

# The Effect of Reinforcement Weight Ratios on the Mechanical Properties of ZrO<sub>2</sub>/ Unsaturated Polyester Nanocomposites

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# Abstract

This work aims to prepare  $ZrO_2/$  unsaturated polyester nanocomposites with reinforcement weight percentage ratios (1, 2, 3, 4, 5, and 6) wt.% and study the effect of this reinforcement weight ratio on some mechanical properties such as hardness, impact strength, and tensile strength. The used powder has a monoclinic structure, P2/m space group, and unit cell parameters a, b, and c = 5.313Å, 5.210 Å, and 5.145 Å, receptively, and angels  $\alpha$ ,  $\gamma = 90^{\circ}$  and  $\beta$ = 99.233° according to XRD data and using Dicvol 91 indexing program. The grain size of the used powder was estimated using Scherrer's equation to be 43.2 nm. The SEM micrograph of ZrO2 nanopowder showed the particle morphology having homogenous irregular grains and appears to be similar to a coral shape. The results of the hardness test showed that increasing the reinforcement weight ratio led to an increase in the hardness values. Impact, tensile, and flexural strength values increase with the reinforcement weight ratio and peaked at 6 wt. %, and decreased at a higher ratio.

**Keywords:** Nanocomposite; Impact Strength; Unsaturated Polyester; Zirconium Oxide Nanopowder; XRD; SEM; Hardness; Tensile Strength

# 1. Introduction

Since the beginning of the sixties of the last century, the interest of researchers in these materials has increased because of the requirements of industrial development that led to the development of the science and technology of composite materials trying to manufacture new materials. The main purpose of preparing composite materials is to acquire new properties that cannot be obtained from the constituent material when separated, thus obtaining material that meets the requirements of a specific design, whether physical, chemical, electrical, magnetic, etc. [1, 2]. A composite material can be characterized as a blend comprising at least two materials of various particulars (each material retains its mechanical, physical, and chemical properties) to form an engineering material possessing certain properties not possessed by the constituents. The constituent materials of the composite substance may be organic, inorganic, or natural [3-6]. The major features of composite materials are higher stiffness and strength, improved torsion, stiffness, impact properties including fracture and residual strength are excellent, reduced energy dissipation, corrosion resistance, lightweight, improved friction and wear properties, dimensional stability, and excellent damping features [7, 8]. The composite material's behavior generally depends on three elements: matrix, reinforcement, and the phase between them [9, 10]. Composite materials are classified into three groups according to their matrix phase: ceramic matrix composites (CMCs), metal matrix composites (MMCs), and polymer matrix composites (PMCs).

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The reinforcement materials can be oxides, carbides, or nitrides and their shape (short and continuous fibers, particulates, and whiskers) [11]. Zirconium oxide (ZrO2), sometimes known as zirconia, is an extremely important oxide because of its useful properties. Zirconia is the main compound in materials science and innovation since it joins fantastic mechanical, synthetic, and dielectric properties: high softening point (2700 °C), electronic conductivity, high strength, and upgraded break durability, etc. [12, 13]. To date, many approaches have been proposed and used to prepare various sorts of nanocomposites. In 2017, S. Sousa et al. concentrated on the mechanical conduct appraisal of unsaturated polyester polymer mortars (PM) loaded up with  $Al_2O_3$  and  $ZrO_2$  nanoparticles by utilizing flexural, compressive, and Shore D hardness tests. They found that the PM changed with nano zirconia showed better mechanical execution contrasted with the unmodified PM [14]. S. Sapuan et al., in 2020, studied the mechanical properties of longitudinal basalt/woven-glass-fiber-built up unsaturated polyester-tar cross breed composites where the hybridization of basalt and glass fiber improved the mechanical properties of mixture composites. This study showed that the expansion of basalt to glass-fiber-supported unsaturated polyester sap expanded its thickness, and elastic and flexural properties [15]. Salt treated sisal fiber-integrated silanized ZrO2 scattered unsaturated polyester composites were created with a filler stacking of 5, 15, 25, 35, 45 wt.% by B. Biswas et al. in 2021. A noticeable improvement in the mechanical properties of the unsaturated polyester network because of the joining of the fillers (sisal or potentially ZrO2) was noticed [16].

This work aimed to prepare the ZrO2/unsaturated polyester nanocomposites with reinforcement weight percentage ratios (1, 2, 3, 4, 5, and 6) wt.% and study the impact of this support weight proportion on a few mechanical properties like hardness, influence strength, and rigidity.

### 2. Experimental Part

#### **2.1 Materials**

Used unsaturated polyester resin is supplied from B-chem, Marmolit, Italy, and dilution by dibenzoyl peroxide catalyst paste as a hardener. Zirconium oxide nanopowder (ZrO2) is supplied from Hongwu International Group Ltd, China, with a purity of 99.9% and particle size (40-50) nm.

#### 2.2 Synthesis

The used nanocomposites were prepared by hand lay-up molding. Molded samples as a sheet shape in the dimensions of  $(13 \times 13 \times 0.4 \text{ cm}^3)$ . These sheets of unsaturated polyester resin were prepared for each percentage weight (1, 2, 3, 4, 5, and 6) of ZrO<sub>2</sub> nanopowder. The samples are cut by laser according to ASTM (D-2240, ISO- 179, D-638, and D-790 m-86) of the hardness, impact strength, tensile strength, and flexural strength tests methods.

#### **2.3 Characterization**

XRD and SEM tests were performed to prove the particle size of the used powder in nanoscale. The hardness, impact strength, tensile strength, and flexural strength of used prepared nanocomposite. The hardness was estimated by (Durometer Hardness) type (Shore D) factory by (TIME GROUP INC./ ITALY) company. The impact strength was examined by a (US-made) instrument supplied by testing machines inc. (tmi). Tensile properties were obtained using a ZWICK universal testing machine. The flexural strength was measured by the piston-on-three-ball test.

#### 3. Result and Discussion

The crystallography and phase formation of  $\text{ZrO}_2$  nanopowder were investigated using X-ray diffraction (Shimadzu XRD-6000) with Cu K $\alpha$ 1 radiation ( $\lambda$  = 1.540 Å) and 2 $\Theta$  values from 20° to 100°. The peaks in the XRD pattern of  $\text{ZrO}_2$  nanopowder (Figure 1) were in accordance with JCPDS No. 24-1165 which expressed the monoclinic structure [13]. The results of indexing for  $\text{ZrO}_2$  nanopowder performed by using (Dicvol 91) program are (unit cell parameters a, b, and c = 5.313Å, 5.210 Å, and 5.145 Å, receptively, with P2/m space group and angels  $\alpha$ ,  $\gamma$  = 90° and  $\beta$ = 99.233°. The presence of sharp peaks in the XRD pattern of ZrO2 nanopowder indicates the formation of nanoparticles, and that as the width of the peak increases, it represents a decrease in the size of the nanoparticles, and the absence of additional peaks in the used nanopowder proves its high purity. The average grain size for used  $\text{ZrO}_2$  nanopowder was determined by Scherrer's equation, which was found to be about (43.2 nm).

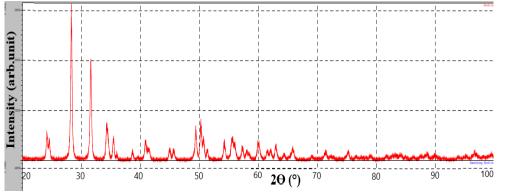


Figure 1 XRD pattern of ZrO2 nanopowder

surface morphology of ZrO<sub>2</sub> nanopowder was observed by scanning electron microscopy (Figure 2). SEM micrograph showed the particle morphology having homogenous irregular grains. The image <u>appears to be similar to a coral shape</u>.

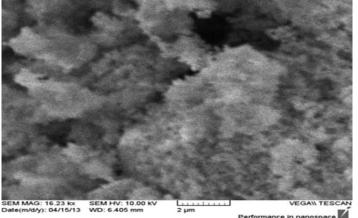


Figure 2 SEM image of ZrO2 nanopowder

The hardness test gives a good idea of the strength and consistency of the material mass. Figure 3 shows the effect of reinforcement weight ratios on hardness values. The results showed that an increase in the reinforcement weight ratio increases the hardness values, return to increasing the degree of cross-linking, the overlap and stacking between the resin and the additive, this leads to a reduction in the movement of dislocation of polymer molecules and an increase in the resistance of materials to scratch, thus become more resistance to plastic deformation because of the possibility of nanoparticles to share the fundamental stage of carrying out forces and stresses imposed on them and thus increasing its hardness, this behavior of material agrees with what (R. Akaluzia et al. & S. Chelladurai et al.) reached [17, 18].

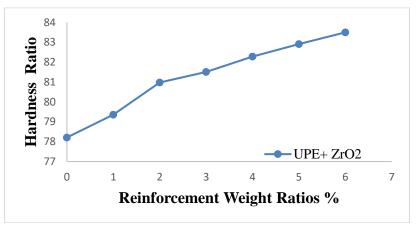


Figure 3 Relationship between hardness and reinforcement weight ratio

The behavior of materials under the effect of rapid forces was studied based on the by the impact strength test. Impact strength represents a measure of the strength of the material and its ability to absorb energy to breakage. Figure 4 shows the relationship between the reinforcement weight ratios of  $ZrO_2$  nanopowder and the impact strength. The neat unsaturated polyester resin has low impact strength and increases with the increase in the reinforcement weight ratios because the nanoparticles work to withstand part of the impact stress exerted on the composite material and prevent the spread of the cracks from increasing to increase the bond between the base material and the reinforcing materials, this behavior of material agrees with what (V. Mohanavel et al.) [19]. As the reinforcement weight ratio increases, the interfacial adhesion between the additive and matrix diminishes, lowering the impact strength value at a reinforcement weight ratio of (6). Occurrence of agglomeration of nanoparticles, which are areas of weakness in the sample, the behavior of the material agrees with what (Mirjalili et al) reached [20].

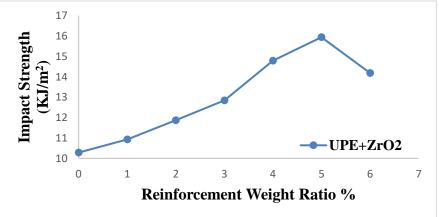


Figure 4 Relationship between impact strength and reinforcement weight ratio

The maximum load a specimen can withstand before it breaks under a gradually increasing load applied slowly was investigated by a tensile test. The tensile strength of the unsaturated polyester resin is very low, and therefore it is considered a brittle material; when you add the  $ZrO_2$ , its tensile strength will be significantly improved. The polymer's low elasticity is what gives it its tensile strength. There is an increase in tensile strength. Increasing the reinforcement weight ratio of  $ZrO_2$  because, it occupies more space within the resin, allowing for better load distribution. It is noted from Figure 5 that adding  $ZrO_2$  nanopowder by (5 wt. %) increases the tensile strength, but any increase after that will be at the expense of the decrease in tensile strength.

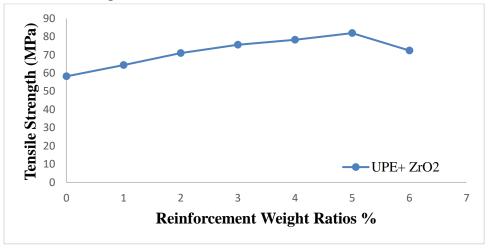


Figure 5 Relationship between stress and reinforcement weight ratio

The resistance of a material to elastic deformation is measured by bending modulus (flexural modulus). The bending characteristics often rely on the bonding between the fillers and the matrix materials. Figure 6 shows the variation of flexural strength with reinforcement weight ratios for  $ZrO_2$ /unsaturated polyester. The flexural strength of the resin increase with increasing ZrO2 content because the filler material's high flexible modulus contrasted with that of the lattice material. While the worth of flexural strength decreases at a reinforcement weight ratio (6) because the agglomerations of  $ZrO_2$  nanoparticles result in inhomogeneous distribution, weakening the interaction between the filler and matrix.

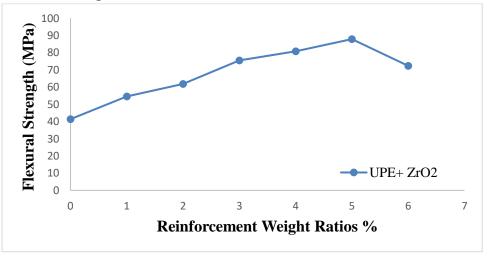


Figure 6 Flexural strength and reinforcing weight ratio in relation to each other

## 4. Conclusions

The addition of ZrO2 nanoparticles to unsaturated polyester is an improvement in mechanical properties. Impact strength, tensile strength, and flexural strength of composites increase with the increase of reinforcement weight ratio, and at (6 wt. %), these properties decreased with the increase of reinforcement weight percentage. Hardness increases with the reinforcement weight ratio and reaches a maximum value of (6 wt. %).

# Reference

[1] Yi, X. S., Du, S., & Zhang, L. (2018). Composite Materials Engineering, Volume 2.

[2] Choo, V. K. (1990). *Fundamentals of composite materials*. Knowen Academic Press.

[3] Barbero, E. J. (2018). Introduction to Composite Materials Design (3rd Editio). *Boca Raton*. [4] Jones, R. M. (2018). *Mechanics of composite materials*. CRC press.

[5] Bahl, S. (2021). Fiber reinforced metal matrix composites-a review. *Materials Today: Proceedings*, *39*, 317-323.

[6] Campbell, F. C. (2010). Structural composite materials. ASM international.

[7] Benzait, Z., & Trabzon, L. (2018). A review of recent research on materials used in polymer–matrix composites for body armor application. *Journal of Composite Materials*, *52*(23), 3241-3263.

[8] Buragohain, M. K. (2017). *Composite structures: design, mechanics, analysis, manufacturing, and testing*. CRC press.

[9] Bahl, S. (2021). Fiber reinforced metal matrix composites-a review. *Materials Today: Proceedings*, *39*, 317-323.

[10] Jasso-Gastinel, C. F., & Kenny, J. M. (Eds.). (2016). *Modification of polymer properties*. William Andrew.

[11] Trinh, S. N., & Sastry, S. (2016). Processing and properties of metal matrix composites.

[12] Bakr, N. A., Al-Saadi, T. M., & Hameed, N. A. (2014). Study of nanocrystalline structure and micro properties of ZrO2 powders by using Rietveld method. *Int. J. Curr. Res, 6*, 5743-5748.

[13] Hassan, N. S., & Jalil, A. A. (2022). A review on self-modification of zirconium dioxide nanocatalysts with enhanced visible-light-driven photodegradation of organic pollutants. *Journal of Hazardous Materials*, *423*, 126996.

[14] Sousa, S. P. B., Ribeiro, M. C. S., Novoa, P. R. O., Pereira, C. M., & Ferreira, A. J. M. (2017). Mechanical behaviour assessment of unsaturated polyester polymer mortars filled with nano-sized  $Al_2O_3$  and  $ZrO_2$  particles. Ciencia & Tecnologia dos Materiais, 29(1), e167-e171.

[15] Sapuan, S. M., Aulia, H. S., Ilyas, R. A., Atiqah, A., Dele-Afolabi, T. T., Nurazzi, M. N., ... & Atikah, M. S. N. (2020). Mechanical properties of longitudinal basalt/woven-glass-fiber-reinforced unsaturated polyester-resin hybrid composites. *Polymers*, *12*(10), 2211.

[16] Biswas, B., Hazra, B., Mukherjee, N., & Sinha, A. (2021). Nanomechanical behaviour of ZrO<sub>2</sub> dispersed sisal-based polymeric composites. *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications*, 14644207211016015.

[17] Akaluzia, R. O., Edoziuno, F. O., Adediran, A. A., Odoni, B. U., Edibo, S., & Olayanju, T. M. A. (2021). Evaluation of the effect of reinforcement particle sizes on the impact and hardness properties of hardwood charcoal particulate-polyester resin composites. *Materials Today: Proceedings*, *38*, 570-577.

[18] Chelladurai, S. J. S., & Arthanari, R. (2020). Prediction of hardness of stir cast LM13 aluminum alloy-copper coated short steel fiber reinforced composites using response surface methodology. *Materialwissenschaft und Werkstofftechnik*, *51*(2), 221-229.

[19] Mohanavel, V., Raja, T., Yadav, A., Ravichandran, M., & Winczek, J. (2021). Evaluation of Mechanical and Thermal Properties of Jute and Ramie Reinforced Epoxy-based Hybrid Composites. *Journal of Natural Fibers*, 1-11.

[20] Sathish, S., Kumaresan, K., Prabhu, L., Gokulkumar, S., Karthi, N., & Vigneshkumar, N. (2020). Experimental investigation of mechanical and morphological properties of flax fiber reinforced epoxy composites incorporating SiC and Al2O3. *Materials Today: Proceedings, 27,* 2249-2253.

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