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ENHANCEMENT OF MECHANICAL PROPERTIES OF UNSATURATED POLYESTER REINFORCED WITH AL_2O_3 NANOPARTICLES

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ABSTRACT

The experimental investigation of the effect of adding alumina oxides Al_2O_3 nanoparticles on mechanical properties of unsaturated polyester resin has been studied. The Mechanical properties were evaluated by tensile, hardness and impact test. The study involved adding alumina oxide nanoparticles with size 45nm in different weight ratio which are 0,3,5,7, and 9%. The specimens were manufactured using hand-layup technique. The results showed that the increasing of AL_2O_3 into the polyester matrix can improve the tensile strength, elastic modulus, hardness, and impact strength of polyester to 18.91, 10.44, 1.21, and 221.53%, respectively, when the AL_2O_3 weight fraction reached 5%. However, the mechanical properties decreased when the Al_2O_3 nanoparticles weight fraction reached 7% and 9% . The Optical microscopes observations revealed that the nanoparticles were more homogeneously dispersed at 5%.

KEYWORDS

Polyester resin, Alumina nanoparticles, Tensile, Hardness, Impact test.

1. INTRODUCTION

Composite materials are combinations of two or more materials whose specific properties are created by carefully combining various constituents (Rashid, W.T, 2023). The process of creating materials that are specifically designed to perform a given task while being more lightweight, more powerful, and safer to use. In addition to improving several mechanical characteristics of the material, such as hardness and tensile strength. The popularity of composite materials stems from their ability to enhance basic materials qualities and their wide variety of applications in diverse manufacture (Hamzah et al., 2021). Despite extensive testing of composite materials, little progress has been achieved in the comprehending and creating of a predictive method for composite failure. Mechanical flaws of any size can have a noticeable impact on the composite material's strength (Dookhi and Tahir, 2023). A composite reinforcement includes the fibers, flakes, and particles. Each of these has a unique set of properties that can be added to composites, and thus offering a unique range of applications (Ngo, 2020). Inorganic particle fillers, such as the use of aluminum oxides (Al_2O_3) to process polymer composites, are commonly mixed constituent phases, are vastly and cost-effectively used materials in the engineering ceramics family. Furthermore, at high temperatures, it is wear-resistant, hard, and boasts excellent insulator characteristics, strong resistance to alkalis and acid attacks, as well as hight stiffness and strength (Farhan and Hussein, 2020). Many researchers have studied the mechanical characteristics of composite material. For examples, (Baskaran et al., 2011) investigated the mechanical properties of the unsaturated polyester matrix (UP) filled with different weight fractions (3,5,7 and 9 wt.%) of alumina nanoparticles with a diameter of about (60–70 nm). The results showed that the tensile, flexural, and impact properties increased with the addition of nanoparticle filler up to (5 wt. %) and then decreased gradually with more alumina nanoparticle addition. Additionally, the result indicated that the ductile breakage behavior of UP is changed into brittle breakage with the inclusion of Al_2O_3. (Latief et al., 2019) investigated polyester-based composites with varying alumina (Al_2O_3) concentration (0, 1, 5, and 10 vol%) and particle size of less than 10 μm were created. The result showed that the composites containing 5 vol% alumina exhibited the highest tensile strength, stiffness, and hardness. In contrast, composites containing 10 vol% alumina showed a slight loss in tensile strength, stiffness, and hardness. (Swain et al., 2013) investigated the effects of nano-silica and nano-alumina addition with various weight fractions (0.1, 0.5, 1.0, 1.5, 3.0, and 5.0 wt.%) on the mechanical properties of polymer (PU). The particle size of alumina nanoparticles with a size 13 nm and silica nanoparticles 7 nm. The nanofiller was distributed in the polymer matrix at various weight fractions using the ultrasonic vibrator mixing method. The finings indicated that incorporating both nano-silica and nano-alumina significantly improved the mechanical properties of the nanocomposites. Interestingly, silica nanoparticles offered better tensile strength enhancement at lower concentrations (0.1 - 0.5 wt.%) compared to nano-alumina at the same concentrations. This was attributed to silica nanoparticles having a higher surface area. Specifically, the study noted a 50% increase in tensile strength with the addition of 1 wt.% alumina nanoparticles into the PU matrix. For silica nanoparticles, a similar weight fraction yielded an approximately 41% increase. However, at higher weight fractions, alumina nanoparticle-filled nanocomposites showed greater tensile strength. This superior performance was linked to the significant mechanical enhancement provided by alumina nanoparticles. The study concluded that 0.5 wt.% was the optimum weight fraction for silica nanoparticles, while 1 wt.% was optimal for alumina nanoparticles. At higher weight fractions (e.g., 1.5% and above), the tensile properties started to decrease due to nanoparticle accumulation, highlighting the importance of proper dispersion. (Al-Allaq., 2006) prepared Eighteen polymeric blends from epoxy resins (EP), unsaturated polyester (UP) and polysulfide rubber (PSR) with (0% - 100%) ratios. The mechanical properties were studied including impact, bending, compression, hardness and tensile tests and thermal conductivity. Heat treatment has been done to all the blends at temperatures of (50oC, 75oC, 110oC and 220oC) to find out its effect on the thermal and mechanical properties of the blends. It has been noticed that the (40% EP/ 60 % UP / 0% PSR) (20% EP / 40% UP / 40%PSR) / (20% EP / 60% UP / 20% PSR) (20% EP / 40% UP / 40% PSR) ratios give higher impact strength while a high compression strength was obtained at (80% EP / 0% UP / 20% PSR). The ternary blend shows a decrease in the value of EP, UP and PSR in thermal conductivity except the ratios (40% EP/ 30% UP/ 30% PSR) which show a little increase in thermal conductivity. (Khashaba., 2016) investigated the influence of adding 1.5wt.% of SiC and AL_2O_3 nanofillers with particle diameters of (15 nm and 20 nm), respectively on the flexural properties of the epoxy matrix. The result showed that the flexural strengths were increased by (28.6% and 26.4%) with the addition of (SiC and AL_2O_3) correspondingly, whereas the flexural modulus was improved by (4.7% and 12.0%) in contrast to the pure epoxy matrix.

Many researchers have investigated the effect of adding various particles to polyester resin. However, there's a notable gap in the existing literature regarding the specific impact of adding alumina oxide (Al_2O_3) nanoparticles on the mechanical properties of unsaturated polyester resin. Therefore, the primary objective of this work is to determine the optimal Al_2O_3 nanoparticle content for reinforcing unsaturated polyester composites. This will involve evaluating the mechanical properties, specifically tensile strength, impact resistance, and hardness, of composites containing Al_2O_3 nanoparticles at weight ratios of 0, 3, 5, 7, and 9%. Additionally, optical microscopy will be used to analyze the dispersion of the nanoparticles within the composite matrix.

2. METHODOLOGY

2.1. Materials

In this study, polyester resin was used as the matrix material. The resin was mixed with methyl ethyl ketone peroxide (MEKP) as a hardener at a weight ratio of 2 mg of hardener per 100 mg of resin. The polyester resin was supplied by Start-up Speed Pox GmbH (Holz-Steiner-Straße 6 Objekt 4a, 2201 Seyring, Austria) and packed in Turkey. High-purity alumina (Al_2O_3) nanoparticles with a purity of 99.99% and an average particle size of 45 nm (γ-phase) were used as the reinforcing material. These nanoparticles were supplied by Shanxi Xinhui Alumina Nano Company, Ltd. (Shanxi, China), and their standard properties are shown in Table 1. Alumina is an inorganic substance with excellent strength and toughness.

Table1. properties of oxide aluminum (from data sheet).

Properties	Values
Purity (%)	99.99
Particle Size, (nm)	<45
Specific Surface Area, (m^2/g)	5-10
Young's Modulus, (GPa)	347
Poisson's Ratio	0.22

2.2. Sample Preparation

Composite samples were prepared using the direct mixing method. Aluminum oxide (Al_2O_3) was incorporated into the unsaturated polyester resin at various weight fractions (3, 5, 7, and 9%). Initially, both alumina and polyester were accurately weighed using a digital balance. The alumina was then mechanically mixed with the polyester resin for 30 minutes at room temperature. To ensure homogeneous dispersion and prevent agglomeration of alumina particles within the resin, an ultrasonic device was employed as an additional processing step. Subsequently, as the final step, the hardener was added at a specified ratio to each batch. The mixture was then gradually and slowly poured into pre-prepared molds to avoid the formation of air bubbles. The samples were left at room temperature for 24 hours until complete solidification. Following this, the samples were subjected to post-curing in a drying oven at 70°C for 3 hours, then stored at room temperature for three weeks to ensure complete stabilization prior to conducting the required mechanical tests. The following Fig. 1 shows the sample preparation steps.

3. OPTICAL MICROSCOPES

An optical microscope is a device used to magnify small items that are not visible to the naked eye, or to reveal the fine intricacies of composite materials. As shown in Fig. 2, it was used to determine if the distribution of alumina within the samples was homogeneous or heterogeneous and to identify the presence of agglomerates as the alumina percentage increased. The microscope features a revolving nosepiece (or turret) at the bottom of the tube, which can be rotated to select the desired objective lens. A stage is located under the objective lenses to hold the sample. The composite material sample (polyester with alumina) is placed on this stage and then viewed through the eyepiece. An opening in the middle of the stage allows light to pass through and illuminate the sample being examined. A magnification of 20x was used for the samples, which yielded optimal results for this analysis.

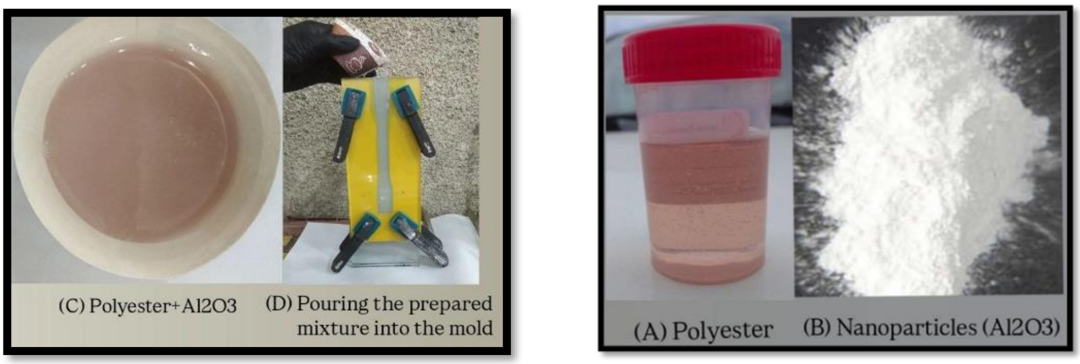

Fig. 1. Sample preparation procedures.

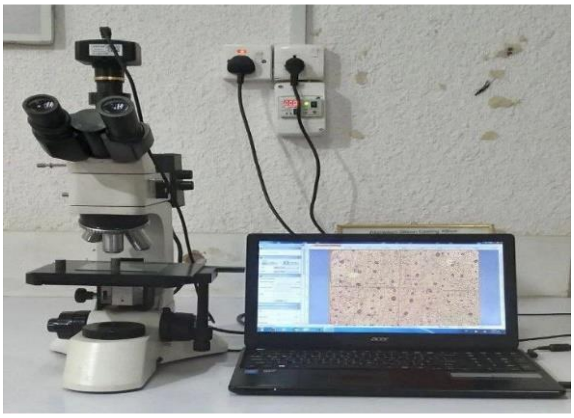

Fig. 2. Optical microscope device.

4. MECHANICAL TESTS

4.1. Tensile test

The tensile test is one of the most important and simplest tests performed using Universal Testing Machine Type (WDW-5E), as shown in the Fig. 3. The sample was prepared with standard dimensions according to ASTM D638 shown in the Fig. 4 (ASTM D638, 2014). The tensile test was performed by applying a uniaxial load to both ends of the sample at a crosshead speed of 2 mm/min and a temperature of 25∘C. Three sample values were reported, and their average was then determined (Kiran et al., 2018). Several key properties of the samples were determined, including stress, modulus of elasticity, and strain.

Fig. 3. Tensile test machine (WDW-5E)

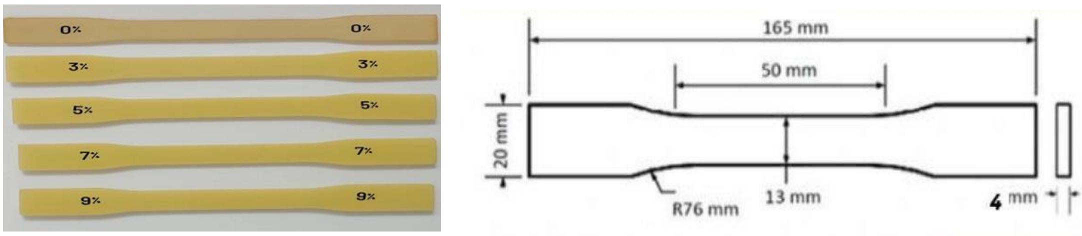

Fig. 4. Tensile test specimen and dimensions.

4.2. Hardness test (shore D)

Hardness is defined as a material's resistance to permanent deformation, such as denting, corrosion, or scratching. The importance of hardness testing lies primarily in the relationship between hardness and other material properties. The main reason for using Shore D is that it is designed to measure the hardness of polymeric materials with high hardness that cannot be evaluated on a lower scale such as (Shore A), which makes it an essential tool in selecting and evaluating materials used in engineering and mechanical applications. The specimen was prepared for hardness testing according to ASTM D2240 (ASTM D2240, 2015), where the dimensions were (20×20×6) mm, as shown in the Fig. 5. Each specimen was first placed on a hard, flat surface. The device's indenter was then pressed into the sample, ensuring the durometer's presser foot was flush with the surface, as shown in Fig. 6. The hardness was read within one second of contact with the sample. Five readings were taken for each sample to ensure the homogeneity of the material, checking for the absence of agglomerates or pores and confirming a consistent mixing process.

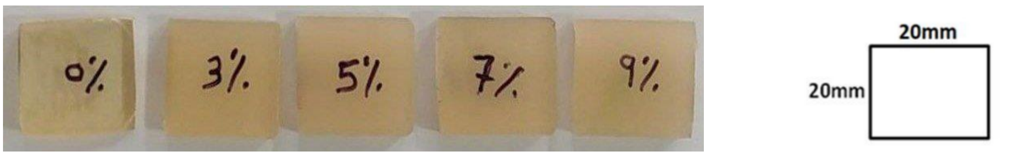

Fig. 5. Hardness test specimen and dimensions.

Fig. 6. Hardness test device(TIME5431).

4.3. Impact test (Charpy test)

The Charpy impact test was performed using a pendulum impact tester (Type WP400) to determine the energy absorbed by the material. As shown in Fig. 7, the test was performed according to the ISO 179 standard (ISO 179, 2010). The specimen was fixed in a transverse position. The pendulum then swung down, striking and breaking the specimen into two parts as a result of the impact from a specific and fixed height. Three sample values were reported, and their average was then determined (Kiran et al., 2018). The samples prepared for the impact test had standard dimensions, as shown in Fig. 8.

Fig. 7. Impact test device (WP400).

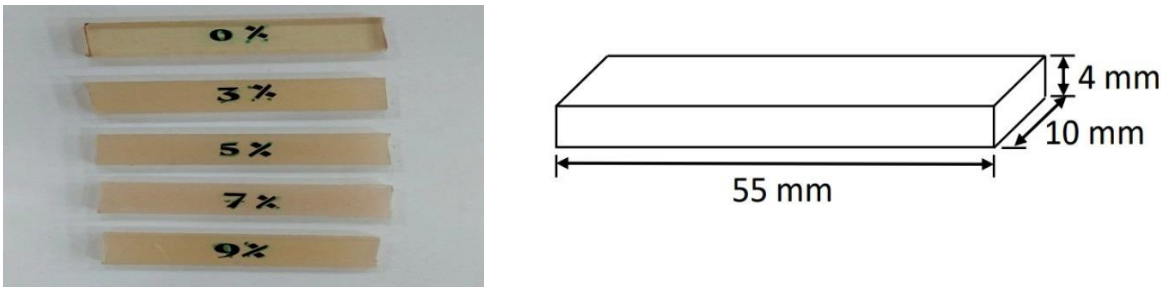

Fig. 8. Impact test specimen and dimensions.

5. RESULTS AND DISCUSSION

5.1. Microstructure analysis

Optical microscopy studies conducted on polyester and alumina samples at various concentrations (3, 5, 7, and 9 wt%) revealed different microscopic results, reflecting changes in structure and properties. These changes included particle distribution, grain size, dispersion, and the overall uniformity of the composite material's microstructure. It was noted that at 3 wt% alumina, the distribution within the polyester resin was improved. At 5 wt% alumina, the particles were equally distributed and scattered throughout the matrix. At 7 wt% alumina, some agglomerates were observed. However, at 9 wt% alumina, significant and clear agglomeration was evident, leading to an inhomogeneous distribution of alumina powder throughout the matrix, as shown in Fig. 9(a-d). These microscopic images of the polyester-alumina mixture were captured at 20x magnification, and the test was conducted at room temperature. Dispersion refers to the distribution of alumina particles within the polyester matrix. As the alumina concentration increased, the formation of clusters or agglomerates became more pronounced. Consequently, optical microscope observations of the microstructures revealed that the dispersion of particles at lower weight fractions was more uniform, while higher weight fractions led to increased agglomeration, particle separation, and porosity.

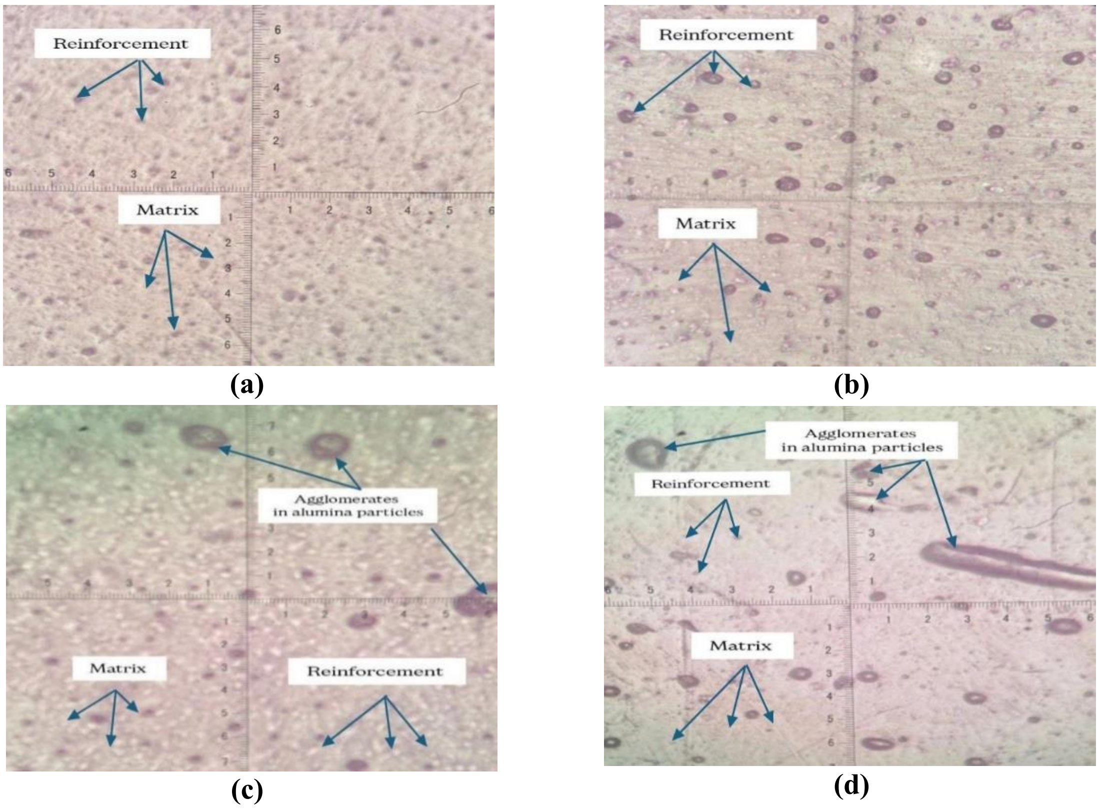

Fig. 9. Optical microstructure images. (a) composite with 3wt.% AL_2O_3 (b) composite with 5 wt.% AL_2O_3. (c) composite with 7 wt.% AL_2O_3. (d) composite with 9 wt.% AL_2O_3 adhesion. Magnification 20x).

5.2. Tensile Properties

All tensile composite specimens underwent testing. Table 2 summarizes the average results obtained from the three measurement datasets. The modulus of elasticity and tensile strength of the tested samples were measured as the weight fraction of particles increased. Fig. 10 depicts the stress-strain diagram. Fig. 11 illustrates the influence of alumina particle content on the maximum tensile strength. Notably, the tensile strength and elastic modulus reached their maximum values of 44 MPa and 0.74 GPa, respectively, in the sample containing 5 wt.% of alumina. This may be attributable to the more uniform dispersion of the fillers (Latief et al.,2019). The enhancement in tensile strength with the addition of nano-sized alumina particles is attributed to their uniform dispersion within the polymer matrix, which facilitates effective stress transfer. This agrees with the earlier report by (Madugu et al., 2009). At alumina content higher than 5 wt.%, the tensile strength and elastic modulus of the composite decreased again, as shown with 9 wt.% of Al_2O_3, where the values were 30 MPa and 0.61 GPa, respectively. According to the literature, the key element influencing the mechanical characteristics of polyester composites is the homogeneous distribution of fillers in the microstructure. Thus, the reduction in tensile strength for the composite with 9 wt.% Al_2O_3 can be explained by the presence of agglomerates, flaws such as voids, and the poor dispersion of the fillers. This is consistent with the earlier report by (Latief et al.,2019).

Table. 2. Tensile test results of composite material of polyester reinforced with alumina particles

Wt%	Elastic modulus E (GPa)	average	Tensile strength (MPa)	Average
0%	0.62 0.68 0.71	0.67	33 35 43	37
3%	0.69 0.71 0.79	0.73	35 38 41	38
5%	0.69 0.75 0.78	0.74	39 44 49	44
7%	0.58 0.64 0.67	0.63	39 43 47	43
9%	0.57 0.62 0.64	0.61	27 30 33	30

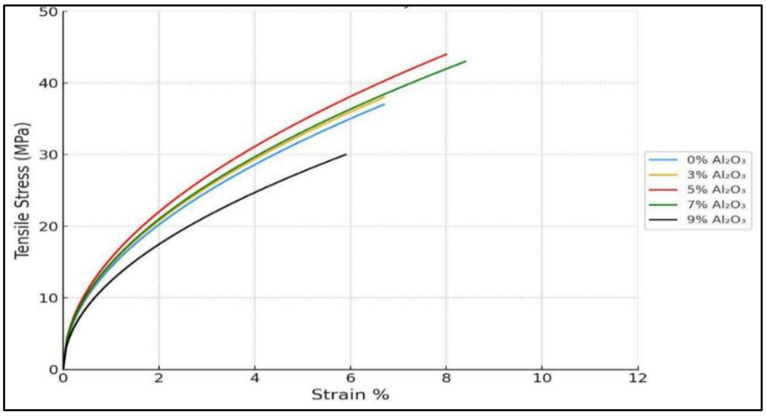

Fig. 10. Stress strain diagram of polyester resin-filled AL_2O_3 composite with varying weights.

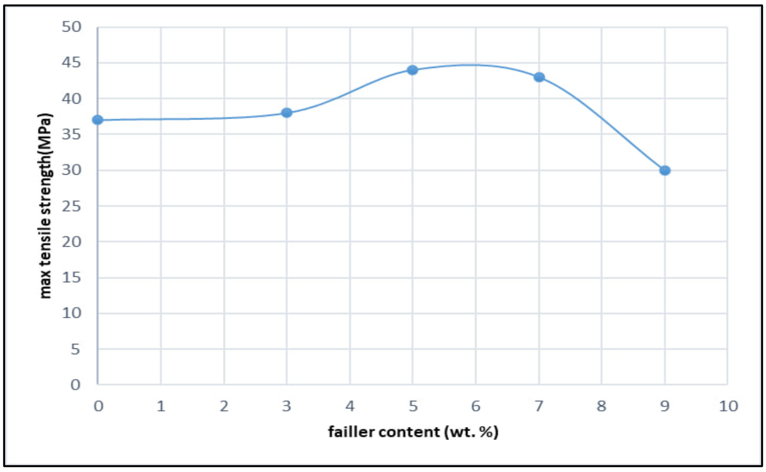

Fig. 11. Max strength vs. AL_2O_3 filler content diagram.

5.3. Hardness testing

Fig. 12 shows the hardness values of composites as a function of Al_2O_3 content. Hardness is defined as a material's resistance to localized plastic deformation. As shown in Fig. 12 and Table 3, the addition of Al_2O_3 fillers increased the hardness of the composites. The hardness of a material can be related to the bonds between the molecules, particularly the filler and the polymer matrix. Therefore, a higher hardness is often indicative of a more uniform dispersion of Al_2O_3 in the polyester matrix.

As shown in Fig. 12, the maximum hardness was achieved by the composites containing 5 wt.% Al_2O_3 (Latief et al., 2019). The reason for the increase in hardness when adding alumina particles to polyester is the hard nature of alumina, which means that it adds great resistance to deformation when mixed with a relatively soft polymeric material such as polyester. The presence of hard particles within the polyester matrix hinders the movement of the polymer chains and thus reduces the surface's susceptibility to abrasion or scratching, which manifests as an increase in surface hardness. The added particles hinder the movement of the polyester chains, leading to an increase in the material's resistance to mechanical deformation and thus higher hardness. This agrees with the earlier report by (Gao et al., 2022). Conversely, composites with higher alumina content (e.g., 9 wt.%) showed a decrease in hardness, likely due to irregular distribution.

Table.3. hardness test result of composite material of polyester reinforced with alumina particles.

Wt.%	Hardness shore (D)	average
0%	81.8 82.1 83.4	82.4
3%	81.2 82.5 85.3	83
5%	81.8 83.1 85.3	83.4
7%	81.7 83.3 84.3	83.1
9%	79.5 79.1 80.5	79.7

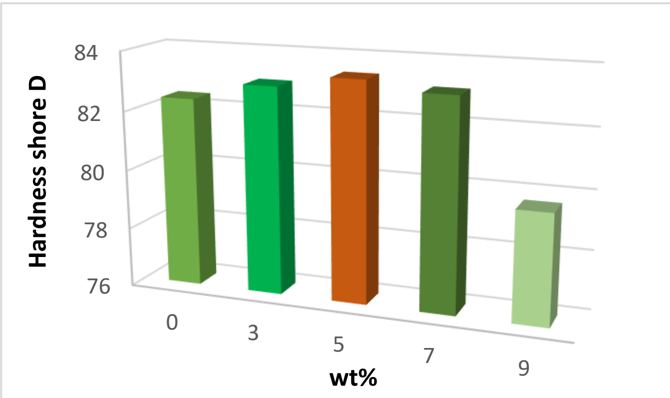

Fig. 12. Variation in hardness with Al_2O_3 wt. %

5.4. Impact strength

Several factors influence the impact strength of a particle composite, including the strength of the reinforcement, the adhesion strength of the reinforcement to the matrix, and the fracture nature of the matrix. Fig. 13 displays the impact strength of bulk polyester and polyester samples reinforced with alumina.

Regarding the bonding between the alumina filler and the polyester matrix, the best results were obtained when the alumina content was 5 wt.%. Table 4 summarizes the impact test values for specimens at different aluminum oxide concentrations.

The reason for the increased impact strength when adding aluminum oxide granules to polyester can be attributed to the presence of good bonding between the Al_2O_3 granules and the polyester chains. This helps absorb energy rather than break the material, thereby increasing its shock resistance (Farhan and Hussein, 2020).

Conversely, the decrease in impact energy at 9 wt.% particle concentration can also be explained as a result of the reduced plasticity of the composites and the decreased deformability of the matrix phases. As the filler content increases, there is often poor surface contact and reduced adhesion/bonding between the polyester matrix and the alumina. This leads to the creation of microcracks and stress concentration points at the moment of impact, which consequently requires less energy to initiate and propagate cracks in composite materials. This, in turn, reduces the amount of impact energy absorbed for fracture.

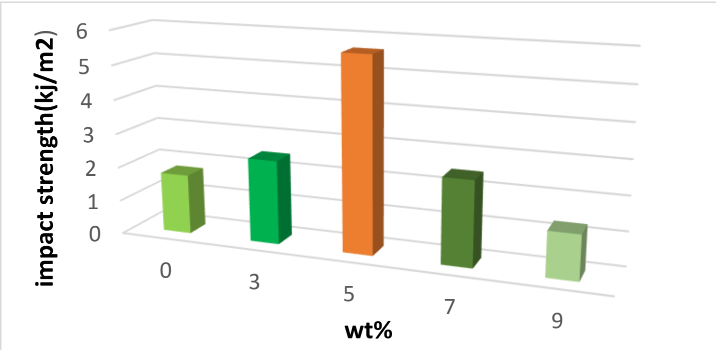

Fig. 13. Variation in impact energy with AL_2O_3 wt.% .

Table 4. impact result of composite material of polyester reinforced with alumina particles.

Wt.%	Impact strength	average
	1.545	
0%	1.901	1.755
	1.820	
	1.965	
3%	2.499	2.453
	2.895	
	4.679	
5%	6.727	5.643
	5.523	
	2.125	
7%	2.501	2.468
	2.778	
	1.102	
9%	1.290	1.301
	1.511	

5.5. Standard Deviation and Variance

The distribution is used to determine the variance or standard deviation of the measurements. To determine these parameters, first compute the deviation (error) di of each measurement xi from the mean value x_{mean} in a collection of measurements x_1, x_2. x_n. of a constant quantity, the most likely true value is the mean given by:

$$x_{mean} = \frac{x1+x2+\cdots+xn}{n} \quad (1)$$

$$d_i = x_i - x_{mean} \quad (2)$$

The variance (Vs) of the set of measurement is formally defined as the mean of the squares of the deviations

$$v_s = \frac{d1^2+d2^2+\cdots+dn^2}{n} \quad (3)$$

The standard deviation (σ) of the set of measurement is defined as the square root of the variance:

$$\sigma = \sqrt{v_s} = \sqrt{\frac{d1^2+d2^2+\cdots+dn^2}{n}} \quad (4)$$

Unfortunately, these mathematical definitions for the variance and standard deviation of data are constructed with regard to an infinite population of data values, but in all practical circumstances, we can only have a finite collection of observations. The mean value x_m of a finite set of measurements will deviate from the genuine mean m of the theoretical infinite population of measurements of which the finite set is a member. This suggests that there is an error in the mean value x_{mean} use to determine di in Eq.2. Because of this, Eqs.3 and 4 Provide a biased estimate that tends to underestimate the variance and standard deviation of an endless set of measurement. Applying the Bessel correction factor(n/(n-1)) to the formula for Vs in Eq.3 allows for a more accurate estimate of the variance of an infinite population (Holman, 2001).

$$v = \left(\frac{n}{n-1}\right) vs = \frac{d1^2+d2^2+\cdots..+dn^2}{n-1} \quad (5)$$

Now we find calculate the standard deviation and Variance for each of the following tests: tensile, modulus of elasticity, hardness and impact test according to the mathematical relationships shown above.

Tensile test

$xmean = 38.4$

$d1 = -1.4\,; d2 = -0.4\,; d3 = 5.6;\; d4 = 4.6\,; d5 = -8.4$

$vs = 31.3\,;\; \sigma = 5.60$

modulus of elasticity

$\sigma = 0.421$

hardness test

$\sigma = 1.51$

impact test

$\sigma = 1.71$

The standard deviation of four mechanical properties was analyzed: tensile, modulus of elasticity, hardness, and impact. The results showed that the highest deviation was in the tensile test (5.60), followed by the impact test (1.71), indicating significant variation among samples, due to inhomogeneous particle distribution or the presence of manufacturing defects such as gaps or microcracks. In contrast, the lowest deviation was in the modulus of elasticity (0.421), reflecting good homogeneity and stability. While the hardness showed a medium variation (1.51), which may be related to the variation in surface composition or particle distribution. Fig.14 shows a visual comparison of these variations.

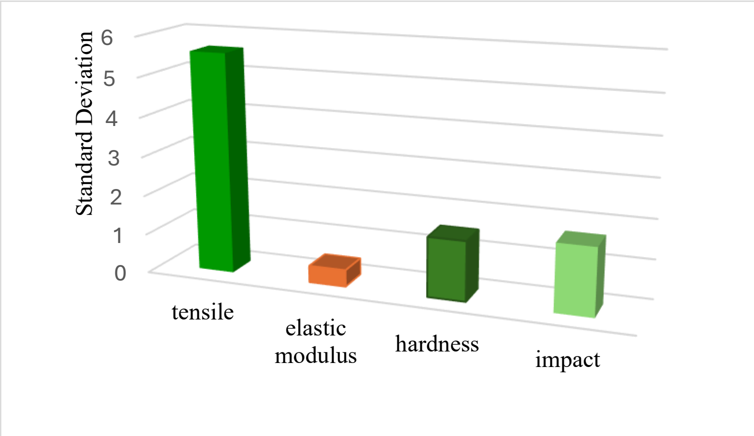

Fig. 14 standard deviation of mechanical properties.

6. CONCLUSIONS

The mechanical properties of composite materials made of polyester reinforced with alumina nanoparticles were experimentally investigated. The alumina particles were incorporated at various weight ratios (0, 3, 5, 7, and 9 wt%). Based on the findings, the following conclusions can be drawn:

The addition of alumina oxide nanoparticles to unsaturated polyester resin enhances the mechanical properties of these composites, peaking at 5 wt%. Beyond this concentration (at 7 wt% and 9 wt%), the properties decreased due to the agglomeration of alumina nanoparticles, which resulted in heterogeneous dispersion.

The addition of alumina oxide nanoparticles to unsaturated polyester significantly improves the mechanical properties of the composite. At 5 wt% (the optimal concentration), tensile strength increased by 18.91%, elastic modulus by 10.44%, hardness by 1.21%, and impact resistance by 221.53% compared to the unreinforced sample..

The agglomeration of particles can cause a reduction in the mechanical properties of materials due to the formation of stress concentration zones.

Optical microscope images showed an almost uniform distribution of particles throughout the matrix at 5 wt% alumina content.

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