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The Effect of Nano Sized TiO_2 Particles on Improving the Stress Resistance and Thermal Stability of Unsaturated Polyester

Rana J. Mizban[1*] and **Widad H. Jassim**[2]

[1,2] Department of Physics, College of Education for Pure Science (Ibn Al-Haitham), University of Baghdad, Baghdad, Iraq.

* Corresponding Author

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Abstract

Unsaturated polyester's (UPE) mechanical and thermal properties were modified by incorporating nanosized titanium dioxide (TiO_2) particles with 1, 2, 3, 4, and 5% weight ratios. 3% of TiO_2 is the best weight percentage that can be added to UPE to improve its impact strength from 8.74 MPa to 8.87 MPa, its hardness value from 82.23% to 86.63%, its tensile strength from 7.698 MPa to 24.76 MPa, and its thermal conductivity from 0.308499 W/m.^{o}C to 0.566916 W/m^{o}C. Also, the thermal stability of UPE/3% TiO_2 was improved using thermogravimetric analysis (TGA). XRD measurement was employed to verify the change caused by adding TiO_2 particles to the internal structure of unsaturated polyester. Also, the change in the microstructure of unsaturated polyester after adding TiO_2 nanoparticles was studied using scanning electron microscope analysis, where the TiO_2 nanoparticles showed a good distribution in the matrix, and the TiO_2 had good compatibility in the unsaturated polyester matrix.

Keywords: Unsaturated polyester, Titanium dioxide, Mechanical tests, Thermal conductivity, Thermal stability.

1. Introduction

A composite is a mixture of two or more materials of different properties to get a material with new physical and chemical properties. There are two phases in composite material: the continuing (matrix) phase, like ceramic, polymer, and metal, and the reinforcing phase, like the fibers, particles, laminates, or fillers (1–5). Many composites have high tensile strength, good hardness, low density, good stiffness, and high performance at high wear resistance and high temperature, and these properties depend on the nature of the interface between the two phases (6–8). Polymer composite materials have a wide range of applications in engineering fields using a matrix made from resins such as epoxies or unsaturated polyesters to obtain the best features. These resins are reinforced with different kinds of fillers to change the physical, mechanical, and thermal properties of this polymer composite (9–12). Unsaturated polyester is used as an inexpensive matrix and a good adhesive in many new life applications. Some synthetic or natural fillers have been used to reinforce this resin and to improve its mechanical properties. TiO_2 is used as reinforcement in a polymeric matrix (13).

The researchers (14) have used titanium dioxide (TiO_2) as a synthetic filler with a nano size, which is compatible with unsaturated polyester. Composite impact strength and toughness improve when compared with pure unsaturated polyester, because the TiO_2 particles and unsaturated polyester are flexible. In (15) used TiO_2 as a reinforcement agent to improve the resin strength against mechanical tests, and they studied the influence of parameters such as matrix kind, additives, formation methods, time, temperature, and also pressure on the mechanical strength of the resin matrix loaded with TiO_2 particles.
In this research, XRD measurement and scanning electron microscopy were employed to study the microstructure of unsaturated polyester before and after incorporating nanosized particles (TiO2) in the appropriate ratio. The microstructure of the unsaturated polyester composite was changed, leading to improvements in mechanical and thermal.

2. Materials and Methods

2.1. Unsaturated Polyester

Unsaturated polyester (UPE) with a density of 1.04 g/cm^3 has been used, and after adding its hardener (MEKP) in the ratio of 2% g and 2% g of cobalt as an accelerator, the unsaturated polyester transforms from a transparent liquid into a homogenous solid-state polymer after 5 minutes of mixing at room temperature.

2.2. Titanium Dioxide (TiO_2)

TiO_2 powder has a particle size of 46.3 nm and a 4.23 g/cm3 density. It has been used as reinforcement.

2.3. Method

The unsaturated polyester resin was reinforced by weight ratios (1, 2, 3, 4, and 5 % of TiO_2) powder to prepare the (unsaturated polyester/TiO_2) mixtures. These mixtures were poured into the silicone molds with dimensions matching the dimensions for each mechanical test. The samples were left in the molds for two hours before mechanical tests.

2.3.1. Mechanical tests

2.3.1.1. Impact test

An impact test (Charpy) instrument has been carried out on the samples with dimensions (ISO-179).

2.3.1.2. Tensile test

A tensile test was carried out on samples that were molded according to ASTM D-638 at room temperature using a microcomputer-controlled electronic universal testing machine (WDW-200E) made in China.

2.3.1.3. Flexural strength

The flexural strength of unsaturated polyester/TiO_2 composites was determined on samples that were cast according to ASTM D790 using a three-point bending test.

2.3.1.4. Hardness test

The TH2IO-Shore (D) device was employed on the three specimens.

2.3.2. Thermal tests

2.3.2.1. Thermal Conductivity

The thermal conductivity of unsaturated polyester and UPE/TiO_2 composites was determined using Lee’s disk device.

2.3.2.2. Thermo-gravimetric (TGA)

The TGA test was performed on pure unsaturated polyester and on unsaturated polyester reinforced by TiO_2 to determine their thermal stability. The testing was carried out using the instrument Shimadzu DTG-50, and the samples were heated from 20 to 600 °C with a heating rate of 10 °C/min).

2.3.3. Structural tests

2.3.3.1. XRD

The XRD technique was employed to study the microstructure of unsaturated polyester and unsaturated polyester reinforced by TiO_2 using an XRD-6000, Shimadzu instrument.

2.3.3.2. Scanning electron microscope (SEM*)*

To study the microstructure of unsaturated polyester and the UPE/TiO_2 composite and determine their degree of homogeneity, an electronic scanning device (Tescan) manufactured in England was used.

3. Result and Discussions

3.1. Mechanical Properties

3.1.1. Impact strength

Figure (1) and **Table (1**) explain that the impact strength value of unsaturated polyester is 8.74 MPa. This value will decrease by adding TiO_2 particles with lower weight percentages (1% and 2%). This is because the unsaturated polyester chains are stretched around TiO_2 particles, and the rate of stress transfer between the TiO_2 and resin is very low, tan to the lowest in the values of composite impact strength (14).

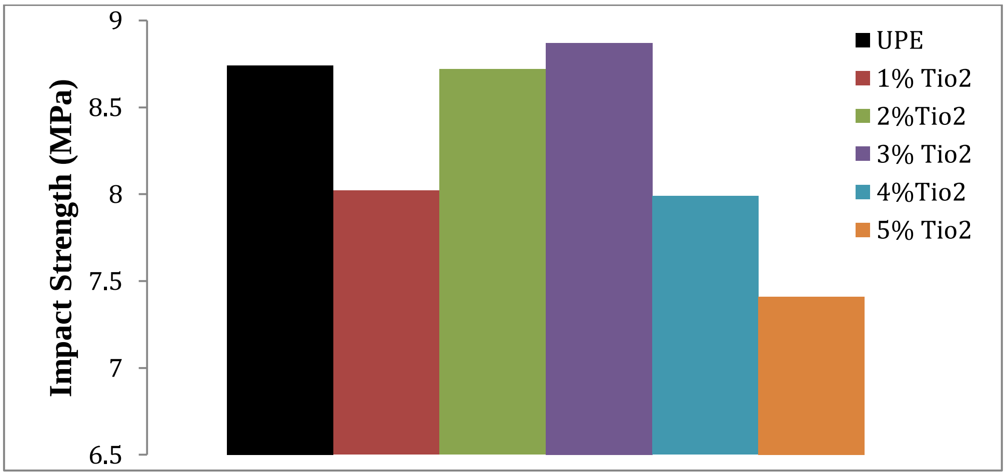

Figure 1. Impact strength values of UPE and (UPE/TiO2) composites.

Table 1. Impact strength maximum values of UPE and (UPE/TiO2) composites.

Sample	Impact strength maximum values
UPE	8.74
1% TiO_2	8.02
2% TiO_2	8.72
3% TiO_2	0.07
4% TiO_2	7.99
5% TiO_2	7.41

By increasing filler content in unsaturated polyester to a weight percentage of 3%), the impact strength value increases to a higher value (8.87 MPa), which means the bonding between the filler and matrix will improve. Also, the good flexibility of the interface between the filler and matrix leads to more absorption and dispersing of energy and prevents the initiation of cracks (16).

Then the impact strength value will decrease and reach a lower value (7.41 MPa) by reinforcing unsaturated polyester with 5% filler. This is because of the agglomeration effect, which leads to the weakness of the interfacial area, and also because the agglomerates act as strength concentration sites or crack initiation points. These results agree with (17, 18).

Increasing the weight percentage of TiO_2 filler in unsaturated polyester (UPE) samples can lead to increased strength and improved bonding between the filler and the matrix through several mechanisms:

- At lower weight percentages (1% and 2%), the unsaturated polyester chains are stretched around TiO_2 particles, resulting in a low rate of stress transfer between the filler and resin. This weakens the composite impact strength. However, as the filler content increases to 3%, the bonding between the filler and matrix improves, indicating better interaction between TiO_2 particles and the UPE matrix.
- A higher weight percentage of filler (3%) may lead to the development of a flexible interface between the filler and matrix. This flexibility allows for better energy absorption and dispersal, preventing crack initiation and propagation, thereby enhancing the impact strength of the composite.
- At higher weight percentages of filler (5%), the agglomeration of TiO2 particles may occur, leading to the formation of clusters within the composite. This agglomeration weakens the interfacial area between the filler and matrix, reducing the bonding strength and resulting in a decrease in impact strength.

3.1.2. Hardness test

The resistance of any material surface to indentation is its hardness (13,19). **Figure (2)** and **Table (2)** illustrate that the hardness values of unsaturated polyester reinforced with TiO_2 increase with an increase in TiO_2 weight percentage. This is because of the good dispersion of TiO_2 particles and the decrease in inter-TiO_2 particle distance in the unsaturated polyester, which leads to an increase in hardness. The unsaturated polyester filled with TiO_2 has a high hardness compared to the pure unsaturated polyester, which is because TiO_2 particles are harder than the unsaturated polyester. The results agreed well with (17).

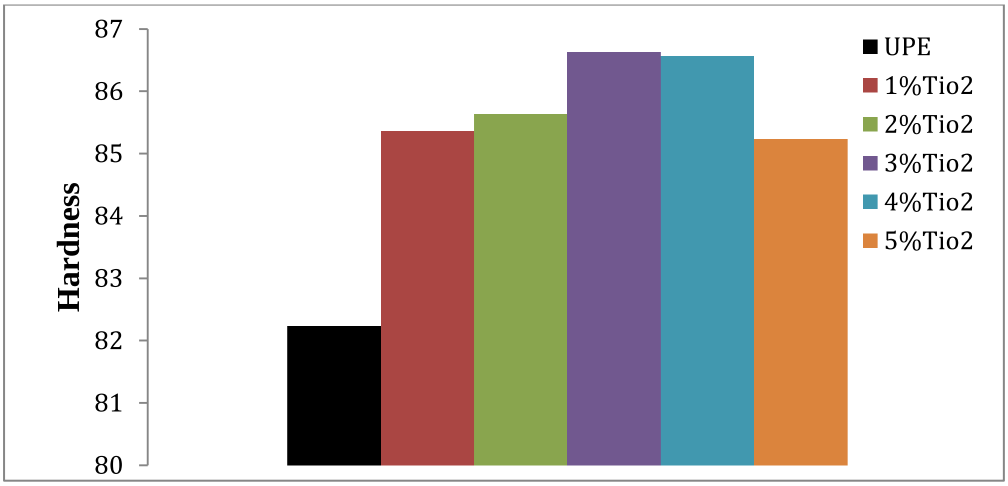

Figure 2. Hardness values of UPE and (UPE/TiO_2) composite.

Table 2. Hardness maximum values of UPE and (UPE/TiO_2) composites.

Sample	Hardness maximum values
UPE	82.23
1% TiO_2	85.36
2% TiO_2	85.63
3% TiO_2	86.63
4% TiO_2	86.56
5% TiO_2	85.23

The hardness shore (D) value improved from 82.23% for pure unsaturated polyester to a higher value (86.63%) for the UPE/3% TiO_2 composite because of the compatibility between TiO_2 and unsaturated polyester and the good dispersion of nanoparticles of TiO_2 in the interface region between the matrix chains and TiO_2 particles. These results are consistent with (16, 20).

The improvement in hardness for the reinforced samples was demonstrated by comparing the hardness values of pure unsaturated polyester (UPE) and UPE reinforced with TiO_2 particles.

- The hardness shore (D) value for pure unsaturated polyester (UPE) was 82.23%.
- The hardness shore (D) value for the composite UPE/3% TiO_2 was 86.63%.

Therefore, the addition of 3% TiO_2 to the unsaturated polyester resulted in an increase in hardness from 82.23% to 86.63%.

3.1.3. Tensile strength

Figure (3) and **Table (3)** explain how the addition of TiO_2 filler oil improves the tensile strength of unsaturated polyester from a lower value (7.698 MPa) for pure unsaturated polyester to a higher value (24.76 MPa) for unsaturated polyester reinforced with 3% weight percentages of TiO_2 nanoparticles.

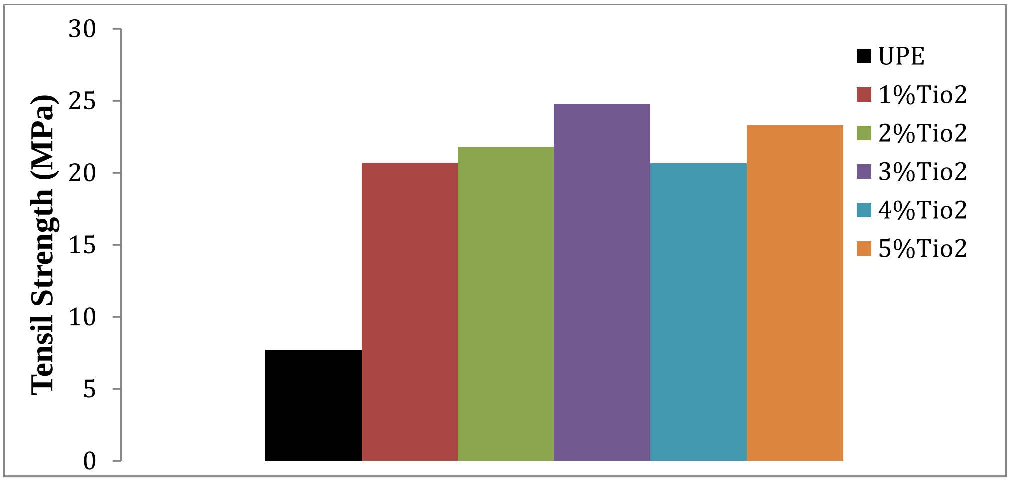

Figure 3. Tensile strength values of UPE and (UPE/Tio2) composites.

Table 3. Tensile strength maximum values of UPE and (UPE/TiO_2) composites.

Sample	Tensile strength maximum values
UPE	7.698
1% TiO_2	20.69
2% TiO_2	21.77
3% TiO_2	24.76
4% TiO_2	20.64
5% TiO_2	23.27

This is because of the good TiO_2 particle dispersion and strong interface adhesion between unsaturated polyester and TiO_2 filler, which leads to better stress transfer in this ratio of filler addition, but by incorporating the filler with higher rates (beyond the 3% weight), the tensile strength values will decrease because of the agglomeration effect. These results agree with (16, 21).

3.1.4. Flexural strength

The flexural strength of composites is the ability of this composite to bend by shedding external stress (22,23).

Figure (4) and **Table (4)** explain that the flexural strength was reduced from a higher value (63.438 MPa) for the unsaturated polyester to a lower value (40.55 MPa) by adding a weight fraction of TiO_2 nanoparticles. This is because of the poor bonding between fillers and matrix. This bonding will increase as the ratio of filler addition increases, reaching a high flexural strength at 3% filler. However, this bonding will decrease due to the effect of filler agglomeration, causing the carbs to propagate within the composite and leading to early failure. These results agree with (16).

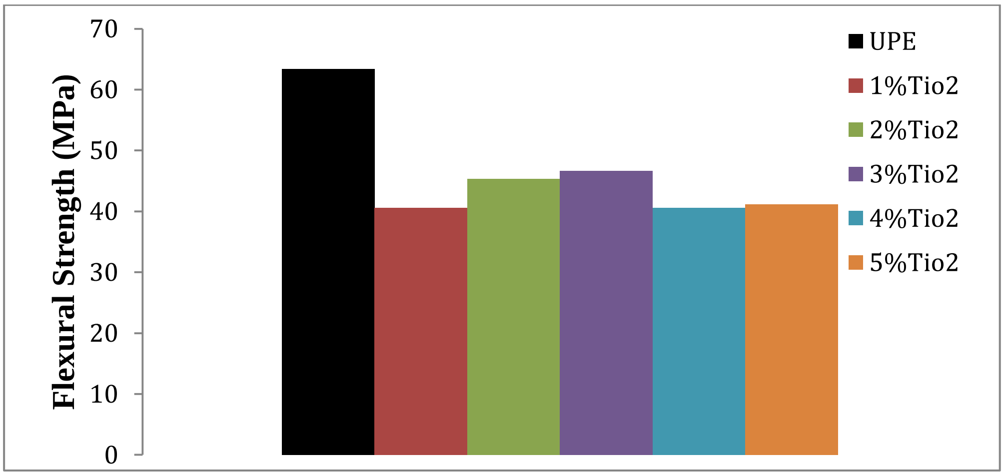

Figure 4. Flexural strength values of UPE and (UPE/TiO_2) composites.

Table 4. Flexural strength maximum values of UPE and (UPE/TiO_2) composites.

Sample	Flexural strength maximum values
UPE	63.438
1% TiO_2	40.55
2% TiO_2	45.37
3% TiO_2	46.67
4% TiO_2	40.61
5% TiO_2	41.16

3.2. Thermal properties

3.2.1. Thermal conductivity

The thermal transmits of unsaturated polyester reinforced by TiO_2 are made of two phases: the first is for the TiO_2 filler, while the second is for unsaturated polyester. The TiO_2 filler has a crystalline structure, while the polyester matrix has an amorphous nature**. Figure (5)** and **Table (5)** explain that the thermal conductivity value of unsaturated polyester is 0.308499 W/m. °C, which increases to a higher value (0.566916 W/m.^{o}C) by adding 3% TiO_2. This is because of the effect of crystallinity that increased by adding TiO_2.

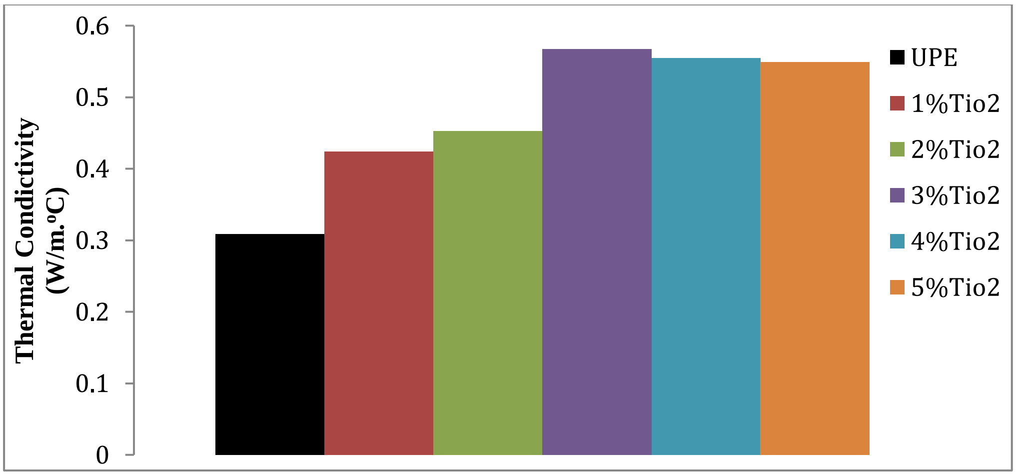

Figure 5. Thermal conductivity values of UPE and (UPE/TiO_2) composites.

Table 5. Thermal conductivity maximum values of UPE and (UPE/TiO_2) composites.

Sample	Thermal conductivity maximum values
UPE	0.308499
1% TiO_2	0.424497
2% TiO_2	0.452429
3% TiO_2	0.566916
4% TiO_2	0.554297
5% TiO_2	0.554936

The unsaturated polyester with a bigger ratio of filler loading, like (4%) and (5%), has lower resistances during the transition of thermals because of the effect of filler agglomeration in the interface region, which leads to crack initiation and reduces the traversal of heat in the unsaturated polyester. These results agreed with (24).

3.2.2. Thermo-gravimetric analysis (TGA)

This technique was employed to determine Teruel stability (25). **Figure (6)** shows that the thermal stability of the UPE/3% TiO_2 composite is higher than that of the pure unsaturated polyester. This is because the thermal stability of the TiO_2 nanoparticle is higher than the thermal stability of the unsaturated polyester resin. Pure unsaturated polyester and the UPE/3% TiO_2 composite have a single stage in the thermal degradation curves. Also, this is because the thermal stability of TiO_2 nanoparticles is higher than the thermal stability of unsaturated polyester resin, which means there, is a big interaction between the nanoparticles TiO_2 and unsaturated polyester. The residue content was increased with the addition of TiO_2 to unsaturated polyester. These results agreed with (24- 28).

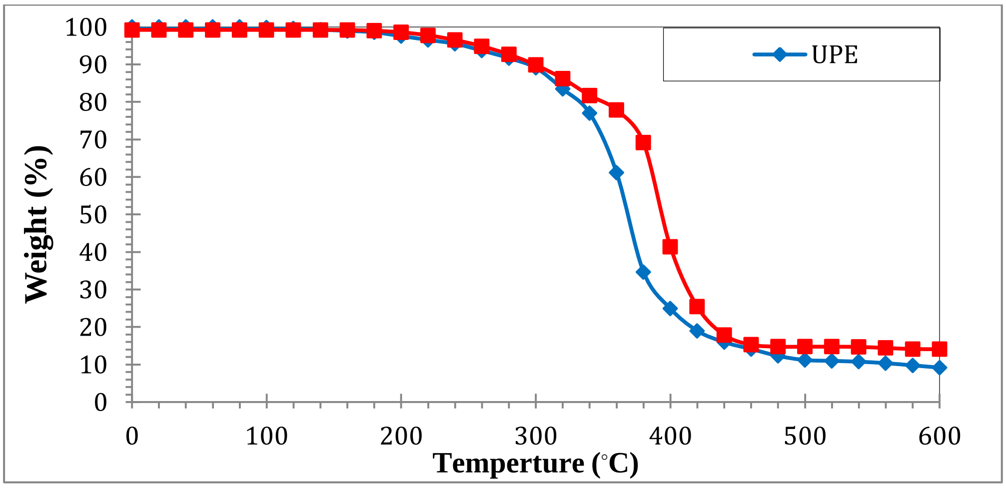

Figure 6. TGA curves of UPE and (UPE/TiO_2) composite.

The thermal stability of the UPE/3% TiO_2 composite is higher than that of pure unsaturated polyester, and it mentions that TiO_2 nanoparticles have higher thermal stability compared to unsaturated polyester resin. Additionally, it indicates that both pure unsaturated polyester and the composite exhibit a single stage in the thermal degradation curves, and the residue content increased with the addition of TiO_2 to unsaturated polyester.

3.3. Structural properties

3.3.1.X-RAY diffraction

Figure (7) explains the X-ray diffraction patterns of the unsaturated polyester, synthesized TiO_2 particles, and unsaturated polyester reinforced with TiO_2.

Unsaturated polyester has a broad peak around ($2\theta=22.490^{\circ\,\circ}$), which is due to the amorphous nature of this resin (18). TiO_2 powder has a crystalline structure that appears in sharp peaks in its XRD spectrum; the highest peak exists around ($2\theta = 26.60^{\circ}$). This pattern is well matched

with the Diffraction Standards (JCPDS) Card File No. 00-001-0562.
The XRD spectrum of unsaturated polyester that is reinforced with 3% TiO_2 particles shows the good conjugation of these particles with the unsaturated polyester, where the broad peak is shifted from position ($2\theta=22.490^o$) to a new position ($2\theta=20.80^o$) and a sharp peak in the same position of TiO_2 particles ($2\theta=26.60^o$), respectively. These results agree with (26).

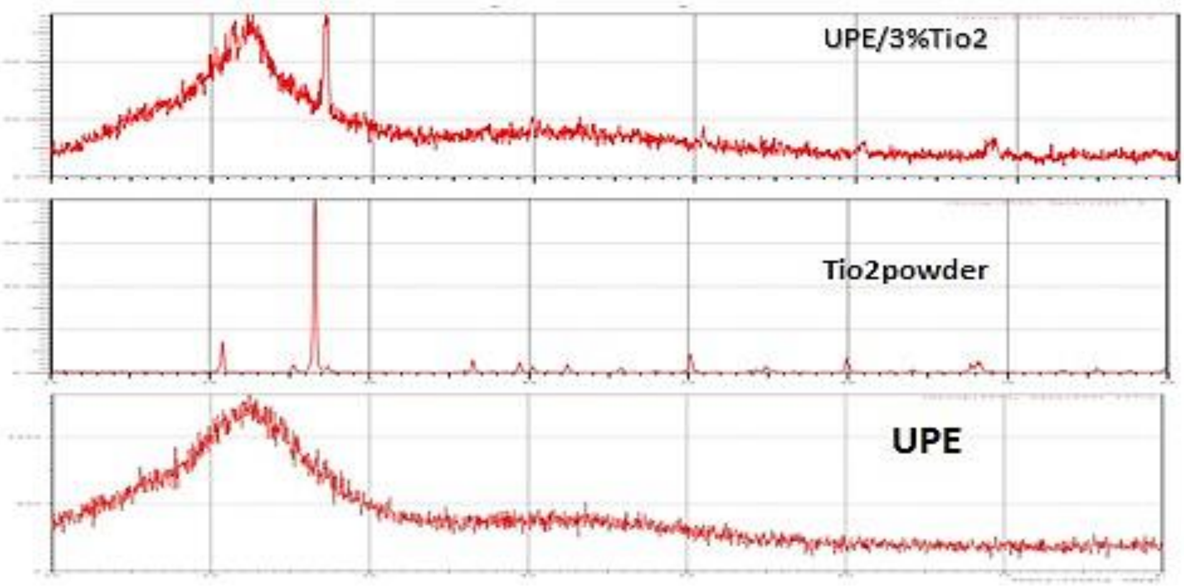

Figure 7. X-ray patterns of UPE and TiO_2 powder and the UPE/TiO_2 composite.

The crystallinity of TiO_2 enhances the properties of reinforced unsaturated polyester (UPE) in several ways:

- Crystalline TiO_2 nanoparticles reinforce within the UPE matrix, enhancing its mechanical properties such as tensile strength, flexural strength, and impact resistance. The presence of well-dispersed TiO_2 particles increases the load-bearing capacity of the composite material, resulting in improved mechanical performance.
- Crystalline TiO_2 nanoparticles have higher thermal stability compared to the amorphous structure of unsaturated polyester. By incorporating TiO_2 into the UPE matrix, the overall thermal stability of the composite is increased. This is particularly important in applications where the material is exposed to high temperatures or thermal cycling.
- The crystalline structure of TiO_2 facilitates efficient heat transfer within the composite material. As a result, the thermal conductivity of the reinforced UPE is enhanced, leading to better heat dissipation and thermal management properties. This is beneficial in applications where thermal insulation or heat dissipation is critical.
- The XRD spectrum indicates good conjugation between the TiO_2 particles and the unsaturated polyester matrix. The shift in the peak position of the unsaturated polyester, along with the presence of sharp peaks corresponding to TiO_2, suggests strong interaction and compatibility between the two materials. This ensures effective load transfer and stress distribution within the composite, leading to improved overall performance.

2.3.2. Scanning electron microscopy

The electron microscope images were employed to fix the homogeneity degree of unsaturated polyester composites and improve their mechanical and thermal performance (29). The scanning electron micrograph showed glassy and smooth surfaces of the impact specimens of the sample UPE, as shown in **Figure (8),** and **Figure (9)** shows that the TiO_2 nanoparticles were distributed uniformly throughout the unsaturated polyester. This sample is not very smooth, and the fracture surface has a ductile manner, which leads to the highest impact performance of unsaturated polyester reinforced with TiO_2 particles. Also, this **Figure (9)** shows that the TiO_2 filler has a homogeneous distribution, indicating very good compatibility with the unsaturated polyester. These results will affect both the mechanical properties and thermal performance of the fabricated composite (14, 24, 26, 30).

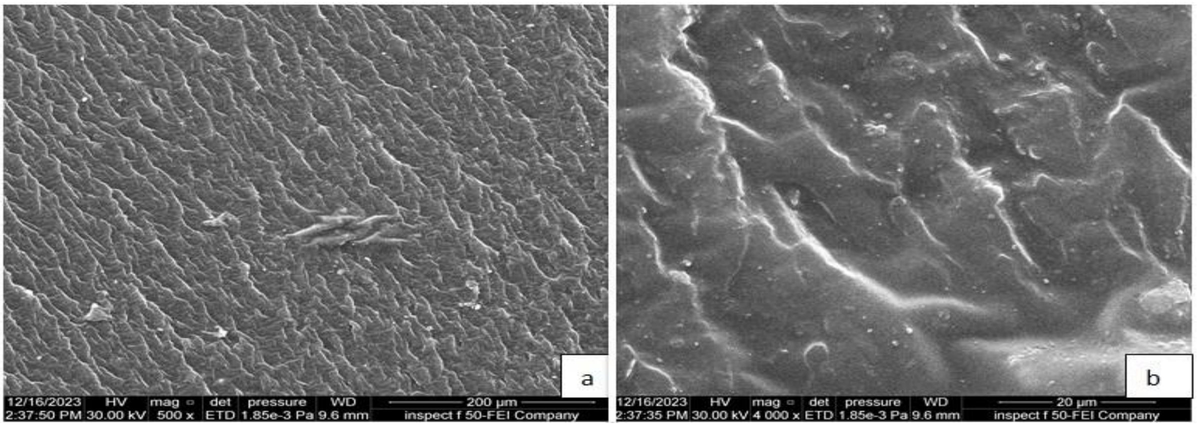

Figure 8. SEM micrographs of UPE with force magnification (a) 500x and (b) 4000x.

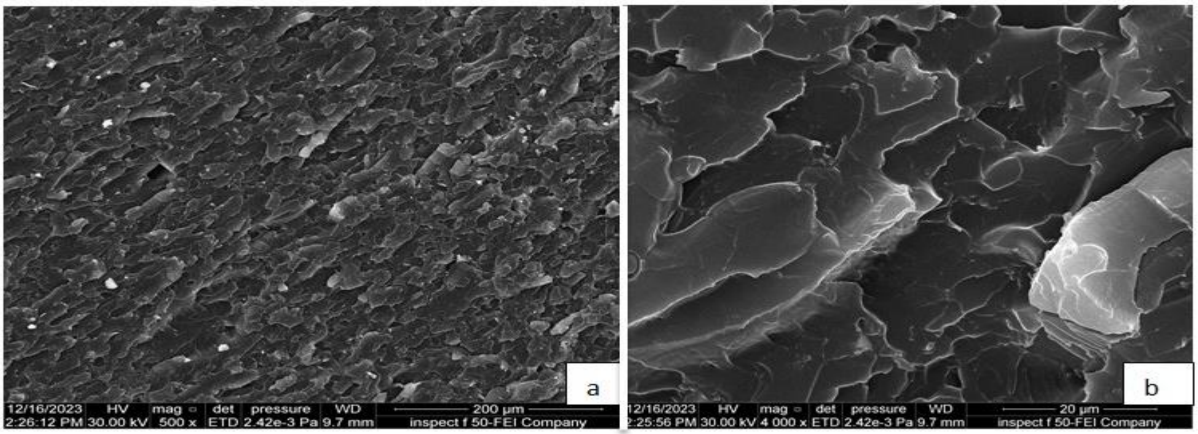

Figure 9. SEM micrographs of (UPE/TiO_2) with force magnification: (a) 500x; (b) 4000x.

Table 6. Roughness maximum values of UPE and (UPE/TiO_2) composites.

Sample	Roughness maximum values
UPE	0.503
1% TiO_2	0.691
2% TiO_2	0.732
3% TiO_2	0.741
4% TiO_2	0.789
5% TiO_2	0.812

Given the roughness maximum values provided in **Table (6)**, it's evident that the samples containing TiO_2 nanoparticles have higher roughness values compared to pure unsaturated polyester (UPE). This indicates that the addition of TiO_2 nanoparticles contributes to increased surface irregularities and roughness. Therefore, it's reasonable to infer that the surfaces observed in **Figure (8)** are indeed rough rather than smooth, aligning with the roughness values presented in the **Table (6).**

4. Conclusion

The properties of unsaturated polyester reinforced by TiO_2 filler depend on the filler weight fraction in the matrix and on the nature of the interface between the unsaturated polyester resin and filler. The reinforcement of unsaturated polyester with 3% TiO_2 particles plays a good role in getting a higher impact strength, hardness, tensile strength, flexural strength, and thermal conductivity; beyond 3%, all these properties decrease because of the agglomeration effect.

TiO_2 particles play a good role in increasing the crystallinity of unsaturated polyester, which leads to improving the mechanical properties and both the thermal stability and thermal conductivity to get a new composite that is resistant to external stress and changes in temperature.

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Conflict of Interests

The authors declare that they have no conflicts of interest.

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