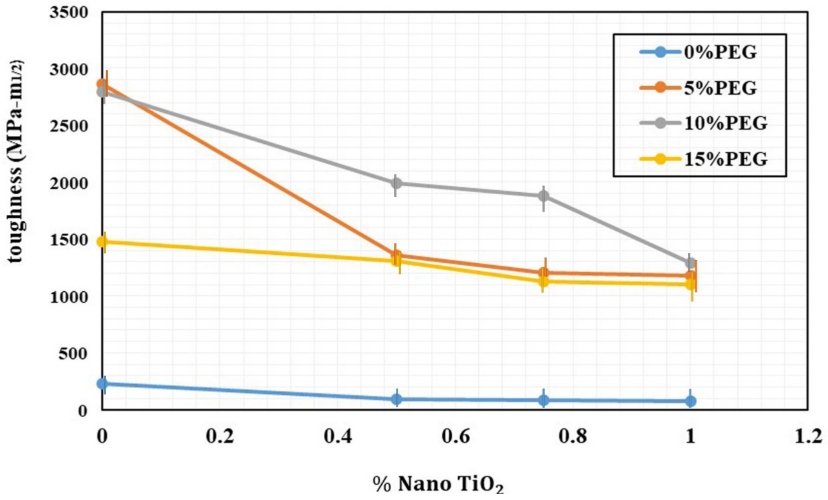

Fig. 6. Comparison of different wt. percentages between toughness and concentrations of nano TiO_2 and PEG as Reinforcement.

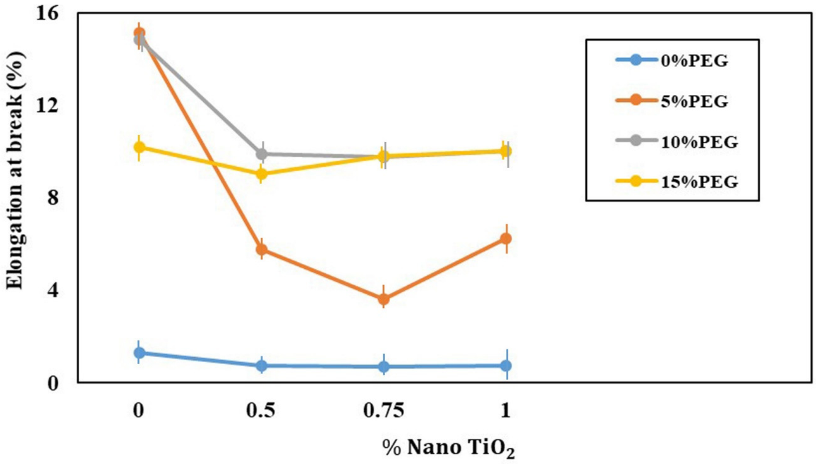

Fig. 7. Comparison of different wt. percentages between elongation and concentrations of nano TiO_2 and PEG as Reinforcement.

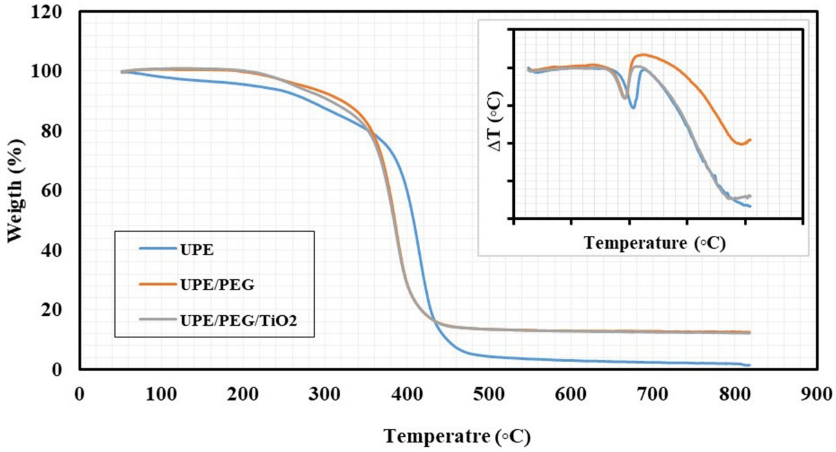

Fig. 8. TGA and DTA thermograms of UPE, UPE/PEG and UPE/PEG/TiO_2 nanocomposite.

Table 1. TGA data of pure UPE, UPE/PEG and UPE/PEG/TiO_2 nanocomposite.

Sample	Tg (°C)	Tm(°C)	Residue content%
UPE	360	420	2
UPE/PEG	340	390	12
UPE/PEG/TiO_2	340	380	12

the incorporation of TiO_2 nanoparticles and PEG. In addition, an increase in the residue content with the TiO_2 and PEG in polymer matrix was detected. Fig. 8 shows profiles of Differential thermal analysis (DTA) of UPE and composite at 0.5 wt% of TiO_2 and 10 wt% PEG. As shown in Fig. 8, inset shows that the addition of 0.5 wt% TiO_2 nanoparticle and 10 wt% PEG to the UPE matrix reduces the melting point and glass transition of composite. Basically, for the UPE/PEG/TiO_2 nanocomposite, *Tm* and *Tg* were observed that were lower and a residue content which was much higher compared the pure polymer due to the good interaction between fillers and matrix. (Table 1).

CONCLUSIONS

TiO_2 nanoparticles were synthesized through a modified sol–gel (SG) method. The UPE/PEG/TiO_2 nanocomposites were prepared at various concentrations of synthesized TiO_2 nanoparticles and PEG by direct mechanical mixing technique. On these nanocomposites, various tests were conducted to determine the mechanical, morphological and thermal properties of these composites. The tensile strengths, elongation and fracture toughness of UPE/PEG/TiO_2 nanocomposites were increased to a maximum, when nano-TiO_2 and PEG concentrations were 0.5wt% and 10wt% respectively and decreased with further addition of nano-TiO_2 and PEG.

The morphological properties of samples were also examined through SEM, XRD and IR. The results showed that when the nanoTiO_2 and PEG concentrations were 0.5wt% and 10wt% respectively, a homogeneous distribution of the fillers and a good compatibility with the polymeric matrix were observed.

The TG-DTA result also showed that the mentioned addition lowers the glass transition temperature and brittle fracture of UPR. In addition, brittle fracture of UPR is converted into ductile fracture with the addition of 10wt% PEG.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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