# Effect of $TiO_2$ nanoparticles on Mechanical Properties of Epoxy-Resin System

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**Abstract**— Nanocomposites are new materials made with fillers having nanosize. The purpose of this study is to analyse the mechanical properties of epoxy resin with titanium dioxide nanoparticles. In this present work, titanium dioxide nanoparticle is prepared by sol gel method, using Titanium tetra isopropoxide and acetic acid. The synthesized Titanium dioxide nanoparticles are characterized by XRD and the grain size in nanoscale is conformed. The sheets of neat epoxy resin and epoxy with addition of $TiO_2$ are primed by solution casting method. Mechanical studies of developed polymer sheets reveal much variation by incorporation of $TiO_2$. This is probably due to different dispersion of nanoparticles in epoxy resin.

**Keywords**— TTIP, Sol Gel method, Titanium dioxide, Solution Casting method, Epoxy, Nanocomposite

## INTRODUCTION

In material research, the combination of an organic phase (generally polymers) with inorganic particles has drawn considerable attention since last decades. Nano sized particles allow to improve the mechanical properties of polymer. Mechanical properties like, yield stress, tensile strength and Young's modulus generally suffer a huge increase when compared to pure polymers [1]. Nanosized $TiO_2$ is of particular interest in nanocomposites, because of their specifically size-related properties, non-toxicity, low cost and long term stability [2].

The effective reinforcement by $TiO_2$ nanoparticle of epoxy resins favoured in aerospace and other industries. Depending on the dispersion and particles surface, several interfaces can be observed, which can result in special properties. Thus to attain maximum performance from the $TiO_2$ nanoparticles, uniform dispersion within the matrix must be ensured [3]. The intent of this study is to understand the influence of $TiO_2$ filler weight percentage on mechanical properties of epoxy incorporated $TiO_2$ nanoparticles.

## MATERIALS AND METHODS

### Synthesis of $TiO_2$ nanoparticles

$TiO_2$ nanoparticle was prepared by a simple Sol gel method. Titanium Tetra Isopropoxide (TTIP) and Acetic acid were used as the precursors. 1M of TTIP was added to 4M of acetic acid and this mixture was stirred for 1 hour in magnetic stirrer. To this mixture double distilled water was added dropwise. While the addition of double distilled water this mixture transformed to white gel. After aging of 24 hours, the gel was dried in an oven 200°C for 30 minutes. The solid crystals formed were powdered using agate mortor. To induce transition from amorphous to anatase phase, this powder was annealed to the temperature 600°C in muffle furnace. After calcinations the sample was finely powdered and was used for characterization [4].

### Synthesis of pure epoxy and epoxy-$TiO_2$ nanocomposites

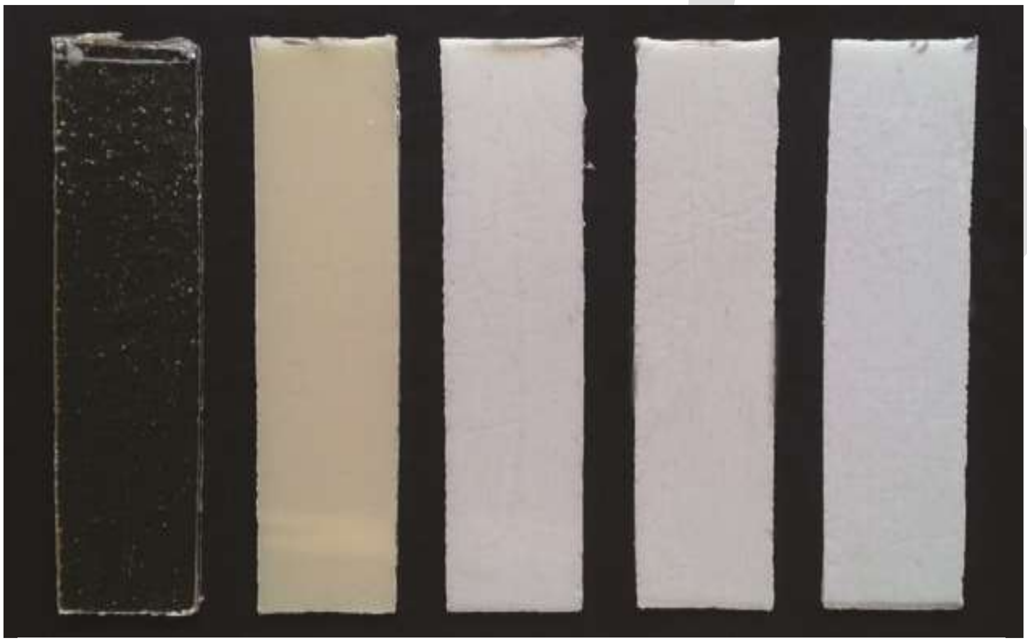
In this study ARALDITE Epoxy resin (DBF 103) and hardener (HY- 951) were used to form pure epoxy and epoxy-$TiO_2$ nanocomposites for different $TiO_2$weight percentage (1wt %, 3wt %, 5wt %, 7wt %).

### *Preparation of pure epoxy sheet*

Epoxy resin of 60gm and hardener of 6gm were poured separately in two beakers. To remove the air bubbles, both were need to be ultrasonicated for 30 minutes. After the completion of this process, the hardener was added to the epoxy resin and it was mixed by hand stirring. Finally it was ultrasonicated to remove air bubbles generated during the mixing process. After degassing, the mixture was poured into the metal mould. The metal mould was kept undisturbed for 1 hour at room temperature. Finally the sample was cured by keeping the mould in an oven at 100°C for 2hours. Thus neat epoxy sheet was obtained.

### *Preparation of epoxy-$TiO_2$ nanocomposite*

The $TiO_2$ fillers (nano) of 1 weight percentage were dispersed into 60gm of epoxy resin, and both were mixed by a high speed mechanical stirrer (at 600 rpm) for 12 hours. It was then ultrasonicated to remove the air bubbles. After the completion of the degassing process, the 6gm of hardener was added slowly into epoxy-filler with hand stirring. The mixture was again ultrasonicated for 5 minutes to remove gas bubbles generated during the mixing process. After degassing, the mixture was poured into the metal mould. Then the sample was cured by keeping the metal mould in an oven at 100°C for 2 hour. The same procedure was repeated for 3wt%, 5wt%, and 7wt%.



Pure Epoxy + Epoxy +
Epoxy + Epoxy +
Epoxy 1wt % $TiO_2$ 3wt % $TiO_2$ 5wt
% $TiO_2$ 7wt % $TiO_2$

**Fig-1 : Photograph of synthesized polymer sheets**

## CHARACTERIZATIONS AND RESULT

### X Ray Diffraction

Powder XRD is used for crystal phase identification and estimation of the crystallite size for synthesized $TiO_2$ nanoparticle. X-ray diffraction is performed using the XPERT-PROdiffractometer system (PW 3050) with automatic data acquisition using CuKα radiation (λ = 1.54060Å) working at 40 kV/30 mA. The crystallite size D is calculated from XRD data using De-bye Scherrer's formula

$$D = K\lambda/\beta cos\theta$$

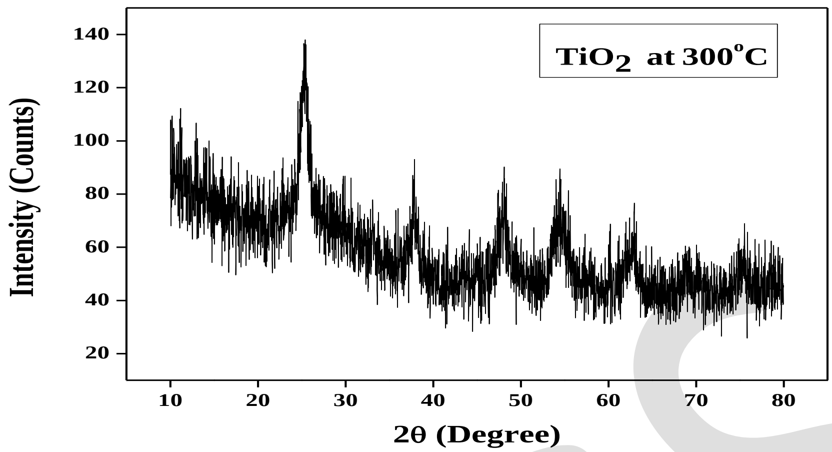



**Fig-2: XRD pattern of $TiO_2$ nanoparticles calcined at 300°C**

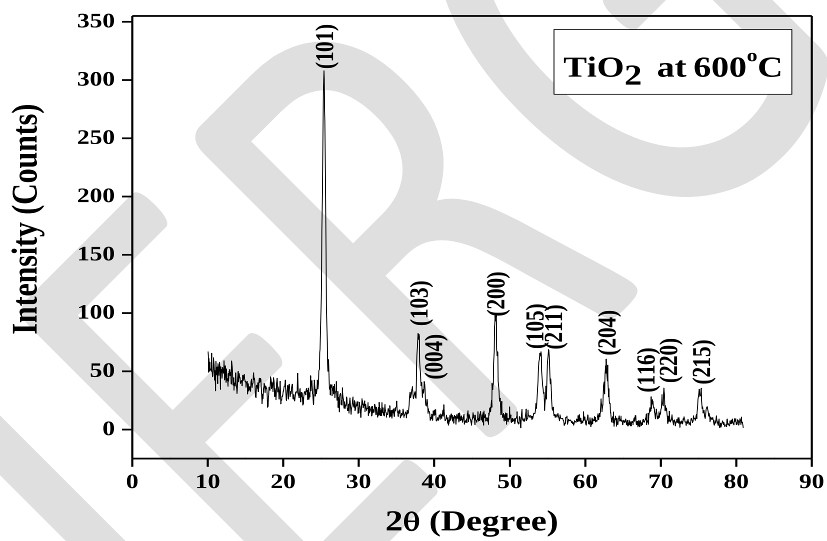



**Fig-3: XRD pattern of $TiO_2$ nanoparticles calcined at 600°C**

The above two XRD patterns illustrate that $TiO_2$ calcined at 300°C is in amorphous structure and annealed at 600°C be in crystalline structure. It concludes that with the increase of temperature to 600°C the $TiO_2$ synthesized by sol gel method acquired enhanced structure. The XRD data of $TiO_2$ nanoparticles calcined at 600°C is given in table 1.

**Table-1 : XRD data of $TiO_2$ calcined at 600°C**

| Angle 2θ (Degree) | Intensity (%) | d- Spacing | hkl |
|---|---|---|---|
| 25.357 | 100.00 | 3.50961 | 101 |
| 36.93 | 4.85 | 2.43224 | 103 |
| 37.90 | 24.41 | 2.37220 | 104 |
| 48.10 | 32.31 | 1.89025 | 200 |
| 53.97 | 21.82 | 1.69755 | 105 |
| 55.12 | 21.49 | 1.66489 | 211 |
| 62.69 | 16.95 | 1.48078 | 204 |
| 68.81 | 5.97 | 1.36319 | 116 |
| 70.27 | 7.74 | 1.33845 | 220 |
| 75.17 | 10.07 | 1.26293 | 215 |

The XRD of $TiO_2$ nanoparticles calcined at 600°C are found to exhibit ten diffraction peaks and of that, (101) reflection plane is very predominant. The crystallite size of $TiO_2$ is calculated by De-Bye Scherrer formula. The crystallite size of synthesized $TiO_2$ is found to be 15.98nm and this confirms that the prepared $TiO_2$ particle is in nanoscale. The X-ray diffraction spectra confirm that the pure $TiO_2$ nanopowder is in anatase crystalline phase.

## Mechanical Properties

The mechanical properties such as tensile strength, elongation at break, flexural strength, modulus, impact strength are measured using a dynamic mechanical analyzer. Tensile test, Flexural strength and Impact test of developed sheets are performed using mechanical analyzer in tensile mode in accordance with the ASTM D-3039 test standard, Flexural mode with ASTM D-790 test standard, Izod impact mode with ASTM D-256 test standard respectively. Before testing, the rectangular samples of fixed size are cut out from sheet using a clean razor blade and the upper side of cut sample for tensile test is polished to make flat surface. And the edges of the sample are polished by sand paper of a mesh of 1200.

### *Tensile Test*

A tensile test measures the resistance of a material to a static or slowly applied force. A machined specimen is placed in the testing machine and load is applied. A strain gage or extensometer is used to measure elongation. The stress obtained at the highest applied force is the Tensile Strength. The Yield Strength is the stress at which a prescribed amount of plastic deformation (commonly 0.2%) is produced. Elongation describes the extent to which the specimen stretched before fracture. Here, tensile test for all samples are carried out on tensile tester as per ATSM D 3039 standard with different load cells. ASTM D3039 is a testing specification that determines the in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fibres.

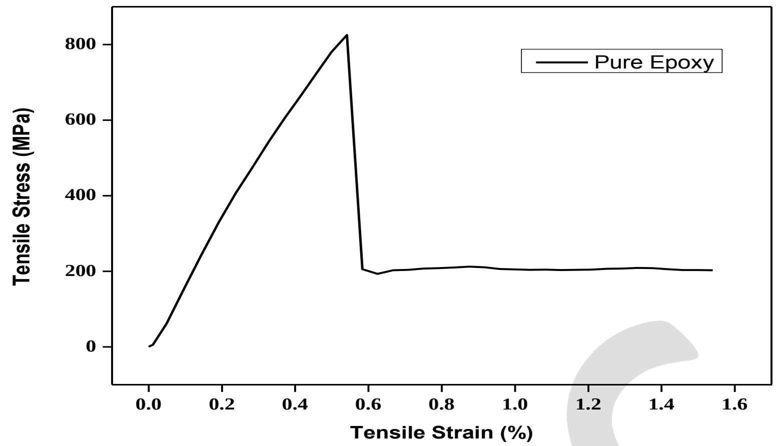



**Fig-4 : Stress Vs Strain graph of pure epoxy**

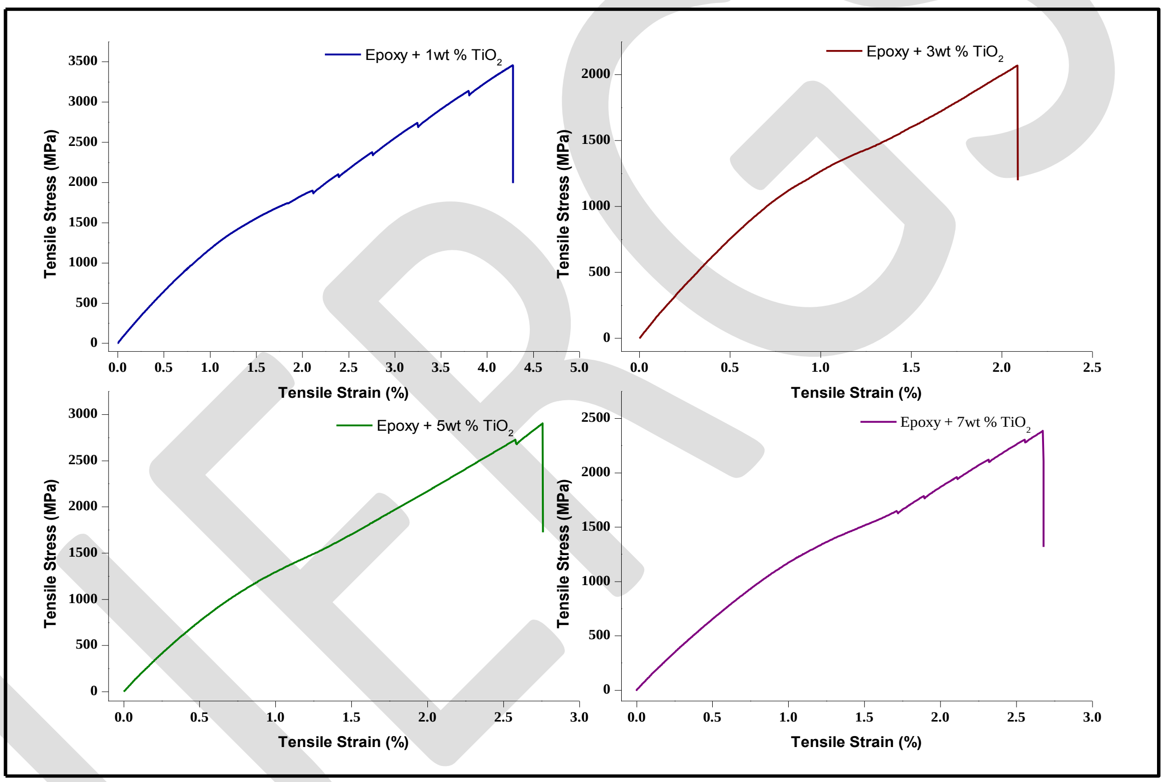



**Fig-5: Tensile stress Vs strain graph of $TiO_2$ modified epoxy**

Tensile modulus is calculated using the formula

$$\text{Tensile Modulus} = \frac{\text{Tensile Stress}}{\text{Tensile Strain}}$$

$$= \frac{\text{Difference in load (N)}}{\text{Difference in extension (mm)}}$$

$$= \frac{\Delta P}{\Delta \delta}$$

**Table-2 : Tensile Properties**

| Sample | Maximum Load (N) | Yield Strength (MPa) | Elongation at break (%) | Ultimate Tensile strength (MPa) | Break Strength (MPa) | Maximum Modulus (GPa) |
|---|---|---|---|---|---|---|
| Pure Epoxy | 825.09 | 4.10 | 1.4582 | 8.48838 | 2.0123 | 1.9335 (1.5-3.6)[6] |
| Epoxy + 1wt % $TiO_2$ | 3,455.8 | 10.3417 | 2.8498 | 35.35057 | 20.0886 | 1.6584 |
| Epoxy + 3wt % $TiO_2$ | 2,068.7 | 7.6536 | 1.3916 | 21.32218 | 12.1967 | 1.8048 |
| Epoxy + 5wt % $TiO_2$ | 2,901.6 | 5.9775 | 1.8388 | 28.82103 | 17.4269 | 1.9069 |
| Epoxy + 7wt % $TiO_2$ | 2,384.7 | 6.5103 | 1.7832 | 24.31754 | 13.1352 | 1.7630 |

Stress - strain curves of pure epoxy, 1wt% 2wt% and 3wt% of $TiO_2$ – epoxy is shown in figure 4 and 5.

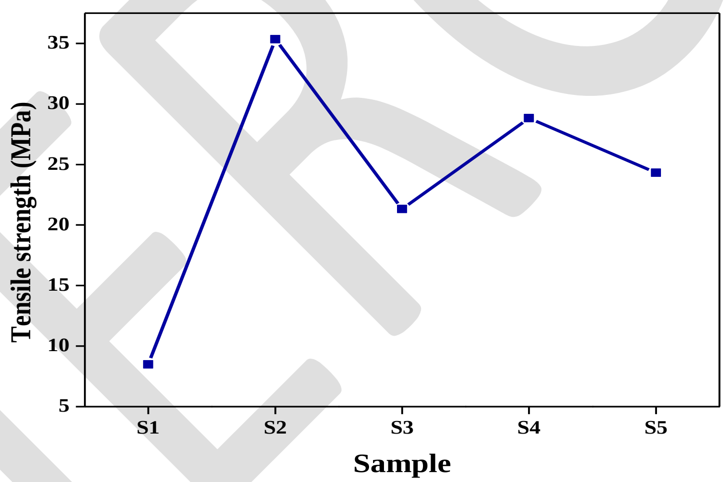



**Fig-6 : Variation of Tensile Strength**

The tensile strength Vs sample graph is shown figure 6. Table 2 of tensile properties shows that the modified epoxy with 1wt % $TiO_2$ can withstands the load by 3,455.87N and this has maximum tensile strength. It is due to high dispersion of nanoparticles with epoxy and amine hardner. The higher (3wt %, 5wt % and 7wt %) percentage loading of $TiO_2$ nanoparticles shows the inferior properties compared to the 1wt% loading of $TiO_2$ nanoparticles but still they are higher than the pure epoxy polymer. The reason for this is explained as particle loading increases the resulting composites will begin to see more and more particle-to-particle interaction rather than the intended particle-to-polymer interaction. Particle to- particle interaction will lead to agglomerated particles and reduced mechanical properties [7]. This is reflects in the maximum load applied for the study. The modulus value of nanocomposite sample increases with the weight percent of $TiO_2$, but at 7wt % $TiO_2$ it decreases due to higher occurrence of agglomeration under gravitational interaction between $TiO_2$ nanoparticles. On the other hand the low value of tensile strength is due to the formation of chain entanglement in matrix system [5].

### *Flexural Test*

Flexure test measures the force required to bend a specimen under 3 point loading condition. Flexural modulus is a measure of stiffness or rigidity and is calculated by dividing the change in stress by the change in strain at the beginning of the test. In this study the flexural test is carried with standard procedures of ASTM D - 790. ASTM D 790 is a method of measuring the flexural properties of a plastic by setting a test bar across two supports and pushing down in the middle until it breaks or bends a specified distance. The obtained flexural properties of samples are tabulated below in table 3.

**Table-3 : Flexural Properties**

| Sample | Ultimate Flexural strength (MPa) | Maximum Flexural Modulus (GPa) |
|---|---|---|
| Pure Epoxy | 95.13 | 2.6262 (2.19)[7] |
| Epoxy + 1wt % $TiO_2$ | 80.67 | 2.8726 |
| Epoxy + 3wt % $TiO_2$ | 57.84 | 2.8877 |
| Epoxy + 5wt % $TiO_2$ | 53.06 | 3.3819 |
| Epoxy + 7wt % $TiO_2$ | 78.21 | 3.2190 |

The result in table shows that there is a gradual increase in the flexural modulus and decrease in flexural strength.

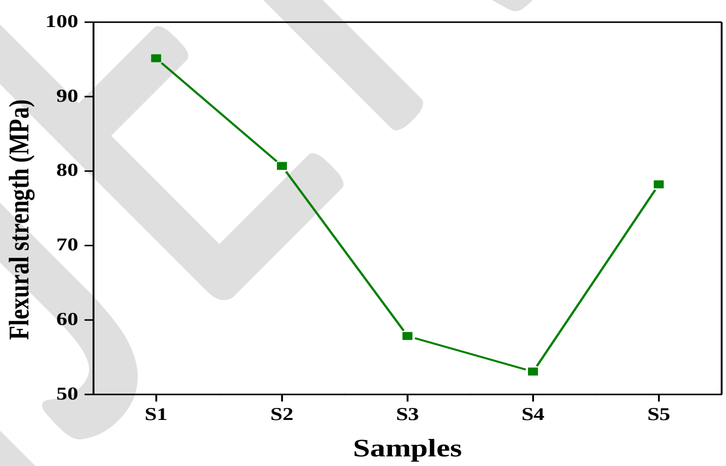



**Fig-7 : Variation of flexural strength**

The figure 7 is the graph of flexural strength Vs sample. For pure epoxy the flexural modulus has low value 2.6262GPa. The flexural modulus is high with 3.3819GPa for 5wt % $TiO_2$ added epoxy. $TiO_2$ 7wt % incorporated epoxy has maximum flexural strength comparing with other modified epoxy, may be due to the plasticizing behaviour of $TiO_2$ into epoxy [6].Thus there is a vast increment in modulus value and shrink in flexural strength with addition of $TiO_2$ to 5wt %. Decrease flexural strength begin, where increasing the addition of filler lead to increasing the constrained between polymer chains, decreasing the length of chains over certain critical length. This lead to decreasing flexural strength which is depend on chain length, but flexural strength still higher for neat epoxy resin because of van der waals bond which is weak bond but with huge numbers [8].

### *Izod Impact test*

Impact test is a measure of the resistance of material when an impact loading is applied. For this effort, Impact strength is determined from Izod impact strength test. It is performed by the procedures of ASTM D 256 standard. It is used for the determination of the resistance of plastics to "standardized" pendulum-type hammers, mounted in "standardized" machines, in breaking standard specimens with one pendulum swing. The standard test for this method requires specimens made with a milled notch. The acquired impact values are noted below in table 4.

**Table-4 : Impact Strength**

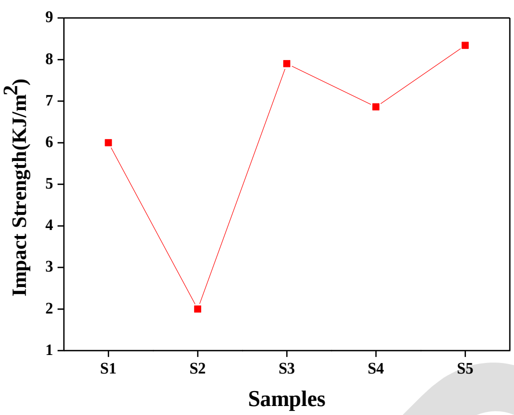



**Fig-8 : Impact strength of various samples**

| Sample | Impact Strength kJ/m$^2$ |
|---|---|
| Pure Epoxy | 6.0 |
| Epoxy + 1wt % $TiO_2$ | 2.0 |
| Epoxy + 3wt % $TiO_2$ | 7.9 |
| Epoxy + 5wt % $TiO_2$ | 6.86 |
| Epoxy + 7wt % $TiO_2$ | 8.34 |

The graph for Impact strength Vs samples is shown in figure 8. Impact strength graph specifies that for pure epoxy the strength is high. When epoxy is modified by $TiO_2$ nanoparticle of different weight percentage the impact value significantly increases with improvement in $TiO_2$ mass. The 7wt % $TiO_2$ added epoxy has high impact strength, shows mechanically it has high strength.

## CONCLUSION

In this study the $TiO_2$ nanoparticles were prepared by sol gel method, and annealed at 300°C and 600°C for 1 h. The structure of the prepared nanopowders has been analyzed by X-ray diffraction technique. $TiO_2$ synthesized at 600°C shows anatase phase. The polymer samples were prepared by solution casting method. The pure epoxy, epoxy + 1wt % $TiO_2$, epoxy + 3wt % $TiO_2$, epoxy + 5wt % $TiO_2$ and epoxy + 7wt % $TiO_2$ were the developed composites. The effect of particle dispersion situation on the mechanical properties of nanocomposites has been studied. Tensile strength of epoxy with 1wt % $TiO_2$ nanoparticle has maximum attainment. The flexural strength of pure epoxy and 7wt % $TiO_2$ added epoxy has enhanced value because of plasticizing behaviour of $TiO_2$ into epoxy respectively. The impact strength of 1wt % incorporated epoxy is found to be very low and it increases by increasing the amount of $TiO_2$. Finally, mechanical studies showed that epoxy with the addition of $TiO_2$ nanoparticles have certain influence on the mechanical properties.

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