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Improving The Properties of a Polyester-Based Composite Using Nanoparticle Fillers

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HIGHLIGHTS

- The tensile test revealed that the latest mix has the better modulus of elasticity at (3% wt).
- This nanocomposite material showed very good roughness, hardness, flexural, and impact resistance.
- The good dispersion of the nanoparticles had a great role in having this material good properties as aggregation may lead to a decrease in the quality of the properties.

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ABSTRACT

In this work, unsaturated polyester, resin, matrix reinforced, with fiberglass mat chopped were developed using TiO_2 and SiO_2 Nanoparticles as filler materials. The individual and combined effects of nanoparticles on the nanocomposite and material properties were examined. An improvement in Young's modulus (39,3%) using combined weight of TiO_2+ SiO_2 was observed to be 3%. Examined nanoparticles have shown normal distribution in grain size indicated to be within standard limit, therefore boosting nanocomposite material to have excellent hardness, roughness, impact, and flexural resistance. DSC was characterized by transitions in this material. SEM and EDS techniques were used to confirm the formation of TiO_2 and SiO_2. The nanocomposites can be used in light-shielding, applications as well as in the industrial sectors.

1. Introduction

Composite materials can be characterized as heterogeneous materials comprised of at least two solid phases which are firmly interconnected. Specifically, nanocomposite materials are structured with nanoscale materials embedded in a ceramic, or polymer matrix at scales of 0-D, 1-D, and, 2-D.

The properties of nanocomposites depend on different factors, particularly grid material which can show measurements of the Nanoscale, the level of scattering, size, stacking, shape, direction of the Nanoscale second phase and interactions between the subsequent phase and the lattice. Nanomaterials have an ordinary grain size of under 100nm, while micromaterials are recognized by grain size of about 500nm. The unsaturated polyester resins are recently used in different applications like manufacturing the thermosetting polymers vital. Structurally, this resin fortifies with different fibers and fillers to improve mechanical and physical properties. Additionally, the chemical properties of this polymer rely upon the cross-linking agents, initiators, diced, and other additives. Many researchers have investigated this field over the last few years, M.T. Rahman et al., considered unsaturated polyester resin UPR, matrix-based polymer nanocomposites which were made by blending sol-gel Fe_2O_3, TiO_2 and co-precipitation synthesized $NiFe_2O_4$ nanoparticles. The mechanical, electrical, and optical properties of the fabricated polymer, nanocomposites were estimated. Their results indicated that both tensile and strength properties have increased in $NiFe_2O_4$/ UPR and Fe_2O_3+TiO_2+$NiFe_2O_4$ /UPR, Nanocomposite and Young modulus improved in $NiFe2O_4$/UPR composite only [1]. Sudad I. Younis et. al. have examined the effect of Nano, SiO_2, particles on wear demeanor test and other properties like ultimate tensile, strength, impact, strength, fracture, and hardness shore D of specimen's composite material.

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The best results were observed from unsaturated polyester resin matrix reinforcement with 4% weight fraction glass fiber, and 1%, 3% and 5% weight fraction of Nano SiO_2 particle. The results showed that the specimen UP+4% woven glass fiber+5% nano, SiO_2 gives preferable mechanical properties [2]. Suresh J.S et al. have investigated the effect of filler material like titanium oxide TiO_2, and silicon, carbide SiC, particulates on mechanical properties of Glass fiber. They found that the mechanical, properties like tensile, strength, flexural, strength, impact, strength, and hardness of the glass-reinforced composites are modified with the joining, of fillers [3]. T. Ahmed. et al. Examined SiO_2 Nanoparticles and glass fibers, in unsaturated, polyester. Mechanical tests like tensile, impact and micro-hardness were implemented on the obtained polymer hybrid, composites. The results of hybrid polymer matrix composites showed good development in mechanical properties [4]. Sudirman et al. Investigated, the effects of silica, as filler. The investigation appeared that the mechanical properties like tensile, strength, elongation, and Young's modulus, increase with the addendum of SiO_2 nanoparticle up to 1.0 wt%, and then decrease, over 1.0 wt% [5]. In this study, the main objective is to improve the properties of a polyester-based composite using nanoparticle fillers. The (Unsaturated Polyester, Resin) (UPR), is used as the matrix which was tested alone and then mixed with other materials to select the one that has better mechanical properties. The unsaturated polyester resin was reinforced with mat glass fiber. The Titanium Dioxide TiO_2 and Silicon Dioxide SiO_2 Nanoparticles are utilized as fillers in the planning of test polymer composite materials provided by Hongwu International, Group (Ltd). The purity of TiO_2 and SiO_2 are (99.9% and 99.8%) respectively. While the particle size of TiO_2 and SiO_2 are 30-50$\pm$5 nm [6] and 20-30$\pm$5 nm [7] respectively.

2. Materials and Methods

2.1 Materials

Unsaturated Polyester Resin, glass fiber, methyl ethyl, ketone per Oxide, TiO_2, and nanoparticles SiO_2 Nanoparticles were used as filler.

2.2 Methods

in order to synthesize composite materials, several specimens were prepared with different materials as follows:

1. Pure polyester: Unsaturated polyester resin which is a clear liquid had been used for casting. It has a density equal to 1.15gm/cm^3 and a viscosity of 450 mPa.s. It is elastic with medium reactivity. The most common usage is the production of marbles by adding up filling materials in the proportion of 70-80%. Furthermore, it can be safely used in the production of cuvettes, sinks, kitchen counters, table edges-legs, buttons, ornaments, etc. [8]. As UPR is viscous and liquid, 20,wt% methyl, ethyl ketone perOxide, (MEKP) [9] it was added as a curing agent hardener and blended very well at ambient temperature.
2. UPR reinforced with mat glass fiber: The mat glass fiber (chopped) with a density equal to 2g/cm^3 was added to the mix of (UPR) and hardener where the mold should be covered with wax matrix to avoid abrasive and insure flattening. Then roller and brush were used to apply pressure for distributing the mix resin and hardener through the fiber then another layer of the mix was added on the fiber then for ensuring air removal and wet out, a torch is passed near the mixed surface.
3. UPR reinforced with glass fiber (mat) with TiO_2 Nanoparticles as filler: Unsaturated polyester resin-based polymer nanocomposites were manufactured by using 1 wt%, 2 wt%, and 3 wt% of Titanium Dioxide Nanoparticles respectively. Where the nanoparticles were added to the UPR in a plastic vessel and the mix were blended using a blending device mixer drill with an angular velocity of 750 cycle/minute, then the hardener was added to the mix and blended in the same blending device for 5 minutes to make the mixture ready for casting and the reinforcing fiber was added in the same way that previously mentioned.
4. UPR reinforced with mat glass fiber with SiO_2 Nanoparticles as filler: The UPR based polymer nanocomposites were fabricated by using 1 wt%, 2 wt%, and 3 wt%) of Silicon Dioxide and the method of preparation was the same as the preview method with using SiO_2 instead of TiO_2.
5. UPR reinforced with glass fiber (mat) and both TiO_2 and SiO_2 Nanoparticles are used as filler and the reinforcing fiber was added in the same way that previously mentioned. UPR based polymer nanocomposites were fabricated by using 1wt%, 2 wt%, and 3 wt% of both Titanium Dioxide and Silicon Dioxide nanoparticles respectively, and in the same method of mixing used before mixing drill. After completing blending the mix UPR, TiO_2, and SiO_2 and adding hardener, fiber was added in the same way of fabricating fiberglass/polyester. All the specimens were treated at a temperature 27^{o}C and for 24 hr period of time.

2.3 Tensile Test

Tensile tests were used to determine the modulus of elasticity and the tensile strength for each specimen. All test specimens were castin a specially designated mold made from steel as shown in Figure 1-(A) according to the standard geometry of ASTM D 638 [10, 11] as shown in Figure 1-(B) and its dimensions in Table1.

Thirty-three tensile test specimens were prepared for the materials previously mentioned which are three tensile test specimens of UPR as shown in Figure 2-(A), three of UPR+ chopped glass fiber as shown in Figure 2-(B), three of UPR+ glass fiber+ 1% TiO_2 as shown in Figure 2-(C), three of UPR+ glass fiber+ 2% TiO_2 as shown in Figure 2-(D), three of UPR+ glass fiber+ 3% TiO_2 as shown in Figure 2-(E), three of UPR+ glass fiber+ 1% SiO_2 as shown in Figure 2-(F), three of UPR+ glass fiber+ 2% SiO_2 as shown in Figure 2-(G), Three of UPR+ glass fiber+ 3% SiO_2 as shown in Figure 2-(H), three of mix include UPR+ glass Fiber+ 1% TiO2),+ SiO_2 as shown in Figure 2-(I), three of mix include UPR+ glass fiber+ 2% TiO_2,+ SiO_2 as shown in Figure 2-(J), and three of mix include UPR+ glass fiber+ 3% TiO2,+ SiO_2 as shown in Figure 2-(K). The

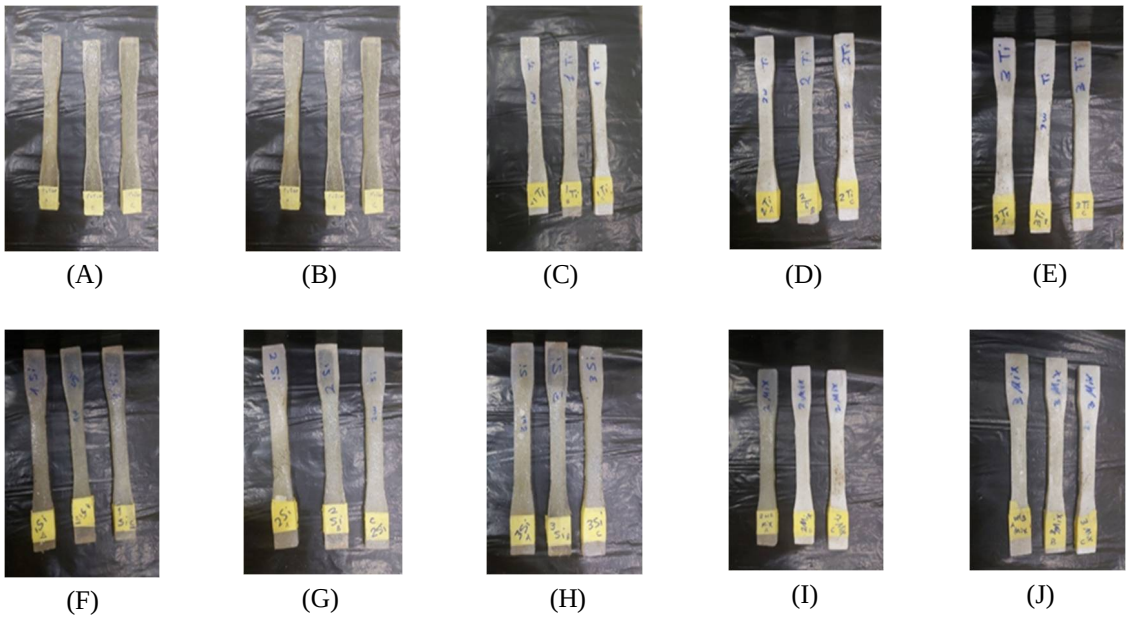
tensile test specimen is mounted vertically in a servo-hydraulic testing machine, and is hydraulically retracted using stroke control with large steel grips). The specimen which is a mix of (UPR+ Fiber+ 3% TiO_2+ SiO_2 revealed the best behavior where it has the highest modulus of elasticity at 1.5 wt% TiO_2+ 1.5 wt% SiO_2 and according to that, It had been chosen to be studied in detail, in addition to the tensile test, other tests have been done for the chosen material to get a sufficient idea of its properties where these tests are:

2.4 Flexural Test

The flexural test was carried out at room temperature by utilizing the all-inclusive test instrument that equivalent utilizing in the ductile test relied on a three-point, bending test technique.

(A) (B)

Figure 1: (A) Tensile test specimens mold (B) Standard geometry of ASTM (D 638)



(A) (B) (C) (D) (E)

(F) (G) (H) (I) (J)

Figure 2: Tensile test specimens for (A) UPR, (B) UPR+ chopped glass fiber, (C) UPR+ glass fiber+ 1% TiO_2, (D) UPR+ glass fiber+ 2% TiO_2, (E) UPR+ glass fiber+ 3% TiO_2, (F) UPR+ glass fiber+ 1% SiO_2, (G) UPR+ 2% SiO_2, (H) UPR+ glass fiber+ 3% SiO_2, (I) mix include UPR+ glass fiber+ 1% TiO_2,+ SiO_2, (J) mix include UPR+ glass fiber+ 2% TiO_2,+ SiO_2, and (K) mix include UPR+ glass fiber+ 3% TiO_2,+ SiO_2

Table 1: Specimen dimensions standard geometry of ASTM (D 638)

Symbols	Description	Dimension (mm)
W	Width of narrow section	12.5
L	Length of narrow section	75
WO	Width overall	20
LO	Length overall	165
G	Gage length	50
D	Distance between grips	115
R	Radius of fillet	20
R2	Radius of fillet	6.5
T	Thickness	3or under

In this strategy the perpendicular load continuously at the center of the Nanocomposite samples at strain, rate, 2mm/min. until a fracture happens to acquire the (load-displacement) curvature for the composite specimen. The flexural modulus and flexural strength are the properties acquired from this test for composite example was proposed in this examination as per the

global norm ASTM D-790, [11, 13] as in the schematic shown in Figure 3A. Figure 3B shows the experimental flexural test specimen.

2.5 The Impact Test

Impact testing is carried out according to international standards (ISO-180), [11, 12, 14] by utilizing (Izod) Impact, test machine type is (XJU)series pendulum (Izod/Charpy) impact testing machine). For (Izod test): the samples were clipped toward one side and held upward cantilevered, beam. Impact test samples may be with or without, snick. Figure 4. (A, B, C) shows the standard sample of the impact test.

2.6 Hardness Test

Hardness is the most popular calculated property of the surface. The shore hardness is measured with an apparatus recognized as a "Durometer hardness". The value of hardness is specified by the penetration, of the Durometer, indenter foot into the Specimen. Due to the versatility of Rubbers, and plastics, the indentation, reading may change, over time so the indentation, time is sometimes recorded along with the hardness number.

Shore (D) Hardness is a standardized, test consisting in measuring, the depth of penetration of a specific, indenter. Test methods used to measure Shore (D) Hardness are ASTM D2240 and (ISO 868) [15, 16].

2.7 Roughness Test

Surface roughness is characterized as the anomalies which are inherent in the production process (for example, cutting instrument or abrasive grit). Since the individual, roughness inconsistencies are very, small to see with the bare, eye and a roughness estimating, the instrument is required. A little pointer is drawn across the surface at a consistent, speed for a set distance. An electrical, signal is gotten and intensified to create a much-amplified vertical amplification. This sign might be shown, on both chart and screen, outputs, together, with mathematical qualities that give details of the surface as shown in Figure 5.

2.8 Density calculation

Density is the mass per unit volume of a material. The total density (ρ) was determined by taking a piece of the laminate and measuring its volume and mass as shown in Figure 6.

2.9 Water Absorption Test

Different polymeric materials are powerless to water retention during their life exposures which cause dimensional insecurity, as per that this test is essential to do. The test of water absorption is achieved according to (ASTM D570) [17, 18]. After putting the specimen in distilled water for 24 hr, The water absorption can be calculated according to the following equation [17]:

$$\text{Water absorption percentage} = [(Ws-Wd)/Wd] *100 \quad (1)$$

Where: Wd: Mass of the sample before immersion dry.
Ws: after immersion for 24 hr. in distilled water.

Figure 3: (A) (Schematic for Standard Flexural Test Specimen) , (B) (Experimental Flexural Test Specimen before Testing

Figure 4: (A) Schematic for Standard Impact Test Specimen, (B) Impact Test Specimen

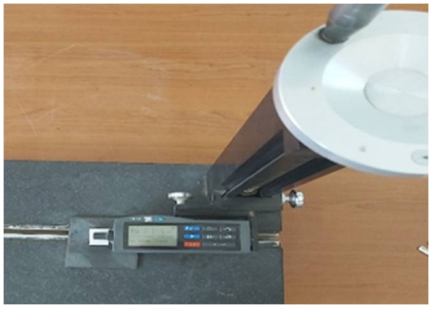


Figure 5: Roughness test device

Figure 6: Measuring the piece's mass

2.10 Antiseptic Resistance Test

The importance of this test appears greatly in the materials entering the manufacture of medical devices that are subjected to periodic sterilization, and it is a test in which a sample of a substance is placed in a sterilizing material for a period of 24 hours to check its resistance.

2.11 Grain Size Test for Nanomaterials

Silicon dioxide SiO_2 and Titanium Dioxide TiO_2 nanoparticles samples were taken from both materials and examined at the Nanotechnology Research Center at the University of Technology to ensure that the granular size is in conformity with the certificate of origin or within the limits of the nanoparticle size and that there was no clumping in the nanomaterial, where the titanium dioxide was examined by trying to dissolve it with water, and it became clear that it was A water-repellent substance that did not dissolve and because of that, the water was replaced with ethanol as a solvent, and the solution was applied to a dispersion device as shown in Figure 7, for a minute and a half, and then examined with a grain size analyzer device which is shown in Figure 8, also SiO_2 was dissolved with water and dispersed by a dispersion device for three minutes and then examined with a grain size analyzer device.

2.12 Differential Scanning Calorimetry DSC

Differential Scanning Calorimetry DSC utilized for quite a while to portray, changes in materials. It is just normal, a sense that it is vital, to know when, on the “baseline” of the information, does the progress, starts or ends. By knowing the baseline, the proper, the legitimate, joining, cutoff points can be chosen, and a precise, estimation of the warmth related, with the progress can be made. DSC can be utilized to gauge the glass transition (T_g) of material because there is a step increase in heat capacity as the sample is heated through its glass transition temperature. But there is more above the glass transition, the polymers, have a great deal of versatility. They wobble and squirm), also, never stay in one situation for extremely long. At the point when they arrive at the correct temperature, they will have acquired sufficient energy to move into exceptionally requested plans, which we call crystals, obviously when polymers fall into these crystalline arrangements, they give off heat. This drop appears in the heat flow as a major plunge in the plot of heat flow versus temperature which is considered as crystallization temperature (T_c). If the polymer kept heating and past its (T_c), eventually it'll reach another thermal transition, one called melting temperature (T_m) which is considered as the big peak on(DS) plot.

2.13 (SEM) Scanning Electron Microscopy

SEM demonstrated the apportionment, and fortifying mechanism, by nanoparticles and glass strings in unsaturated, polyester resin. Scanning electron microscopy, SEM micrographs were done, to the mix of the nanocomposite, samples at different, magnification (700x, 1600x, 2200x, and 2400x).

2.14 Energy Dispersive Spectroscopy (EDS)

Energy Dispersive X-ray Spectroscopy EDXS, also known as EDX analysis, and EDS analysis, is a qualitative, and semi-quantitative, X-ray microanalytical, technique that can give data, about the essential, creation of an example. It is valuable in recognizing metals and particular sorts of polymeric materials with special essential, marks). Like the electron, a beam of the SEM is scanned, across the specimen, surface, it generates, X-ray fluorescence, from the atoms in its path. The energy of each X-ray photon is characteristic of the element that produced it. The EDS microanalysis system collects the X-rays, sorts and plots them by energy, and autOmatically, identifies and labels the elements responsible for the peaks in this energy, distribution.

3. Results and Discussion

3.1 Tensile test

The mechanical properties for all specimens obtained from the tensile test are shown in Tabel 2 below:

From the results above reinforcing UPR with fiberglass could highly increase the modulus of elasticity and the tensile strength with percentage reaches to 47% and 57.96% respectively. On the other hand, adding fillers of nanomaterials like TiO_2 and SiO_2 showed better modulus of elasticity at a weight ratio of 1% and 2% respectively, while the 3wt % revealed decreasing in modulus of elasticity value with percentage reaches to 13.9% and 6.86% in TiO_2 and SiO_2 respectively where this decrease could happen due to some regions of aggregation.

3.2 Flexural test

Figure 9 revealed the load-extension curve obtained from the flexural test. According to the information obtained from this curve, the magnitude of the flexural strength, flexural modulus, and maximum shear stress are gained as shown in Table 3.

3.3 Impact test

The impact results represent the impact strength for the composite specimen prepared in this study which is equal to 5.5 KJ/m^2 and it has broken, at an impact energy of 2.2 J of the pendulum, and impact velocity of 3.5 m/s.

3.4 Hardness test

The hardness esteem was dictated by the infiltration, of the Durometer, indenter foot into the example. Shore hardness measures are dimensionless. It goes between 0 and 100. The higher, number represents the harder, material. Hardness Shore-D =86 which is considered extra hard due to the durometer shore hardness scale.

Figure 7: Dispersion device

Figure 8: Grain size analyzer

Table 2: Experimental mechanical properties of tensile test

Material	**Mechanical properties**					
	1%		2%		3%	
	Young modulus (Mpa)	Tensile strength (Mpa)	Young modulus (Mpa)	Tensile strength (Mpa)	Young modulus (Mpa)	Tensile strength (Mpa)
Mix of	900	39.75	984.99	30.625	1000	46.388
UPR+ Fiber+	666.6	22.5	750	53	964.28	40.5
TiO2+ SiO2	500	42.77	666.66	42.22	1111.11	45
Mix average	688.866	35	600.53	41.74	1025.13	43.96
UPR+ Fiber+	750	52.33	666.6	28.333	500	30.138
TiO2	533.3	17.22	833.33	43.611	666.66	37.333
	750	23.5	500	54.666	583	30.916
UPR+ Fiber+ TiO2 average	677.766	31	666.64	42.22	583.22	32.79
	675	19	800	35	680.5	32.5
UPR+ Fiber+	600.6	29	749	44	750	46.5
SiO2	714.28	24.375	833.3	44.166	788.288	61.25
UPR+ Fiber+ SiO2 average	663.29	24.125	794.1	41	739.596	46.75
	486.11	22				
Pure UPR	270	17.66				
	333.3	29.25				
	800	34.5				
Pure UPR average	397.35	25.85				
UPR + fiber	880	51				
	750	66.5				
	622.22	67				
UPR + fiber average	750.74	61.5				

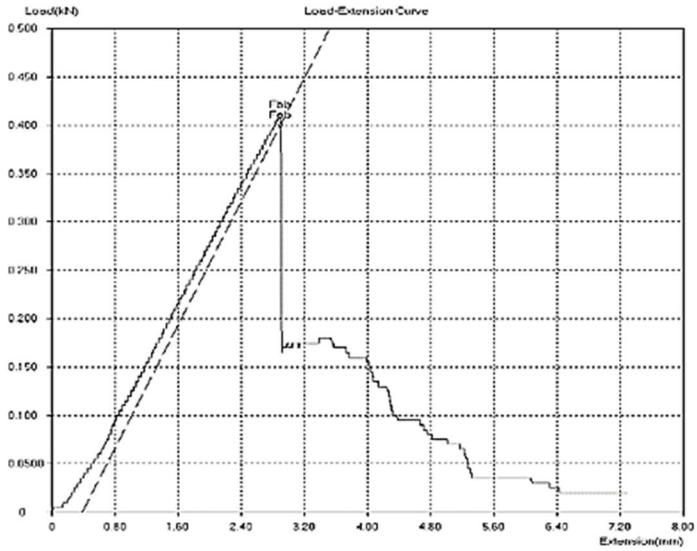


Figure 9: Load-extension curve of flexural test

3.5 Roughness test

The examination of roughness was carried out in the laboratories of the Department of Materials Engineering at the University of Technology, where three readings were taken on the sample surface and these three readings are 2.316, 2.315 and 2.316 nm respectively, where it's clear that the suitable value is equal to 2.316 nm.

3.6 Density calculation

According to the dimension of the specimen, and according to the measured mass, the density is obtained is ρ = 1.547 $\frac{g}{cm^3}$

3.7 Water absorption measurement

For measuring the water absorption, after measuring the mass of the sample before immersion dry which is equal to 6.767 and the mass of the sample after immersion for 24 hr in distilled water which is equal to 6.825, the Water absorption could be obtained as follows: Water absorption percentage $\frac{6.825-6.767}{6.767}$ *100 = 0.8571 %

3.8 Antiseptic resistance test

According to the antiseptic resistance test the sample was placed in the substance of propanol with a density of 0.804 g/mL for 24 hours. After the sample was taken out, it was found to be completely resistant to sterilization materials and no effect occurred in it.

3.9 Grain size test

From the grain size test, it became clear that the granular size for TiO_2 is 34.3 nm as revealed from the grain size summary in figure 10. On the other hand, the grain size test summary shown in figure 11 for SiO_2 revealed that the granular size is 28.8nm, where it is within the limits of the certificate of origin previously mentioned.

3.10 DSC test

DSC test gives the curve related to the heat flow to the temperature as shown in Figure 12. The step appears in the plot when the polymer was heated, past its glass transition temperature at 70°C. Then it showed a major, plunge when the polymer reached its crystallization temperature at 159°C. Then finally it revealed a big peak when the polymer, reached its melting temperature at 184°C.

Table 3: Flexural test results

Flexural properties	magnitude
Flexural strength (MPa)	19.212
Flexural modulus (MPa)	607.15
Maximum shear stress (MPa)	1.729

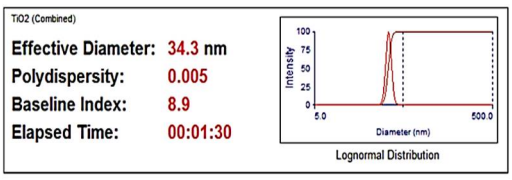


Figure 10: grain size test summary for (TiO_2)

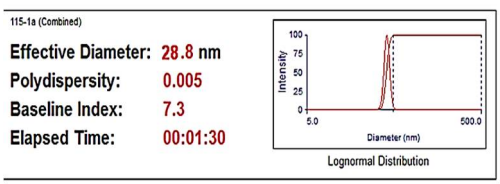


Figure 11: grain size test summary for (SiO_2)

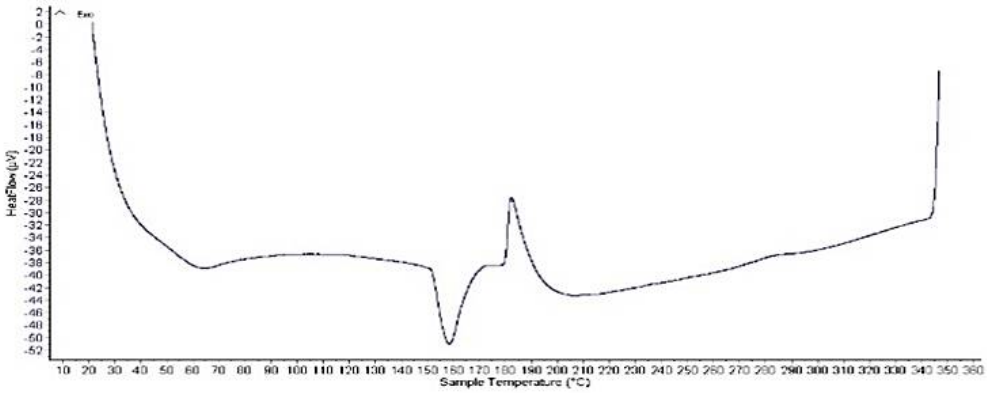


Figure 12: DSC curve

3.11 SEM test

Figure 13 shows the SEM test. This figure shows the presence of glass fibers embedded in polymer matrix and these fibers provide strengthening mechanism to polymer in all directions . SEM micrographs were done to the mix of nanocomposite samples at different magnification 700x, 1600x, 2200x and 2400x were recorded in Figure 13 A, B, C and D respectively. as shown from this figure, in most cases, the system appears to have a semi-continuous morphology, observed for different magnifications. The fibers were buried inside the polymeric matrix material, and that most of the fiber became part of the matrix material which seems to indicate better interfacial adhesion between fibers materials and components of composite materials.

The filler materials Nanoparticles appeared as the white molecule, where the presence, of bigger particles, is because of the molecule, accumulation. Huge district of conglomeration could be feeble, communication between the nanoparticles and the polymer, phase causes a few voids, on the network surface. This condition altogether diminished the mechanical properties of the composite, henceforth to stay away from that, great scattering in low convergence of nanoparticles, particularly, silica in the UPR framework plays a fundamental, job to give all-around shaped, interpenetrating, network IPN structures.

3.12 EDS test

To affirm the arrangement SiO_2-TiO_2, EDS analysis was executed successfully. During the EDS measurement different areas were focused and the corresponding peaks are shown in Figure 14. Both SiO_2 and TiO_2 can be seen in the synthesized composite nanostructure in the EDS spectrum. In this spectrum, the weight composition of Ti, Si, and O were 4.17, 4.86, and 90.97 respectively. Details of the EDS spectra of the nanocomposite worth, measured in atomic and weight % are listed in Table 4.

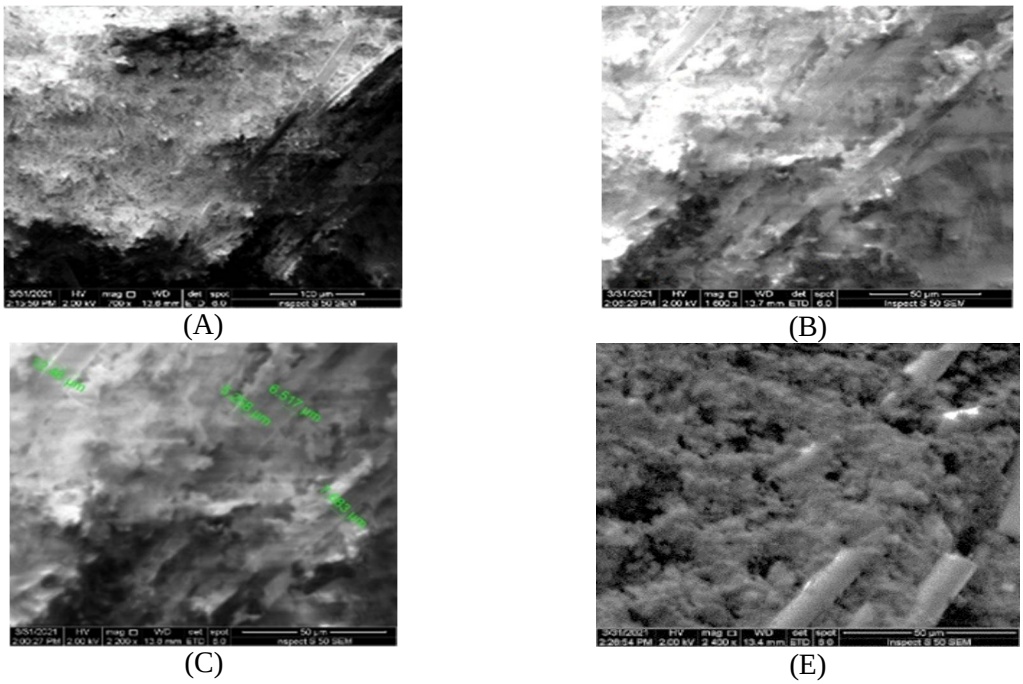


Figure 13: SEM test with different magnification (A):700x, (B):1600x, (C):2200x, (D):2400x

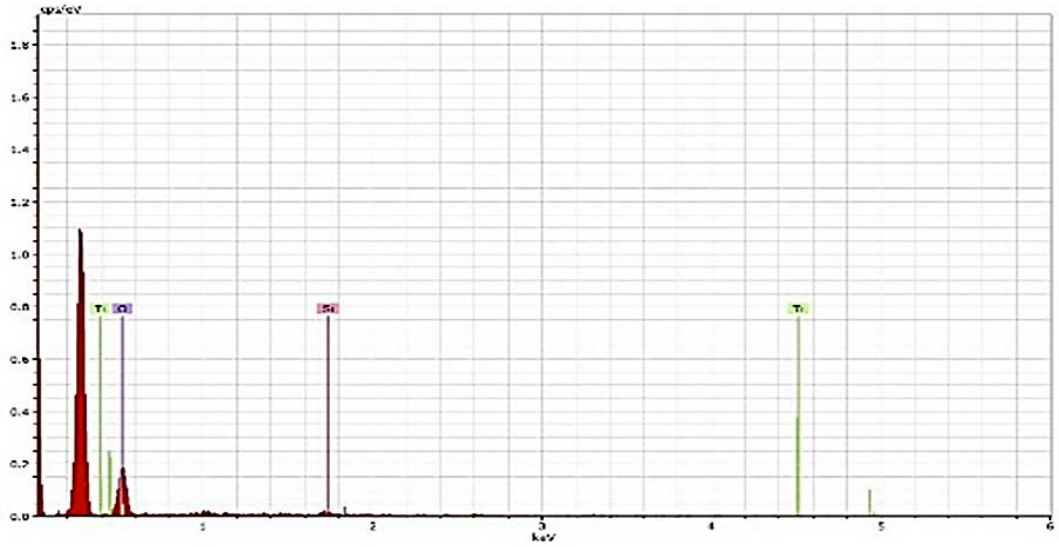


Figure 14: The EDS spectra

Table 4: Details of the EDS spectra

Spectrum: acquisition 3970						
EL	AN	Series	Unn. [Wt.%]	norm [Wt.%]	CAtom. [at.%]	C Error(1Sigma) [Wt.%]
O	8	K-Series	90.97	90.97	95.63	27.01
Si	14	K-Series	4.86	4.86	2.91	0.87
Ti	22	K-Series	4.17	4.17	1.47	1.92
Total:				100.00	100.00	100.00

4. Conclusions

In this study, UPR, UPR reinforced with fiberglass mat chopped, UPR reinforced with fiberglass and SiO_2 or TiO_2 with 1%, 2%, and 3% weight percentage used as a nanoparticles filler, and UPR reinforced with fiberglass and SiO_2 and TiO_2 with 1%, 2%, and 3% weight percentage used as a nanoparticles filler nanocomposites were prepared by casting technique. The tensile test revealed that the latest mix has the better modulus of elasticity at 3% wt and according to that other mechanical properties were gained and other tests were done to examine the resistance and the structure of this nanocomposite material. This nanocomposite material showed very good roughness, hardness, flexural, and impact resistance. In addition, it showed not affected by sterilization materials, up to 100%. On the other hand, the good dispersion of the nanoparticles had a great role in having this material good properties as aggregation may lead to a decrease in the quality of the properties. On the premise, of the acquired, result, the created, nanocomposites can be utilized to deliver differently, items in (medical, structures development, auto, and so forth areas with better, properties. These nanocomposites can likewise be utilized as light safeguarding, applications, and covering material.

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Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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