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MECHANICAL CHARACTERIZATION OF SIC–AL_2O_3 REINFORCED PLA HYBRID COMPOSITES FABRICATED FOR FUSED DEPOSITION MODELING

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ABSTRACT

As FDM technology gains wider acceptance, there has been an increasing interest in polymers with improved mechanical properties. Despite its popularity due to biodegradability, easy processability, and environmental friendliness, PLA usually displays poor performance in terms of stiffness and toughness necessary for engineering applications. To improve the material behavior, hybrid PLA composites reinforced with SiC and Al_2O_3 nanoparticles were fabricated and experimentally analyzed. Three different hybrid filler compositions were used, while keeping the total amount at 3 wt.%, including: 2.25 wt.% SiC + 0.75 wt.% Al_2O_3, 1.5 wt.% SiC + 1.5 wt.% Al_2O_3, and 0.75 wt.% SiC + 2.25 wt.% Al_2O_3. Composite filaments were made by extruding and further used for manufacturing test specimens. Tensile, flexural, compression, and impact tests were conducted to characterize the samples' behavior.

According to the experimental results, the use of ceramic fillers led to an improvement in the mechanical properties of PLA. From all compositions, the highest tensile strength (53.46 MPa) and compressive performance were shown by the sample with an equal proportion of SiC and Al_2O_3 fillers (1.5 wt. %). In turn, the flexural and impact resistance showed maximums for the sample with the majority of Al_2O_3 (0.75 wt. % SiC + 2.25 wt. % Al_2O_3), with 94.90 MPa flexural strength recorded. Thus, the positive effect on mechanical properties may be explained by the synergistic effect of the filler composition used in the analysis.

Keywords: Polylactic Acid, Silicon Carbide, Aluminum Oxide, Hybrid Composites, Fused Deposition Modeling, Tensile Strength, Flexural Strength, Compression Strength, Impact Strength.

1. INTRODUCTION

Additive Manufacturing (AM) technology, otherwise referred to as 3D printing, has recently evolved into an innovative means of manufacture due to the creation of complex geometries from CAD designs through layer-by-layer formation. Fused Deposition Modeling (FDM) is among the many types of additive manufacturing technologies that have gained popularity because of various reasons such as low cost, easy fabrication process, minimal material wastage, and the creation of complicated geometries [1, 2]. Because of these advantages, FDM technology has been applied in different industries including aerospace, automobiles, biomedical, consumer goods manufacture, and rapid prototyping applications [3].

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PLA is among the thermoplastic polymers commonly utilized in FDM technology due to its biodegradability, recyclability, low melting temperature, and easy printing properties [4]. This polymer is made from renewable sources such as corn starch and sugarcane, giving it an advantage over conventional fossil fuels since it is sustainable and eco-friendly [5]. PLA produces parts that are highly accurate and minimizes warpage while printing. Despite these characteristics, PLA is not suitable for engineering applications due to its low tensile strength, brittleness, poor impact properties, and low loading capacity [6].

In order to address these shortcomings, several research works have concentrated on strengthening PLA by using different types of fillers and nanoparticles in order to strengthen its mechanical, thermal and tribological properties [7]. Different reinforcements such as carbon fibers, glass fibers, ceramics particles, and nanoparticles have been used in combination with PLA in order to increase the stiffness, strength, wear resistance and thermal stability of these materials [8]. Ceramic nanoparticles among other types of reinforcements have received much interest due to their hardness, thermal resistance, good wear resistance, and chemical stability [9].

Silicon carbide (SiC) is a ceramic material that has high hardness, high elastic modulus, high thermal conductivity, and high abrasion resistance [10]. Inclusion of SiC particles into polymeric matrices results in better mechanical strength, stiffness, and abrasion resistance. Another ceramic material which has received much attention as a result of having high hardness, corrosion resistance, and thermal stability is aluminum oxide (Al2O3). Aluminum oxide has received interest as a suitable reinforcement material for use in polymeric matrices [11].

Many scientists studied the effect of the separate filler ceramics in the PLA composites and obtained noticeable improvement in terms of tensile strength, flexural strength, hardness, and thermomechanical properties [12-14]. At the same time, there are not many studies that involve hybrid nanocomposite reinforcement systems. The usage of hybrid composites based on two types of nanoparticles could cause the occurrence of synergetic effect that will allow combining the most important features of both reinforcements and thus achieving better performance of the composite material in comparison with materials with single filler. It is expected that the inclusion of SiC and Al_2O_3 nanoparticles into a PLA matrix might improve stiffness, strength, toughness, and dimensional stability because of better distribution and interaction of nanoparticles within the composite material.

The performance of hybrid polymers is strongly associated with many factors such as the concentration of fillers, the size of particles, quality of mixing, bonding between components, and others. Thus, it is crucially important to conduct comprehensive experiments to define the optimal combination of fillers and their effect on the physical and mechanical properties of the produced composite. Such studies will allow using the proposed technique for developing polymer composite with the required properties and using the proposed method for creating the hybrid composite material based on PLA to increase its applications within engineering fields.

Thus, in the present research, PLA-based nanocomposites reinforced with silicon carbide (SiC) and aluminum oxide (Al_2O_3) nanoparticles were produced via melt-blending and filament extrusion. To achieve this, three different compositions of the filler were selected with an equivalent total filler loading of 3 wt.% of all composites. Afterward, the filament samples of the nanocomposites were manufactured, and FDM-based printing was performed to fabricate test specimens. Tensile, bending, compression, and impact tests were carried out to analyze the effect of the hybrid nanocomposite composition on mechanical properties of the created composites.

2. MATERIALS AND METHODS

2.1 Materials

Polylactic acid (PLA) pellets have been chosen as a matrix material due to their biodegradability, processability, and widespread use in fused deposition modeling (FDM) [17]. Two types of ceramic nanopowders, silicon carbide (SiC) and aluminum oxide (Al_2O_3), were selected as fillers as a result of their high hardness, wear-resistance, thermal stability, and other favorable mechanical characteristics suitable for improving polymer composite properties [18,19].

In order to evaluate the effect of the combination of fillers, three different mixes were designed by keeping the total mass fraction of the fillers constant at 3 wt.%, i.e., the mass fractions were as follows: 1.5 wt.% SiC and 1.5 wt.% Al_2O_3; 0.75 wt.% SiC and 2.25 wt.% Al_2O_3; and 2.25 wt.% SiC and 0.75 wt.% Al_2O_3. This particular choice of mass fraction was guided by literature data regarding the positive effect of the ceramic nanoparticles on PLA composites [20, 21].

2.2 Fabrication of Hybrid Composite Filaments

The exact amounts of SiC and Al_2O_3 nanoparticles were accurately weighed in accordance with their respective reinforcement concentrations. First, the nanoparticle powders were properly mixed to yield a homogeneous hybrid reinforcement composition. The synthesized ceramic composite powder was further mixed together with the PLA particles to achieve a well-dispersed nanoparticle reinforced plastic [22].

The resulting composite material was fabricated into filaments via the filament extrusion process, which is necessary for FDM printing. The extrusion process was carried out under precise control of the filament diameter, which amounted to 1.75 mm to facilitate proper feeding through the printer nozzle. The filaments were then cooled down to room temperature and sealed in moisture-proof containers [23].

2.3 Development of Test Specimens

The mechanical testing samples were designed using CAD software named Solid Works. The designs of the sample sizes were made based on the dimensional specifications provided by the ASTM standards. The tensile samples were designed based on ASTM D638 Type IV, the flexural samples based on ASTM D790, the compression samples based on ASTM D695, and the Izod impact samples based on ASTM D256 [24].

After finishing the design of the samples using the CAD model, the file was exported as an STL file and imported to slicing software to prepare the tool paths for FDM printing. The dimensional specifications of the tensile, flexural, compression, and impact test specimens used in this study are summarized in Table 2.1.

Table 2.1 Dimensions of Mechanical Test Specimens

Test	Standard	Dimensions (mm)
Tensile	ASTM D638 Type IV	Overall Length = 115, Gauge Length = 33, Gauge Width = 6, Grip Width = 19, Thickness = 3.2
Flexural	ASTM D790	Length = 127, Width = 12.7, Thickness = 3.2
Compression	ASTM D695	Length = 12.7, Width = 12.7, Thickness = 25.4

Izod Impact	ASTM D256	Length = 63.5, Width = 12.7, Thickness = 3.2, Notch Depth = 2.54, Notch Angle = 45°

2.4 Fabrication of Specimens Using FDM

Specimens were manufactured via the FlashForge Inventor Series FDM printer. Prior to specimen production, several preliminary printings were carried out to establish suitable processing conditions that ensure accurate printing with high quality interlayers and without printing defects [25].

On the basis of the results obtained during optimization of printing, it is possible to conclude that the parameters used in further specimen manufacturing remained constant for each case. Namely, the temperatures of the nozzle and the print bed were 210°C and 60°C respectively. Layer height and speed were 0.20 mm and 50 mm/s respectively. In order to achieve maximum strength characteristics and decrease inner defects, the infill percentage was kept at 100%. Other printing settings include raster angle ±45°, shell thickness 1.2 mm, nozzle diameter 0.4 mm and post-cooling from the first layers on. All specimens were printed in one build orientation to prevent any influence of different orientations on the results [27].

2.5 Mechanical Characterization

The artificial samples were mechanically tested to determine the influence of hybrid ceramic reinforcement on the properties of PLA-based composites.

Specifically, the tensile test was conducted in accordance with ASTM D638 Type IV in order to measure the tensile strength, Young's modulus, and elongation at break. For determining the flexural properties, ASTM D790 was used, and the values of flexural strength and flexural modulus were obtained from the test. Additionally, the compressive properties were determined via ASTM D695 for assessing the compression strength of the manufactured composites. To determine the impact resistance, the Izod impact test was performed in accordance with ASTM D256 [28]. Three specimens for each type of composite were manufactured and tested in equal conditions. The presented data correspond to the average measurement results. Thus, this approach helped minimize experimental errors [29].

In order to achieve an objective evaluation of each composite, similar printing and testing parameters were used throughout the entire experimental part [30].

3. RESULTS AND DISCUSSION

3.1 Tensile Properties

Table 4.1 provides an overview of the tensile behavior of both pure PLA and SiC–Al_2O_3 reinforced PLA hybrid composites. With the introduction of ceramic nanoparticles, there was a marked enhancement in tensile properties of PLA. For pure PLA, tensile strength stood at 43.75 MPa; however, for the case of reinforced composites, the tensile strengths were within the range of 48.45 MPa to 53.46 MPa.

The PLA reinforced composite having 1.5 wt.% SiC and 1.5 wt.% Al_2O_3 had the best tensile strength at 53.46 MPa, with an approximate enhancement of about 22.2% over pure PLA. In addition, PLA reinforced with 2.25 wt.% SiC and 0.75 wt.% Al_2O_3 had a tensile strength of 53.33 MPa; while on the other hand, tensile strength of 48.45 MPa was recorded for PLA reinforced with 0.75 wt.% SiC and 2.25 wt.% Al_2O_3.

Table 4.1 Tensile Properties

Composition	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation (%)

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Pure PLA	43.75	714.91	4.13
PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3	53.46	924.50	3.70
PLA + 0.75 wt.% SiC + 2.25 wt.% Al_2O_3	48.45	628.14	3.90
PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3	53.33	546.98	3.80

Young's modulus raised from 714.91 MPa for pure PLA to 924.50 MPa for PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3, signifying that addition of stiff ceramic particles improved stiffness of the nanocomposite. There was a minor decrease in elongation at break for all types of reinforced nanocomposites, implying that there is limited chain movement owing to filler addition.

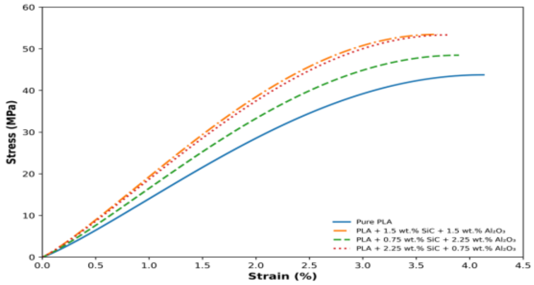

Figure 4.1. Engineering stress–strain curves

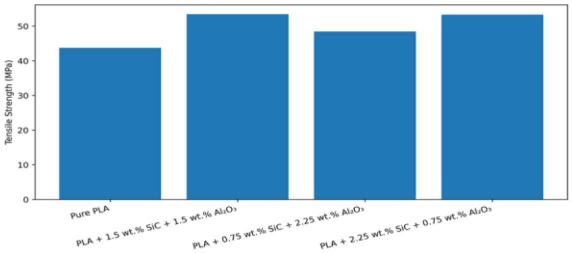

Figure 4.2. Comparative tensile strength of Pure PLA and reinforced composites.

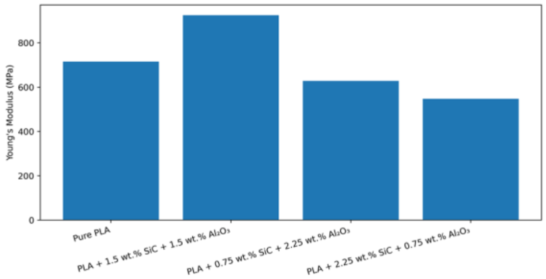

Figure 4.3. Comparative Young's modulus of Pure PLA and reinforced composites.

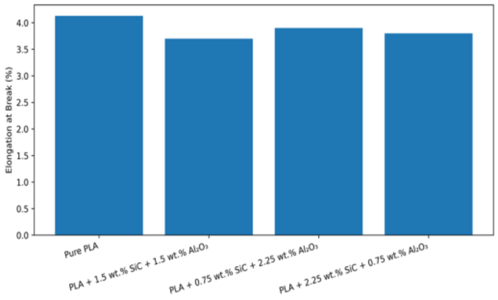

Figure 4.4. Comparative elongation at break of Pure PLA and reinforced composites.

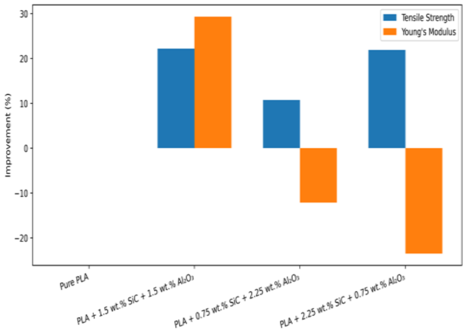

Figure 4.5. Percentage improvement in tensile strength and Young's modulus relative to Pure PLA.

3.2 Flexural Properties

As seen from the findings of the flexural test, there was an appreciable increase in flexural strength after reinforcement. Pure PLA showed a flexural strength of 48.75 MPa, while the reinforced materials recorded much better performance.

In terms of flexural strength, PLA material reinforced with 0.75% wt. of SiC and 2.25% wt. of Al_2O_3 gave the best result, recording a flexural strength of 94.90 MPa and a flexural modulus of 3163.51 MPa. The improved performance is about 94.7% more than that of pure PLA material. This could be due to the stiffer nature of Al_2O_3 nanoparticles under bending stress.

Table 4.2 Flexural Properties

Composition	Flexural Strength (MPa)	Flexural Modulus (MPa)
Pure PLA	48.75	1365.83
PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3	74.09	2233.35
PLA + 0.75 wt.% SiC + 2.25 wt.% Al_2O_3	94.90	3163.51
PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3	78.84	2302.99

The PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3 and PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3 composites also demonstrated substantial improvements, achieving flexural strengths of 74.09 MPa and 78.84 MPa, respectively.

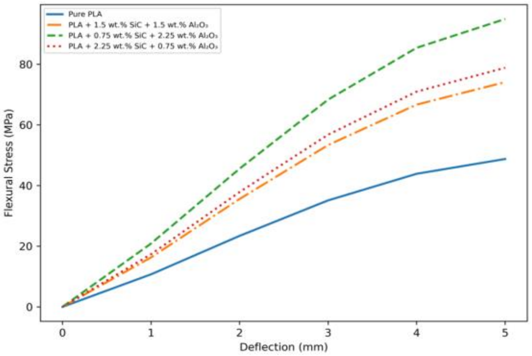

Figure 4.6. Flexural stress–deflection curves.

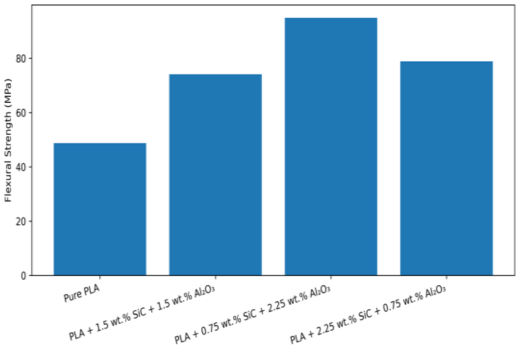

Figure 4.7. Comparative flexural strength.

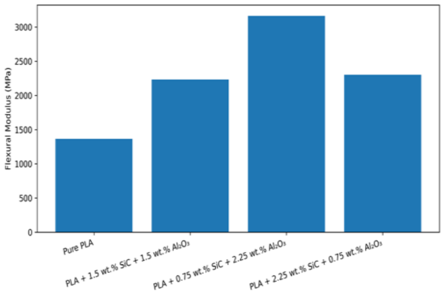

Figure 4.8. Comparative flexural modulus.

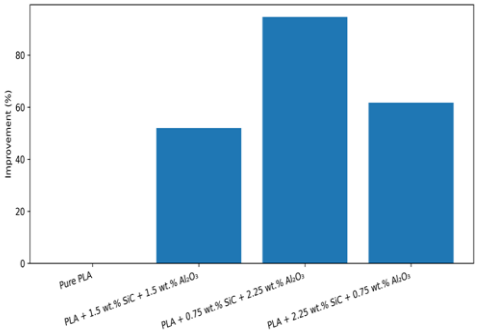

Figure 4.9. Percentage improvement in flexural strength.

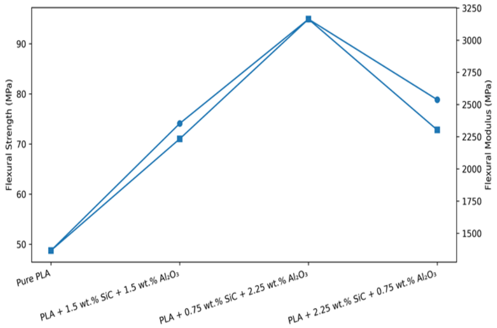

Figure 4.10. Dual-axis comparison of flexural strength and modulus.

3.3 Compression Properties

Compression testing proved substantial improvement in bearing load capacity owing to the use of hybrid reinforcements. Among all tested combinations, PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3 showed excellent compressive performance compared to the other materials. This improvement in compressive performance can be explained in terms of the more even distribution of load and decreased localized deformation by hybrid reinforcement materials. Synergism in strength enhancement occurred because of the well-balanced ratio of the two reinforcing materials; however, the poor performance in compressive performance for PLA + 0.75 wt. % SiC + 2.25 wt. % Al_2O_3 indicated the importance of the reinforcement ratio in the compressive properties of the material.

Table 4.3 Compression Properties

Composition	**Compressive Strength**
Pure PLA	9246.35
PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3	19888.22
PLA + 0.75 wt.% SiC + 2.25 wt.% Al_2O_3	8644.34
PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3	12326.32

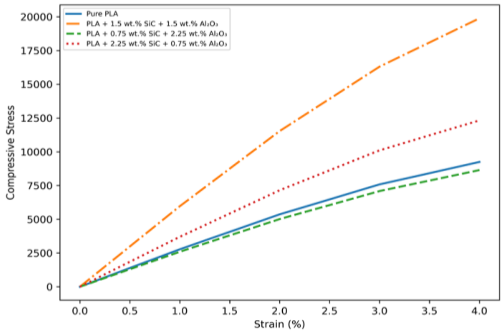

Figure 4.11. Compressive stress–strain curves.

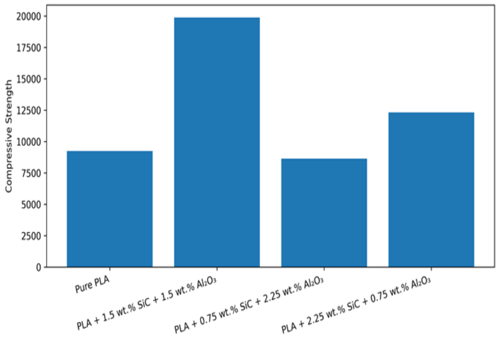

Figure 4.12. Comparative compressive strength.

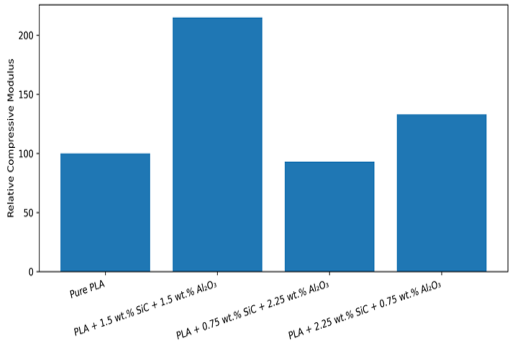

Figure 4.13. Comparative compressive modulus.

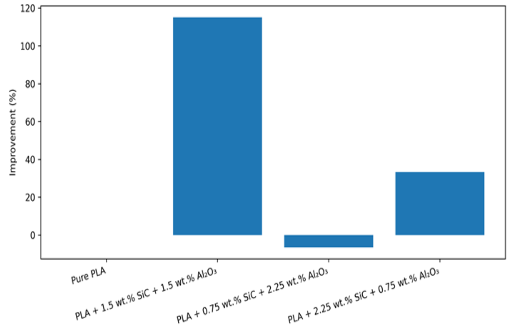

Figure 4.14. Percentage improvement in compressive strength.

4.4 Impact Properties

The results of Izod impact tests revealed that the addition of ceramic nanoparticles had an effect on the energy absorption ability of the

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composites. The impact energy of pure PLA was 0.683 J. The impact energy of PLA + 0.75 wt.% SiC + 2.25 wt.% Al_2O_3 composite was 0.738 J, which is about 8.1% more.

This increased impact resistance of the Al_2O_3-composed composites could be linked to increased energy absorption through the mechanism of crack deflection. It should be noted that a higher impact resistance can also be achieved with the help of PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3 composition, while PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3 composite showed

4.5 Comparative Mechanical Performance

The comparative mechanical performance shows that the composition of the reinforcement material plays an important role in the mechanical properties of the material. The best mechanical performance could be obtained for PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3 composition in terms of tensile and compressive strength.

Table 4.4 Impact Properties

Composition	**Impact Energy (J)**	**Impact Strength (kJ/m²)**
Pure PLA	0.683	16.81
PLA + 1.5 wt.% SiC + 1.5 wt.% Al_2O_3	0.734	18.06
PLA + 0.75 wt.% SiC + 2.25 wt.% Al_2O_3	0.738	18.16
PLA + 2.25 wt.% SiC + 0.75 wt.% Al_2O_3	0.681	16.76

These findings suggest that hybrid reinforcement offers an effective strategy for tailoring the mechanical properties of PLA-based composites for specific additive manufacturing applications.

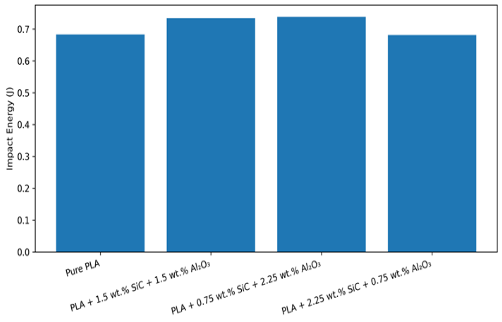

Figure 4.16. Comparative bar chart of impact energy (J).

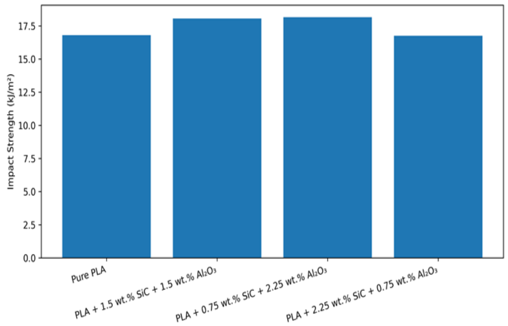

Figure 4.17. Comparative bar chart of impact strength (kJ/m²).

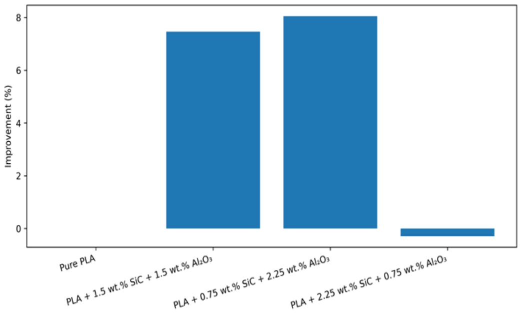

Figure 4.18. Percentage improvement in impact energy relative to Pure PLA.

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5. CONCLUSION

The current research focused on the evaluation of the influence of hybrid ceramic reinforcement on the mechanical properties of PLA composites produced by FDM process. Composite filaments were successfully produced using extrusion method, which involved introduction of SiC and Al_2O_3 nanoparticles into PLA matrix. Hybridization level of ceramic reinforcement was kept constant at 3 wt.%, but ratios of both nanoparticles were varied in order to identify the most effective combination for producing composites with better mechanical properties.

Based on the experimental data, it was found that the use of ceramic nanoparticles resulted in increased mechanical properties of PLA composites compared to the initial polymer. Among all tested samples, the one containing 1.5 wt.% of each type of nanoparticle revealed the best balanced properties. The highest values of tensile strength (53.46 MPa), Young's modulus (924.50 MPa) and compressive strength observed for this sample proved its effectiveness in terms of transferring loads to the reinforcing elements.

When considering mechanical properties that were less related to stiffness but rather determined by strength, the most preferable result was achieved using the composition containing 0.75 wt.% of SiC and 2.25 wt.% of Al_2O_3. The best performance in terms of flexural and impact behavior resulted in the highest values of flexural strength (94.90 MPa) and impact energy (0.738 J).

This increase in mechanical strength can be linked to synergistic effects from the use of SiC and Al_2O_3 nanoparticles that played a role in better stress distribution, high stiffness, and greater resilience against deformations. The dispersion of ceramic particles in the PLA polymer provided additional strength and higher capacity to withstand various loading situations.

In summary, results from this study indicate that using hybrid ceramic reinforcement could effectively mitigate some shortcomings associated with additive-manufacturing of PLA. This work provides promising results about the potential of developed PLA/SiC-Al_2O_3 composite materials as structural elements that can be used in applications where enhanced structural characteristics are required.

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