

Study of PLA/PA12/WA Filament Composite for 3D Printing Process

Nur Munirah Mustaza^a, Farrahshaida Mohd Salleh^{a*}, Nor Arif Aiman Che Mohamad Nor^a, Muhammad Hafiz Musyirif Adnan^a,
 Izdihar Tharazi^a, Nurul Hayati Abdul Halim^a, Ukwueze Bonaventure Emeka^b & Ahmad Hilmi Muhammad^c

^a*School of Mechanical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Malaysia*

^b*Institute of Management and Technology, Nigeria*

^c*PEBBLE3D Sdn Bhd, Lot 691, Level 2&3, Kompleks Hamodal 5, Jalan Lagoon Selatan, 47500 Subang Jaya, Selangor, Malaysia*

*Corresponding author: fsahida@uitm.edu.my

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ABSTRACT

This study investigates the physical and mechanical properties of polymer-ceramic composite filaments composed of Polylactic Acid (PLA), Polyamide 12 (PA12), and Wollastonite (WA) for 3D printing applications, particularly in the medical field. The selected materials—PLA for its use in medical implants, PA12 for its biodegradability and biocompatibility, and WA as a bioceramic suitable for tissue regeneration—were blended to create four compositions, specifically 90PLA/10WA, with PA12 filler content varying from 20 to 40 wt.% in the PLA/WA composite. Rheological analysis was conducted at temperatures of 180°C, 190°C, and 200°C, under loads of 20N, 30N, 40N, and 50N, using a 1 mm die to extrude the feedstock. The objective was to determine the flow behavior index (n) and achieve pseudoplastic behavior ($n < 1$). The extruded composites were then subjected to tensile testing to assess their mechanical properties. The results showed that all composite feedstocks exhibited pseudoplastic behavior. Notably, the composite with 40 wt.% PA12 filler demonstrated superior mechanical properties, achieving an ultimate tensile strength of 28.6 MPa, an ultimate strain of 21.9%, and a Young's modulus of 1220 MPa. These findings suggest that the PLA/PA12/WA composite is a promising material for medical applications, offering both biocompatibility and enhanced mechanical performance.

Keywords: Polylactic Acid (PLA); Polyamide 12 (PA12); Wollastonite (WA); rheo-logical analysis; flow behaviour index

INTRODUCTION

In the modern world, 3D printing is extensively utilized. 3D printing technology is growing in agriculture, healthcare, the automotive industry, and aerospace, where open-source designs are produced in large quantities and customized (Shahrubudin et al. 2019). 3D printing is a sustainable process for industrial use because of its energy efficiency, low post-processing waste, ease of manufacturing, low human involvement, and minimal material waste (Jandyal et al. 2022). This study aims to identify the use of 3D printing, especially in medical applications. This is because it contains numerous possibilities and innovations in the medical field, such as implant material, particularly for bone regeneration

applications, and prosthetics and orthotics (Aimar et al. 2019). 3D printing application is sustainable because of its energy efficiency, low post-processing waste, ease of manufacturing, low human involvement, and minimal material waste.

Polylactic Acid (PLA), combined with Polyamide 12 (PA12) and wollastonite (WA), is studied to evaluate the mechanical properties and composition of the PLA/PA12/WA 3D printed component. PLA is widely used in fused deposition modelling (FDM) due to its low environmental impact and popularity in the medical sector. PLA's mechanical properties range from stiff and strong to soft and elastic, though it is known to be brittle with low impact strength and elongation at break (Miclaus et al. 2017). Its affordability and broad color range make it suitable for

low-stress applications like non-functional prototypes (Cao et al. 2020).

One of the most innovative materials utilized in professional 3D printing is polyamide 12 (PA 12). Due to its mechanical properties, adaptability, and thermal stability, it is an excellent choice for functional prototypes or final products (Martynkova et al. 2021). Owing to its distinctive chemical structure, polyamide 12 (PA12) exhibits a low relative density, low water absorption, low melting point, and poor mechanical qualities (Hooreweder et al. 2010). Furthermore, PA12 may be the optimal material for intricate applications like structural components with exceptional toughness properties and rapid prototyping, given its market price, physicochemical properties, and ease of processing in 3D printing.

Calcium silicate, commonly known as wollastonite, has the chemical formula CaSiO_3 . It belongs to the family of inosilicates and is chemically similar to minerals in the pyroxene group. The calcium silicate wollastonite (WA) exhibits biocompatibility and bioactivity, suggesting that it could be a viable option for bone tissue repair. In recent times, wollastonite has emerged as a promising choice for biomaterials and 3D printing materials due to its advantageous characteristics, such as biocompatibility, biodegradability, nontoxicity, and excellent mechanical properties (Zenebe 2022).

Rheology is the study of the deformation and flow of matter. A comprehensive understanding of the viscoelastic flow behavior of a system can be gained through the rheological characterization of materials. Rheology is important because the rheological responses directly affect the final structure. It plays a significant role in determining the melt processability of polymers and the physical properties of the product (Aho 2011). The focus of studying rheological properties is the viscosity of non-Newtonian fluids, which indicates the flow behavior index, denoted as “ n ”. If the flow behavior index (n) is less than 1, the fluid is pseudoplastic (viscosity decreases with shear rate, also known as shear-thinning). On the other hand, if n is greater than 1, the fluid is classified as dilatant (viscosity increases with shear rate, also known as shear-thickening). When n equals 1, the fluid is categorized as Newtonian, with a constant viscosity (Huang et al. 2018).

The incorporation of WA into the PLA matrix is well-established in injection molding processes but remains relatively underexplored in FDM applications. The PLA/WA composite filament may be stiffer due to the needle-like morphology of the WA bioceramic within the PLA matrix, which enhances its strength (Saravana et al. 2018). However, ductility is also essential for filament production. Therefore, this study utilizes PA12 polymer filler, known for its high elasticity, to improve the ductility of the PLA/

WA composite. The composition PLA/PA12/WA will be used to enhance the potential bioactivity of 3D printed scaffolds for bone tissue engineering and implants. The mixing and blending process will be utilized to produce four different composite feedstocks: Feedstock 1, composed of 80 wt% PLA, 20 wt% PA12, and 10 wt% WA; Feedstock 2, composed of 70 wt% PLA, 30 wt% PA12, and 10 wt% WA; Feedstock 3, composed of 60 wt% PLA, 40 wt% PA12, and 10 wt% WA; and Feedstock 4, composed of 90 wt% PLA and 10 wt% WA. Subsequently, the rheo-logical analysis will be conducted to examine the flow behavior index, n , within a specific temperature range.

METHODOLOGY

MIXING AND BLENDING

The components were mixed and blended to create the feedstocks using an internal mixer (Rheomix 600) shown in Figure 1. The compositions for the composite materials were as follows: Composition 1: 80 wt% PLA, 20 wt% PA12, 10 wt% WA; Composition 2: 70 wt% PLA, 30 wt% PA12, 10 wt% WA; Composition 3: 60 wt% PLA, 40 wt% PA12, 10 wt% WA; and Composition 4: 90 wt% PLA, 10 wt% WA. During feed-stock preparation, specific conditions were maintained, including a temperature of 180°C and a speed of 15 rpm before mixing. Half of the PLA was first added to the tank, and the ‘Run’ button was pressed. After five minutes, the remaining PLA was added at a speed of 30 rpm. The speed was then increased to 45 rpm, and half of the PA12 was added to the tank. Five minutes later, the remaining PA12 was added. Finally, WA was mixed in at a speed of 60 rpm. The entire blending and mixing process took approximately 45 minutes to an hour to homogenize the feedstocks. This process was repeated twice for each composite.

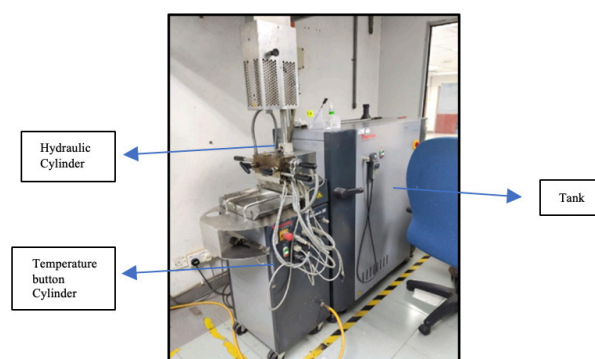


FIGURE 1. Internal Mixer (Rheomix 600)

RHEOLOGICAL TESTING

Rheology tests study the flow and deformation of materials, particularly soft solids, under applied forces or stresses (Ramli et al. 2022). To determine the elasticity and shear viscosity of the materials, a Shimadzu CFT-500EX Capillary Rheometer shown in Figure 2 was used. Factors influencing the rheological properties include temperature, shear rate, and pressure. During testing, temperatures of 180°C, 190°C, and 200°C were applied, along with loads of 20N, 30N, 40N, and 50N. The die diameter for the rheology test was 1 mm, used to extrude the feedstock from the capillary. For the first test, the composite materials were poured into the tank, and the temperature was set to 180°C. Once the capillary die was heated, the sample was forced to flow through the cylinder. This procedure was repeated at temperatures of 190°C and 200°C. Each temperature test was conducted with four different loads. The data obtained from this investigation included shear stress, shear viscosity, flow behavior index (n), and flow activation energy (E_a).

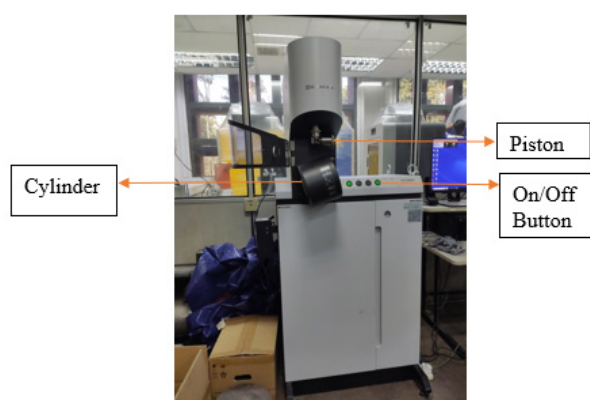


FIGURE 2. Shimadzu CFT-500EX Capillary Rheometer

MECHANICAL TESTING

Wire filaments produced from the rheological study were tested to compare the tensile strength of four compositions: 80 wt% PLA/20 wt% PA12/10 wt% WA, 70 wt% PLA/30 wt% PA12/10 wt% WA, 60 wt% PLA/40 wt% PA12/10 wt% WA, and 90 wt% PLA/10 wt% WA. Tensile tests, which determine how materials behave under tension, were performed to evaluate the mechanical properties of the filaments. In a tensile test, a sample is pulled to its breaking point to measure its ultimate tensile strength (Yalcin, 2021).

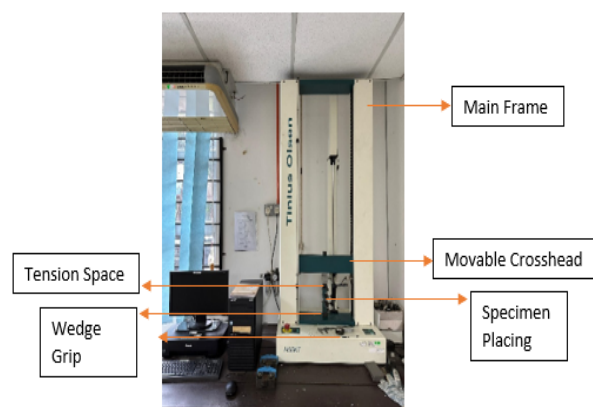


FIGURE 3. Universal testing machine

A Universal Testing Machine as shown in Figure 3 was used to produce stress-strain diagrams and determine the ultimate strength values. The gauge length ranged from 50 to 70 mm, while the filaments were approximately 110 mm long. The tensile test considered factors such as load, thickness, and speed. The test speed was 2 mm/min, the filament thickness was 1.75 mm, and the load cell used was 2.5 kN. Each filament composition was tested five times to ensure accurate results and obtain an average value.

RESULTS AND DISCUSSION

PLA/PA12/WA FEEDSTOCK COMPOSITE

Four feedstocks composition were produced from the mixing and blending process of PLA/PA12/WA composite. The feedstocks composition is Feedstock 1: 80 wt% of PLA/20 wt% PA12/10 wt% WA, Feedstock 2: 70 wt% PLA/30 wt% PA12/10 wt% WA, Feedstock 3: 60 wt% PLA/40 wt% of PA12/10 wt% WA and Feedstock 4: 90 wt% PLA/10 wt% WA. The mixing and blending were conducted using an Internal Mixer (Rheometer 600). Figure 4 shows the raw materials used, including PLA pellets, PA12 powders, and WA powder. Figure 5 displays the granulated feedstocks after the crushing process for each composition. Before conducting the rheological analysis, the granules were stored separately in zip-locked bags based on their composition.

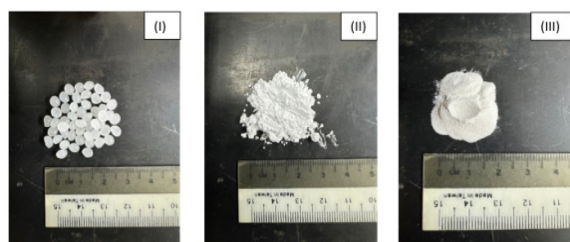


FIGURE 4. (I) Polylactic acid (PLA) pellet; (II) PA12 powders and; (III) WA powder

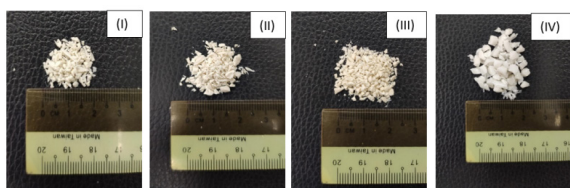


FIGURE 5. (I) 80 wt% of PLA/20 wt% PA12/10 wt% WA granule; (II) 70 wt% PLA/30 wt% PA12/10 wt% WA granule; (III) 60 wt% PLA/40 wt% of PA12/10 wt% WA granule and; (IV) 90 wt% PLA/ 10wt% WA granule

RHEOLOGICAL ANALYSIS

The Shimadzu CFT-500EX Capillary Rheometer was used to analyze the rheological behavior of composite feedstocks, determining whether they exhibit pseudoplastic behavior. The rheology tests were conducted at temperatures of 180°C, 190°C, and 200°C with loads of 20N, 30N, 40N, and 50N to extrude the feedstocks. A die with a diameter of 1 mm was used for the extrusion process. The results were then compared to determine which PLA/PA12/WA composite displayed the best flow behavior index (n) at each specific temperature.

FLOW BEHAVIOR INDEX

The primary analytical focus of the rheological measurement was the viscosity and shear rate of non-Newtonian fluids. Non-Newtonian fluids, which deviate from Newton's law of viscosity, exhibit variable viscosity in response to stress. The flow behavior index (n) indicates the non-Newtonian nature of these fluids, where shear stress is not directly proportional to the deformation rate. In this study, the goal was to achieve pseudoplastic behavior, characterized by an n value between 0 and 1 (Chhabra 2010). The composite comprising 90 wt% PLA/10 wt% WA was established

as the reference for assessing the effects of adding PA12 to the composite. As shown in Table 1, it was demonstrated that the incorporation of WA ceramic into the PLA matrix does not significantly alter the pseudoplastic behavior of the PLA matrix. In fact, the n value tends to remain optimal, within a mid-range value of 0.4 to 0.54, which is between 0 and 1. Notably, the addition of PA12 filler to the PLA/WA composite also maintains pseudoplastic behavior, though with a broader range of n value. For example, composites with 80 wt% PLA/20 wt% PA12/10 wt% WA and 70 wt% PLA/30 wt% PA12/10 wt% WA exhibit slightly higher n value, ranging from 0.6 to 0.8, compared to the 60 wt% PLA/40 wt% PA12/10 wt% WA composite, which shows n value comparable to the reference composite (90 wt% PLA/10 wt% WA), with a range of approximately 0.39 to 0.55. The highest n values for the feedstocks were observed at 200°C, except for the 80 wt% PLA/20 wt% PA12/10 wt% WA feedstock, which had its best n value at 180°C.

From the plotted graphs of shear viscosity versus shear rate (Figures 6, 7, 8, and 9) for all feedstocks at the three temperatures, it is evident that shear viscosity decreases as shear rate increases, consistent with theoretical expectations which confirms that all composites exhibit pseudoplastic behavior. The composite with 90 wt% PLA and 10 wt% WA serves as the reference in this study. The viscosity of the 90 wt% PLA/10 wt% WA composite was observed to range from 69 to 297 Pa.s. The incorporation of PA12 filler into the PLA/WA composite notably altered the viscosity range compared to the reference composite. Specifically, the 80 wt% PLA/20 wt% PA12/10 wt% WA and 70 wt% PLA/30 wt% PA12/10 wt% WA composites exhibited viscosity ranges of 124 to 423 Pa.s. and 148 to 474 Pa.s., respectively. Increasing the PA12 content led to higher viscosity. However, for the 60 wt% PLA/40 wt% PA12/10 wt% WA composite, the viscosity range was between 89 and 420 Pa.s. The higher the temperature, the lower the viscosity. As temperature increases, the thermal energy within the material's molecules also increases, leading to greater molecular mobility and, consequently, a decrease in viscosity. Overall, these viscosity ranges are suitable for FDM 3D printing, which typically operates within a range of 10^2 to 10^5 Pa.s (Bragaglia et al. 2021). The flow behaviour results indicate that all feedstock compositions can be extruded within the temperature range of 180–200°C with low viscosity. This temperature range will guide the settings for filament production.

TABLE 1. Flow behavior index, n of feedstocks at different temperature

Feedstocks	Temperature ($^{\circ}\text{C}$)	Flow behavior index, n
90 wt% PLA/10 wt% WA	180	0.503
	190	0.402
	200	0.538
80 wt% PLA/20 wt% PA12/10 wt% WA	180	0.722
	190	0.658
	200	0.686
70 wt% PLA/30 wt% PA12/10 wt% WA	180	0.614
	190	0.636
	200	0.791
60 wt% PLA/40 wt% PA12/10 wt% WA	180	0.385
	190	0.389
	200	0.549

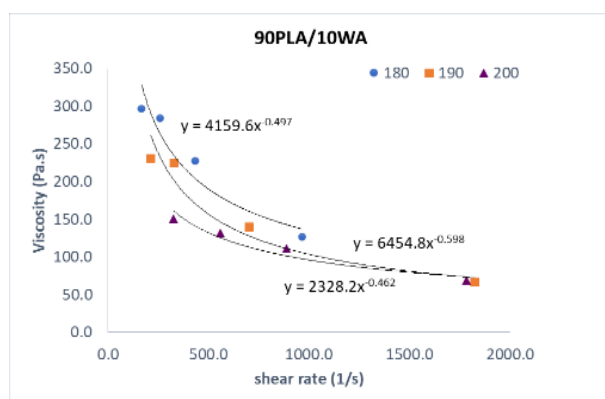


FIGURE 6. Shear viscosity vs shear rate curve of feedstock composition 90 wt% PLA/ 10wt% WA with various temperature

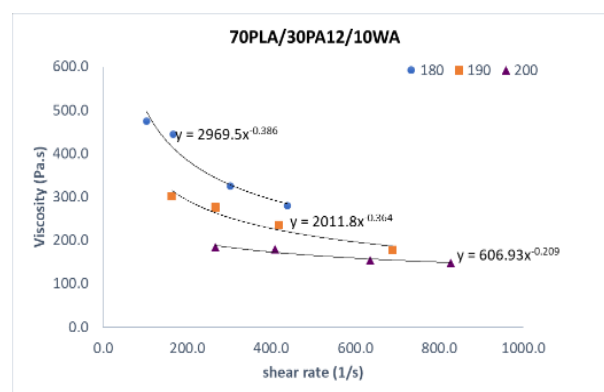


FIGURE 8. Shear viscosity vs shear rate curve of feedstock composition 70 wt% of PLA/30 wt% PA12/10 wt% WA with various temperature.

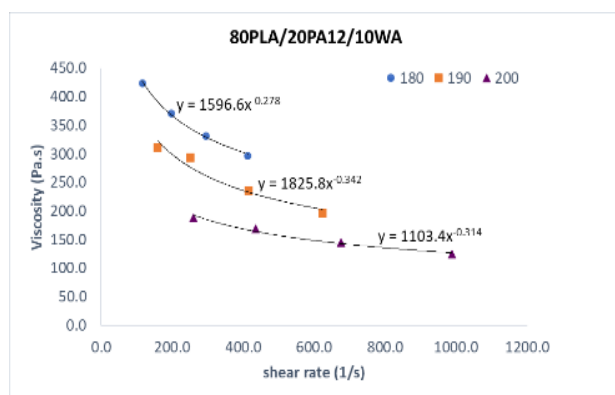


FIGURE 7. Shear viscosity vs shear rate curve of feedstock composition 80 wt% of PLA/20 wt% PA12/10 wt% WA with various temperature

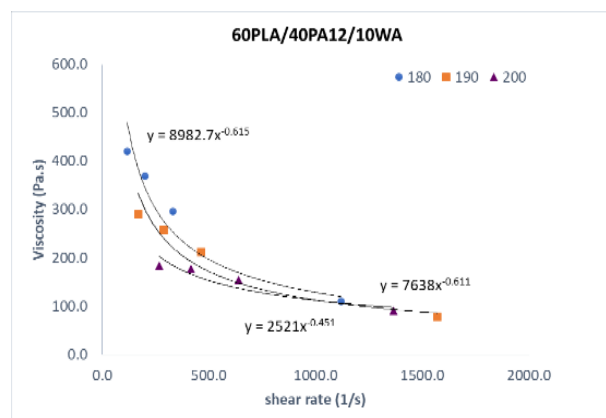


FIGURE 9. Shear viscosity vs shear rate curve of feedstock composition 60 wt% of PLA/40 wt% PA12/10 wt% WA with various temperature

FLOW ACTIVATION ENERGY

The temperature used in the rheological analysis study had an impact on the flow activation energy (E_a) of all feedstocks. The flow activation energy is often employed in the investigation of viscous materials, such as Newtonian and non-Newtonian fluids. The Arrhenius equation is utilized to determine the flow activation energy, establishing a correlation between the chemical reaction and the temperature (Marzuki et al. 2022). The equation for calculating E_a is presented below, where η represents the viscosity of the mixture, denotes the viscosity at the reference temperature, E is the flow activation energy, R is the universal gas constant, and T is the temperature in Kelvin.

$$\eta = \eta_0 \exp\left(\frac{E}{RT}\right) \quad (n < 1) \quad (1)$$

Table 2 shows the result of the E_a value for for Feedstock 1: 80 wt% of PLA/20 wt% PA12/10 wt% WA, Feedstock 2: 70 wt% PLA/30 wt% PA12/10 wt% WA, Feedstock 3: 60 wt% PLA/40 wt% of PA12/10 wt% WA and Feedstock 4: 90 wt% PLA/ 10wt% WA. According to Table 2, the result shows the value of E_a of 80 wt% of PLA/20 wt% PA12/10 wt% WA was higher than the other feedstocks. By referred to the Figure 10, the data trend of the graph is straight line for feedstock of 80 wt% of PLA/20 wt% PA12/10 wt% WA.

TABLE 2. Flow activation energy, E_a of the feedstocks

Feedstocks	Flow activation energy, E_a (J/mol)	Flow activation energy, E_a (kJ/mol)
90 wt% PLA/10 wt% WA	30284.58	30
80 wt% PLA/20 wt% PA12/10 wt% WA	55088.07	55
70 wt% PLA/30 wt% PA12/10 wt% WA	32602.35	33
60 wt% PLA/40 wt% PA12/10 wt% WA	12362.09	12

Figure 10 illustrates the relationship between viscosity and temperature for four different feedstock composites: Feedstock 1 (80 wt% PLA/20 wt% PA12/10 wt% WA), Feedstock 2 (70 wt% PLA/30 wt% PA12/10 wt% WA), Feedstock 3 (60 wt% PLA/40 wt% PA12/10 wt% WA), and Feedstock 4 (90 wt% PLA/10 wt% WA). All compositions show an increase in $\ln \eta$ as $1/T$ increases, indicating that viscosity increases as the temperature decreases. Among the compositions,

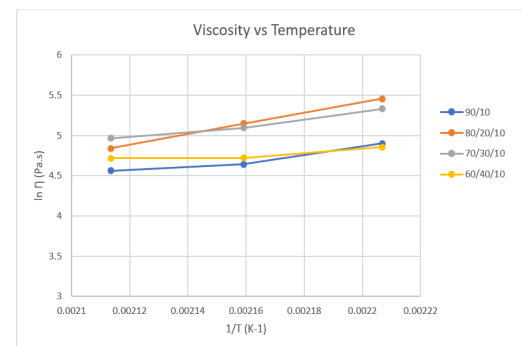


FIGURE 10. Relationship of viscosity and temperature four different feedstock composite

Feedstock 4 (90 wt% PLA/10 wt% WA) has the lowest initial viscosity and exhibits the least steep increase, suggesting more stable viscosity over temperature changes. Conversely, Feedstock 1 (80 wt% PLA/20 wt% PA12/10 wt% WA) has the highest initial viscosity and the steepest increase, indicating greater sensitivity to temperature variations. Feedstocks 2 (70 wt% PLA/30 wt% PA12/10 wt% WA) and 3 (60 wt% PLA/40 wt% PA12/10 wt% WA) fall in between, with Feedstock 3 offering a balance of moderate initial viscosity and a steady rate of increase.

Low activation energy is particularly advantageous for materials that require stable and predictable performance across a range of temperatures. It enhances reliability, efficiency, and energy savings, making it a desirable property for materials. According to Table 2, the flow activation energy (E_a) for the 60 wt% PLA/40 wt% PA12/10 wt% WA composition is the lowest, at 12 kJ/mol. This indicates that the 60 wt% PLA/40 wt% PA12/10 wt% WA composition is the most suitable for applications requiring stable performance in the PLA/PA12/WA composite system.

MECHANICAL TESTING

The tensile tests for all composite filaments were conducted using a Universal Testing Machine and all data were tabulated in Table 3 and illustrated in Figure 12 with standard deviation. Table 3 shows that the ultimate stress value for the 60 wt% PLA/40 wt% PA12/10 wt% WA filament is higher than that of the other filaments, indicating that this composition is less brittle. For the Young's modulus, the 60 wt% PLA/40 wt% PA12/10 wt% WA filament has a value of 1220 MPa, compared to 231 MPa for the 70 wt% PLA/30 wt% PA12/10 wt% WA filament and 525 MPa for the 80 wt% PLA/20 wt% PA12/10 wt% WA filament. The higher secant modulus of the 60 wt%

PLA/40 wt% PA12/10 wt% WA filament suggests superior strength compared to the other composites. This demonstrates that the incorporation of PA12 filler into the PLA/WA composite enhances its ductility, effectively

mitigating the stiffness typically associated with the PLA/WA composite. The stiffness arises from the inclusion of WA ceramic, which has a needle-like morphological microstructure within the PLA matrix (Saravana et al.

2018).

TABLE 3. Data of tensile test for the composite filaments

Properties	60 wt% PLA/40 wt% PA12/10 wt% WA	70 wt% PLA/30 wt% PA12/10 wt% WA	80 wt% PLA/20 wt% PA12/10 wt% WA
Ultimate stress (MPa)	28.6 ± 4.29	13.3 ± 4.78	8.17 ± 5.91
Ultimate strain (%)	21.9 ± 8.62	3.14 ± 2.13	1.71 ± 1.18
Young's modulus (MPa)	1220 ± 319.79	231 ± 116.28	525 ± 75.44
Diameter (mm)	1.75	1.75	1.75

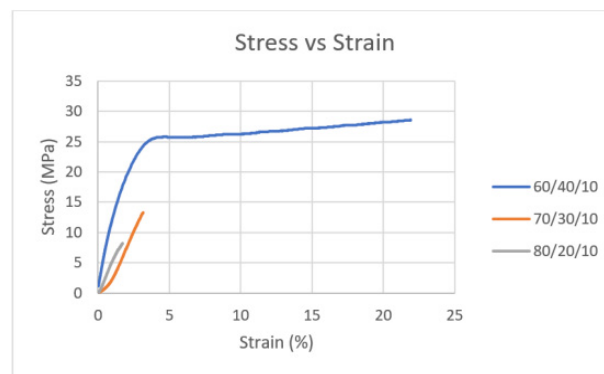


FIGURE 11. Stress vs Strain curve for all filament composites

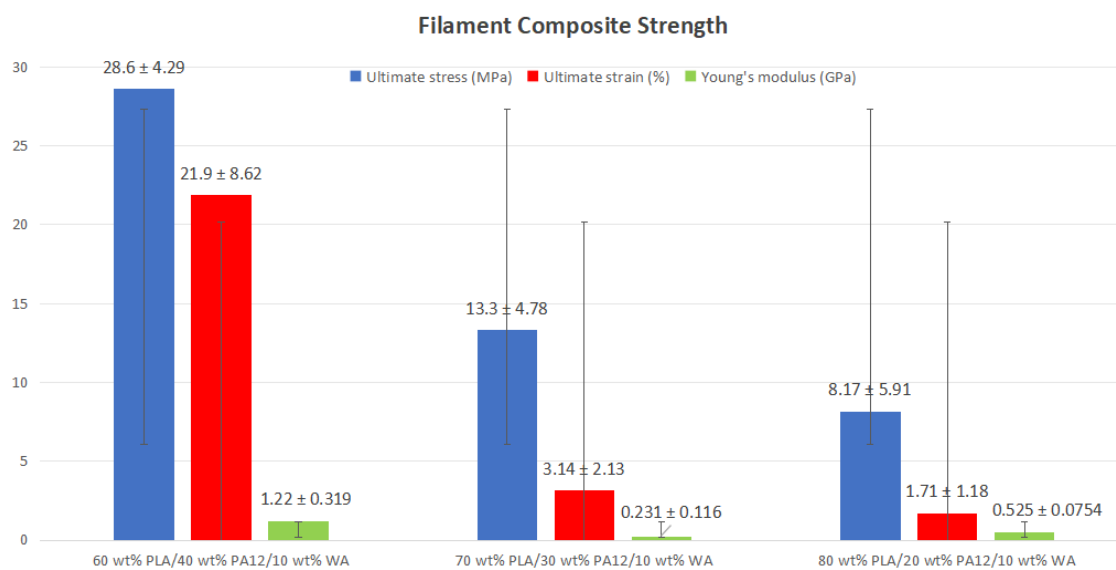


FIGURE 12. Bar graph of all filament composite strength with standard deviation

Figure 11 presents the stress vs. strain curves for the composite filaments with compositions of 80 wt% PLA/20 wt% PA12/10 wt% WA, 70 wt% PLA/30 wt% PA12/10 wt% WA, and 60 wt% PLA/40 wt% PA12/10 wt% WA. The graph reveals that the curve for the 60 wt% PLA/40 wt% PA12/10 wt% WA composite exhibits a ductile behavior, while the curves for the other compositions display more brittle characteristics. The 60 wt% PLA/40 wt% PA12/10 wt% WA filament not only demonstrates the highest strength, as indicated by its ability to withstand the highest stress, but also shows the greatest ductility with the highest strain value.

Conversely, the 80 wt% PLA/20 wt% PA12/10 wt% WA and 70 wt% PLA/30 wt% PA12/10 wt% WA compositions are progressively less ductile. In terms of stiffness, represented by the initial slope of the stress-strain curve, the 60 wt% PLA/40 wt% PA12/10 wt% WA composition is the stiffest, indicating it has the highest Young's modulus. According to Letcher et al. (2014), the ultimate strain of pure PLA filaments at a displacement rate of 5 mm/min was 16.0%, which is higher than the ultimate strains observed in the 80 wt% PLA/20 wt% PA12/10 wt% WA and 70 wt% PLA/30 wt% PA12/10 wt% WA composites, recorded at 3.14% and 1.71%, respectively. However, the ultimate strain of the 60 wt% PLA/40 wt% PA12/10 wt% WA composite was 21.9%, exceeding that of the pure PLA filament.

The incorporation of 40 wt% PA12 filler into the PLA/WA composite resulted in the highest mechanical strength compared to the 20 and 30 wt% PA12 composites, even surpassing that of the commercial PLA filament. The primary purpose of adding PA12 filler to the PLA/WA composite was to enhance the mechanical strength and ductility of the composite filament. The findings confirm that a 40 wt% PA12 filler content is optimal for achieving a composite filament with high ductility and durability, making it particularly suitable for medical applications, especially in bone implant use.

CONCLUSION

At the conclusion of the study, feedstocks with different PLA/PA12/WA compositions were prepared using the mixing and blending process. The compositions were as follows: Feedstock 1 (80 wt% PLA/20 wt% PA12/10 wt% WA), Feedstock 2 (70 wt% PLA/30 wt% PA12/10 wt% WA), Feedstock 3 (60 wt% PLA/40 wt% PA12/10 wt% WA), and Feedstock 4 (90 wt% PLA/10 wt% WA). The feedstocks were successfully crushed into small granules using a crusher machine. The rheological properties of these feedstocks were analyzed at temperatures of 180 °C,

190 °C, and 200 °C. All feedstocks exhibited pseudoplastic behavior ($n < 1$), with the flow behavior index (n) ranging from 0.385 to 0.791. Lower values of flow activation energy (E_a) were associated with more significant changes in viscosity with temperature. The study results indicate that the 60 wt% PLA/40 wt% PA12/10 wt% WA composition is the most favorable due to its optimal flow behavior index (n) and flow activation energy (E_a) values.

Here are some suggestions for additional study to prevent the same issues from arising in the subsequent experiment. Firstly, to avoid moisture and agglomeration, it is advisable to ensure that the raw materials are dry or subjected to overnight heating at 40°C in a furnace. Only after this can further operations be carried out, especially mixing. For materials prone to air entrapment, it is worth considering the use of vacuum mixing to eliminate trapped air bubbles, which can have an impact on the properties of the final product. Filaments, in particular, are susceptible due to their brittle mechanical qualities. Therefore, a new polymer-ceramic composite filament consisting of PLA/WA with the incorporation of PA12 polymer filler is proposed as an alternative composite specifically for medical implant applications. An optimal PA12 filler composition of 40 wt% is expected to reduce brittleness and increase ductility, while the biological activity of the WA bioceramic enhances the suitability of the composite for medical applications using FDM technology.

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DECLARATION OF COMPETING INTEREST

None.

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