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PLA/Wollastonite Composite Filaments for 3D Printing Application: Rheological Properties and Extrusion

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ABSTRACT

The 3D printing has fundamentally transformed the approach to object design and manufacturing, enabling the cost-effective production of intricate and customized components. This study aims to investigate the rheological and mechanical properties of Poly-lactic Acid (PLA)/Wollastonite (WA) composite filaments for 3D printing, particularly for bone implant applications. The powder composition used is 90 wt.% PLA and 10 wt.% WA. The blending process was conducted at a temperature of 190°C and a torque value of 30 rpm. Capillary rheometer testing was carried out at temperatures of 160 °C, 170 °C, and 180 °C on the feedstock. The rheological results of the feedstock show pseudoplastic flow behavior, where viscosity decreases with an increase in shear rate. The flow behavior indices obtained at all temperatures are 0.87, 0.79, and 0.49 respectively. At these temperatures, the flow behaviour index is below 1 ($n<1$), indicating pseudoplastic flow and compatible for the FDM process. The optimal filament extrusion parameters were determined to be 170°C, 190°C, 180°C, and 140°C for the four temperature zones, respectively, with a screw speed of 4.0 rpm and a spool speed of 40 mm/s. The Taguchi Method was employed to evaluate the filament's quality and strength using three different printing settings. The density of the PLA-WA composite filament was 1.32 g/cm³. These results suggest that the PLA-WA composite has promising candidate use in 3D printing applications.

Keywords: Composite filaments, Poly-lactic Acid; Wollastonite; Extrusion; Additive Manufacturing; Rheological properties.

INTRODUCTION

Plastics have become an essential component of modern medicine, with various medical applications relying on different types of medical-grade plastic (Czuba 2014). Plastics are used in the manufacturing of medical devices and equipment, including implants, synthetic lenses, joints, and ligaments, as well as in the production of biodegradable polymers like polylactic acid (PLA) derived from animal or plant sources such as cellulose, starch, corn, fish waste and kitchen waste (Jin et al. 2019). Plastics may be made to have a wide range of qualities and are more economical and energy-efficient to create than other materials like metal or glass. Owing to these properties, polymers find employment in a wide range of health applications, including tissue engineering, joint replacements, sterile packaging for medical equipment, and disposable needles and IV bags (North & Halden 2013). The recycling of medical plastic waste is also possible through proper coordination between the healthcare sector and recycling industries, with new recycling technologies being developed (Joseph et al. 2021). Therefore, it is evident that plastics have become an integral part of modern medicine, contributing to the advancement of medical technology and the improvement of patient care.

Polylactic Acid (PLA) is a commonly utilized thermoplastic material in the field of additive manufacturing. PLA is a polymer that is both biodegradable and bioactive (Meor Sha & Zulkifli 2022). It is created from renewable resources, making it a more sustainable alternative to other types of plastics. PLA is utilized to produce biomaterials that are suitable for various applications, such as the creation of anatomical scaffolds for biomedical purposes (Bakhshi et al. 2023; Puppi et al. 2022). PLA is additionally utilized in the production of cellular structures for biomedical purposes (Singhvi et al. 2019). PLA is characterized by its ease of printing and lack of necessity for intricate post-processing procedures. Nevertheless, it is advisable to utilize PLA for components lacking significant mechanical intricacy, as it possesses lower strength and flexibility compared to other thermoplastics. The brittleness of PLA is a consequence of its rigid structure, which also leads to its inadequate durability and impact resistance. PLA's reduced glass transition temperature (T_g) further diminishes its thermal resistance in comparison to ABS and PEEK filament materials. The temperature at which PLA can be processed is limited to lesser ranges (Musa et al. 2022).

Polylactic Acid (PLA) is a versatile 3D printing material with various applications. While PLA is known for its lightweight, durable, and biodegradable properties, it may require reinforcement with other materials for specific applications that demand enhanced characteristics. For instance, in additive manufacturing, PLA composites have been developed for advanced applications, such as hard tissue engineering. Research has focused on the additive manufacturing of PLA composites, including PLA-Mg composite scaffolds, for hard tissue engineering applications due to PLA's excellent properties (Lee et al. 2023). Additionally, PLA›s use in 3D printing extends to creating detailed prototypes, consumer products, eco-friendly packaging materials, and costumes in the entertainment industry (Haleem et al. 2023). Furthermore, PLA's biocompatibility and ability to enhance bone formation make it a promising candidate for implant materials in bone tissue engineering. Therefore, while PLA is suitable for a wide range of applications, its combination with inforce materials, such as in the form of composites, can further expand its potential for specific uses, including in the biomedical and additive manufacturing fields (Bakhshi et al. 2023).

Bio-silicate constitutes a significant category within bioactive ceramics, establishing a direct chemical bond with bone tissue. (Hench 2006) pioneered the development of the oldest bioactive material, 45S5 bioglass. Ceramics based on CaO-SiO2 are regarded as promising candidates for synthetic bone substitutes owing to their exceptional bioactive and biodegradable attributes (Shamsudin et al. 2017). Wollastonite (WA), a calcium inosilicate mineral, has gained attention for its potential application in implant materials, particularly in bone tissue engineering. Research has explored the use of wollastonite and its composites, such as wollastonite/titanium oxide nanofiber bioceramic, in the development of implant materials for bone replacement and tissue engineering (Zenebe 2022). Wollastonite's favourable properties, including its biocompatibility and ability to enhance bone formation, make it a promising candidate for various bone tissue engineering applications (Curti et al. 2020). Additionally, the incorporation of wollastonite with titanium has been studied for medical applications, with a focus on its use in composite materials for orthopaedic implants (Zakaria et al. 2019). The unique properties of wollastonite, such as its bioactivity and ability to bond with bone, contribute to its potential as an implant material for bone grafts and orthopaedic applications (Shanmuganantha et al. 2021).

The PLA/WA composite exhibits great potential as a material for implant applications and may be produced in the form of filament for 3D printing. The PLA/WA composite is suitable for implant applications because of its advantageous characteristics, such as biocompatibility, capacity to promote bone development, and mechanical robustness (Saadaldin & Rizkalla 2014). Studies have investigated the utilisation of PLA/Wollastonite composite in the fabrication of implant materials for bone replacement and tissue engineering (Moreno et al. 2023). Moreover, PLA/WA composite can be produced as filament for 3D printing, enabling the creation of intricate structures with exceptional precision and accuracy (Choudhary et al. 2022). The incorporation of PLA and WA features in a composite material can synergistically augment their respective qualities, rendering it a highly promising contender for a wide range of implant applications, such as bone grafts and orthopaedic implants.

Fused deposition modelling (FDM) is an additive manufacturing (AM) technology used to create items from three-dimensional (3D) models. In the past decade, there has been a significant surge in the adoption of FDM technology (Guo & Leu 2013; Horn & Harrysson 2012). It has been said that this technology has the potential to bring about a revolutionary change in production techniques across all sectors (Parandoush & Lin 2017). The layer-by-layer manufacturing approach is employed by a rapid prototype (RP) computer to regulate this technology, enabling the production of parts composed of porous materials (Zhang et al. 2020). FDM typically entails creating a digital design using 3D design software, which is then divided into a sequence of laminations or layers. The data of each layer is transmitted to the printer, which then replicates the design by building each layer sequentially until the final model is achieved. The mechanical characteristics of FDM printed components are influenced by several factors, including the material used, the structural parameters (such as raster angle, infill density, printing orientation, and stacking sequence), and the manufacturing variables (such as printing speed, extrusion temperature and rate, layer time, nozzle transverse speed, and bed temperature) (Junpha et al. 2020; Pertuz et al. 2020; Zhang et al. 2021).

Diverse effects are produced by the parameters utilised in the FDM process on the attributes and properties of the printed material. The input parameters provided to the FDM machine, including but not limited to layer height, infill density, and orientation, have a direct impact on the quality of the printed product (Sammaiah et al. 2020). As a result, the attributes of a product will diverge in accordance with the printing parameters that are employed. Fused Deposition Modelling (FDM) is a sophisticated process that presents considerable difficulties in attaining impeccable configurations owing to the abundance of contradictory parameters that impact the quality of components and the properties of materials (Mohamed et al. 2016). Consequently, to produce composite products with optimal properties, the selection of fabrication techniques and the application of processing parameters including temperature, time, and pressure should be meticulously considered (Hartono et al. 2023).

To develop a composite that is appropriate for 3D printing and implant applications, the production of a filament composite of PLA/WA necessitates addressing several challenges. As their combination may result in enhanced mechanical properties and biocompatibility, it is initial necessity to ensure that PLA and WA are compatible (Liu et al. 2020). Further, to fulfil the demands of diverse implant applications, it is imperative to optimise the mechanical properties of the composite, including strength, flexibility, and durability (Rathod et al. 2023; Tümer & Erbil 2021). Furthermore, it is imperative to develop a PLA/WA composite filament that is FDM-compatible, thereby guaranteeing printability, accuracy, and precision (Dey et al. 2021). To guarantee the desirable properties for particular implant applications, including dental, bone graft, and orthopaedic implants, the optimal composition of the PLA/WA filament must be ascertained (Liu et al. 2020). It is possible to obtain a PLA/WA composite filament suitable for 3D printing in the field of implants by attending to the aforementioned considerations.

This study aims to establish the flow characteristics of the PLA/WA feedstock by conducting rheology studies. The objective is to assess the compatibility of the PLA/WA composite for the extrusion technique used in filament production. Additionally, the density of the extruded PLA/WA filament will be examined. This study aims to generate substantial results about the improvement of PLA/WA filament composite for its potential application in 3D printing.

METHODOLOGY

RAW MATERIALS

Figure 1(a) shows the pallet PLA medical grade was supplied by Vistec Technology Sdn. Bhd. was used as based material for feedstock preparation. Wollastonite powder with an average size of 8.7 µm was purchased from CNPC Powder Material Co., Ltd., China as shown in Figure 1(b). The density measured were 1.25 g/cm^3 and 2.99 g/cm^3 for PLA and WA respectively.

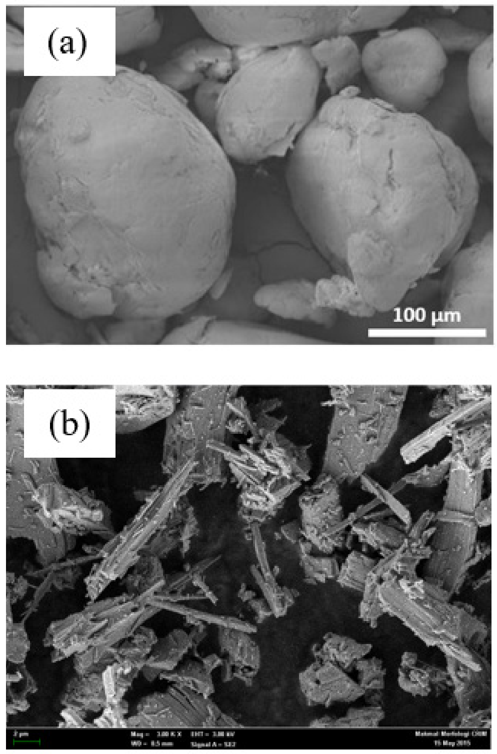

FIGURE 1. (a) SEM images of PLA pallet and, (b) WA powder
Source: Marzuki et al. (2022a)

PLA/WA FEEDSTOCK PREPARATION

Saravana & Kandaswamy (2019) identified that the best composition of biocomposite material for PLA/WA is 90 wt.% of PLA and 10 wt.% of PLA. The feedstock was prepared by using VT Sigma Blade Internal Mixer at mixing temperature of 190°C and torque at 40 rpm. The PLA/WA feedstock then crushed into small particle pallets using crusher machine.

RHEOLOGICAL TESTING

A rheological analysis was conducted utilizing a RH2000 Capillary Rheometer equipped with a 1 mm diameter capillary in order to ascertain the flow properties of the PLA/WA feedstock. The rheology test was conducted following the standards outlined in the ASTM D1238 standard. The flow parameters of the input material were examined by examining viscosity and shear rate measurements. The experiment was conducted at three distinct temperatures: 160°C, 170°C, and 180°C. The flowability of a material can be assessed by employing the Power-Law equation shown in Equation 3.2, which calculates the index value representing the flow behaviour. This enables one to ascertain the material's flowability.

$$\eta = K\gamma^{n-1} \tag{1}$$

where *E* is a viscosity (Pa.s), *K* is a consistence index, is a shear rate (s^{-1}). and *n* is Power Law exponent.

The viscosity and shear rate values can be established by utilising the findings of this test. Consequently, the activation energy, represented by E, can also be computed. The value of E can be calculated using Equation (2), and subsequently converted into logarithmic form using Equation (3) as follows:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right) \tag{2}$$

$$\ln \eta = \ln \eta_o + [\left(\frac{E}{R}\right)\left(\frac{1}{T}\right)] \tag{3}$$

where, *E* is the activation energy value (kJ/mol). *R* is the value of the gas constant (8.314 J/mol K), and *T* is the temperature in Kelvin units.

FILAMENT EXTRUSION

The filament was extruded using a 3devo Filament Make Precision - an advanced extruder (Atoomweg, The Netherlands). This equipment is equipped with a hopper that supplies the pellet extruder with the specific material using a worm mechanism which is spun via a motor attached to the worm mechanism. Prior to utilization, the pellets undergo a drying process in an oven at a temperature of 50 °C for a duration of 5 hours. The pellets traverse four heating zones characterized by temperatures set on the machine via the "parameter" menu which gradually exceeding the polymer's melting point. Subsequently, the substance in a thick and sticky form exits the filter via a nozzle and is propelled by two bearings that elongate the substance to assist in achieving a constant filament diameter. This elongation process, facilitated by a thickness control mechanism, effectively controls the diameter of the filament. The filament is then passed on to winder which spools it into a complete spool ready to be used for 3D printing. The thickness and is selected in the "material configuration" menu accessible on the extruder and have been set to 1.75 mm.

Table 1 displays the parameters configured on the extruder machine, including temperature (in 4 heating zones), screw speed, fan speed, filament diameter, and spool speed. Once the filament has reached a consistent thickness within a tolerance of ±0.10 mm, it is then placed onto the coil and the spooling process begins. Once the filament is produced, it is stored in a desiccator at room temperature until it is required.

TABLE 1. Extrusion parameters

Parameters	Setting
Screw Speed	3-5 rpm
Temperature Zone 1 (T1)	175°C
Temperature Zone 2 (T2)	190°C
Temperature Zone 3 (T3)	180 °C
Temperature Zone 4 (T4)	160 °C
Fan Speed	30%
Filament Diameter	1.75mm
Spool Speed	40 – 60 mm/min

The optimisation parameters for filament extrusion were determined using Taguchi's approach, specifically the L9 orthogonal array. This array consisted of 4 components, each having 3 levels, as indicated in Table 2. The extruded filament will be optimised according to the density measurement. The temperature of Zone 3 (T3) was maintained at a constant 180 °C, whereas Zone 4 (T4) was kept at a constant 160 °C in order to melt the feedstock from the beginning.

The optimised extruded filament is subsequently subjected to a tensile bar form printing test, following the ASTM D638 standard, utilising the 3D printer model Original Prusa i3 MK3S+. This test aims to determine the printability of the filament. In future research, the mechanical characteristics and biocompatibility of the printed component will be examined to determine the suitability of PLA/WA printed composites for usage as implant materials.

TABLE 2. L9 orthogonal array

T1 (°C) - A	T2 (°C) - B	Screw Speed (rpm) - C	Spool speed (mm/min) - D
190	170	3	40
190	180	4	50
190	190	5	60
200	170	4	60
200	180	5	40
200	190	3	50
210	170	5	50
210	180	3	60
210	190	4	40

RESULTS AND DISCUSSION

RHEOLOGICAL PROPERTIES OF PLA/WA FEEDSTOCK

The graph in Figure 2 depicts the relationship between temperature, viscosity, and shear rate for PLA/WA feedstock. Based on the rheological data, it was observed that the viscosity of each powder loading reduces as the shear rate increases. The powder loading flow characteristics can be accurately defined as pseudoplastic. Moreover, this illustrates that both PLA and WA powders will possess the ability to effectively enable smooth movement in the next filament extrusion procedure. This flow characteristic was previously utilised to demonstrate the suitability of the feedstock for injection moulding (Marzuki et al. 2022b). Moreover, this rheological data can also serve as evidence to demonstrate that the filament is capable of being extruded in the extrusion process (Khalid et al. 2023). German & Bose (1997) state that the suitable range for the shear rate in the PIM process is between 100 s^{-1} and 100,000 s^{-1}, with a viscosity value below 1000 Pa.s. When the viscosity and shear rate of a feedstock fall within the specified range, it facilitates the injection process in injection moulding and mitigates the occurrence of errors. In this study, the minimum shear rate observed was 20 s^{-1}, whereas the maximum shear rate recorded was 6382.52 s^{-1}. The viscosity parameters were evaluated, and the highest value obtained was 333.06 Pa.s, which falls below the acceptable threshold. Consequently, the PLA/WA feedstock meets the specified range and may be used in the filament extrusion process.

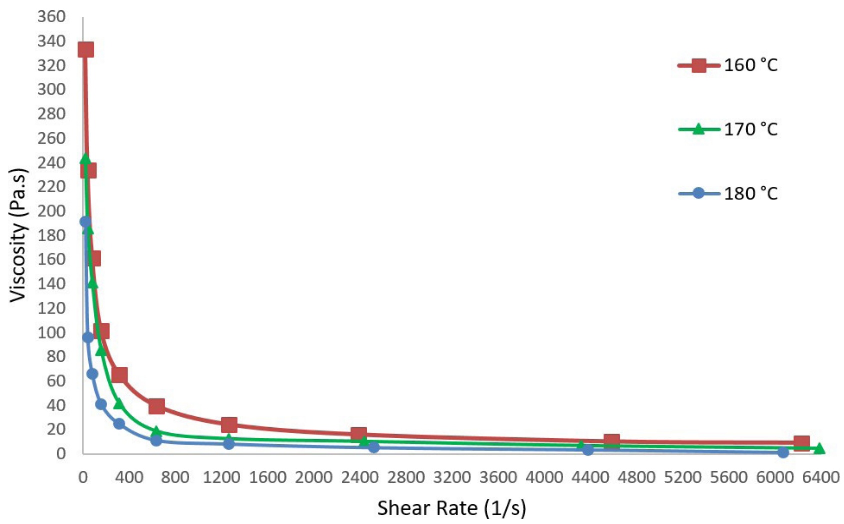

FIGURE 2. Graph of viscosity vs shear rate for PLA/WA feedstock at three different temperatures

The obtained flow behaviour index value can be located in Table 3. The flow behaviour indices determined at temperatures of 160 °C, 170 °C, and 180 °C were 0.87, 0.79, and 0.49 correspondingly. The flow behaviour index for these temperatures is less than 1 ($n<1$), indicating that the feedstock flow is pseudoplastic and hence acceptable for later filament extrusion. This also demonstrates that the feedstock does not undergo powder-polymer separation. The interplay between the powder particles of WA and the molten polymer of PLA affects the rheological characteristics of the feedstock when subjected to shear. As the temperature increases, the powder particles become more mobile, resulting in a decrease in the value of the flow behaviour index (Basir et al. 2020).

TABLE 3. Flow behaviour index, n for three temperatures

Temperature (°C)	Flow Behaviour Index, n
160	0.87
170	0.79
180	0.49

The activation energy value is the measure of a feedstock's susceptibility to changes in temperature and pressure. A low activation energy value is crucial and indispensable in the filament extrusion as it guarantees that the viscosity of the feed material remains relatively stable despite fluctuations in temperature during the extrusion process with different temperatures applied.

The determination of the activation energy, denoted as E, can be achieved by utilising the Arrhenius formula, as seen in Equation (2). In order to determine the value of E, this formula necessitates multiplying the slope of the graph by R. Figure 3 depicts the correlation between the viscosity of the input substance and the temperature under a shear rate of 1000 s^{-1}. The activation energy for the ratio of 90:10 wt.% was determined to be 41.57 kJ/mol. In comparison to Marzuki et al.'s (2022) investigation on PLA/Hydroxyapatite (HA), which reported an E value of 49.54 kJ/mol, the activation energy value achieved in this study was notably lower. This indicates that the activation energy value was attainable, and it would result in reduced sensitivity of the feedstock to temperature, hence minimising the occurrence of stress concentration, fracture, and flaws during filament extrusion process.

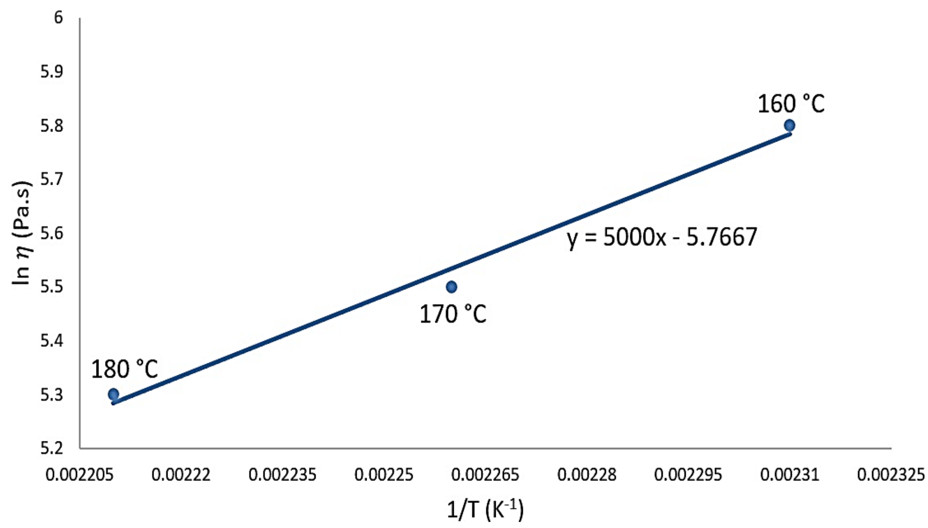

FIGURE 3. Graph of viscosity against temperature for the PLA/WA feedstock

PLA/WA FILAMENT EXTRUSION

An optimisation analysis was performed on the process parameters of filament extrusion utilising PLA/WA feedstock at a weight ratio of 90:10 wt.%. The Taguchi L9 design approach was utilised for this analysis. This approach utilises parameter optimisation that relies on the density properties of extruded filament, which are determined using the Archimedes principle (Patel et al. 2024). To achieve the ideal density, the signal to noise ratio (S/N) employed follows the "larger is better" approach. The calculation of the signal-to-noise ratio (S/N ratio) is derived from Equation (4):

$$S/N = -10 \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Yi^2}\right) \tag{4}$$

Where n is the total number of observations, Yi is the measurement of response test results in each run. The parameters used in this study are as in Table 2. The results of the density of green bodies obtained through the Taguchi L9 method are shown in Table 4. The average maximum density value obtained is on the sixth run which is 1.327 g/cm^3.

In the sixth trial, T1 was heated to a temperature of 200 °C, while T2 was heated to a temperature of 190 °C, which proved sufficient to cause the melting of PLA. The temperature applied to this feedstock surpasses the melting point of PLA. Furthermore, the maximum density achieved at L6 can be attributed to the screw speed of 3 rpm, which is the lowest speed, as well as the average spool speed of 50 mm/min. The reduced rotational velocity of the screw enables the feedstock to undergo complete melting prior to its extrusion into filament form. The pace at which the spool rotates has an impact on filaments with a consistent diameter. Insufficient roller speed might lead to filament diameter enlargement. Conversely, an excessively high roller speed can result in a reduction in filament diameter (Darsin et al. 2023). Overall, the findings of density values for all runs exhibit little discrepancies. However, to get the most favourable parameters, it is necessary to conduct a comprehensive comparison of the entire experimental process on the mechanical characteristics such as tensile test of the filament. This will allow us to observe the impact of the interplay between the established parameters.

TABLE 4. L9 orthogonal array for density of feedstock

Ex[eriment	Parameters				Density (g/cm^3)			S/N (dB)
	A	B	C	D	Repitation experiment 1	Repitation experiment 2	Repitation experiment 3	
1	1	1	1	1	1.319	1.32	1.326	2.422
2	1	2	2	2	1.32	1.312	1.327	2.409
3	1	3	3	3	1.312	1.315	1.338	2.421
4	2	1	2	3	1.309	1.306	1.32	2.356
5	2	2	3	1	1.324	1.327	1.32	2.436
6	2	3	1	2	1.329	1.328	1.323	2.455
7	3	1	3	2	1.322	1.325	1.323	2.433
8	3	2	1	3	1.32	1.319	1.319	2.407
9	3	3	2	1	1.325	1.328	1.324	2.449
							Σ	**21.789**
							T	2.421

Figure 4 shows the curve illustrating the signal-to-noise ratio response of PLA/WA filament density. The obtained settings for the filament extrusion process are optimal. The temperature of Zone 1 (T1) is set at 210 °C, while the temperature of Zone 2 (T2) is set at 190 °C. The screw speed is set at 5 rpm, and the spool speed is set at 40 mm/min. The analysis of the graph's slope revealed that the spool speed factor has the most significant impact on the extrusion process of PLA/WA feedstock.

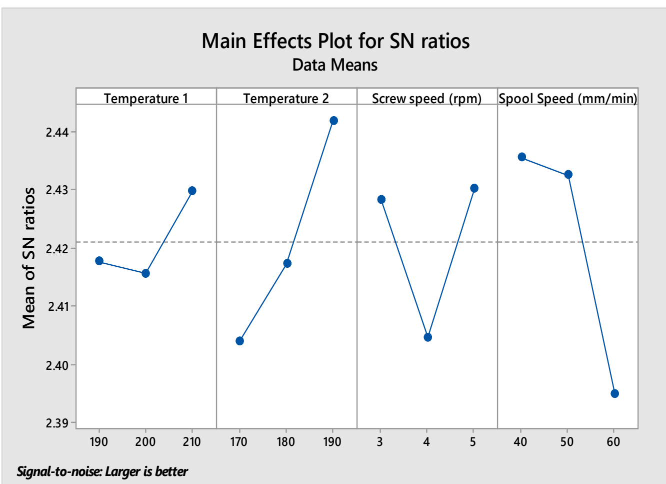

FIGURE 4. S/N ratio for green PLA/WA feedstock

There is a need for a more thorough and extensive optimization procedure to study the impact of temperature fluctuations, screw speed, and spool speed on density qualities and mechanical properties. Figure 5 shows a tensile bar shape successfully printed using PLA/WA filament at nozzle temperate of 210 °C, printer bed temperature of 70 °C and fan speed of 100%. The infill for printing was set to 100%. This demonstrates that the filament is appropriate for printing without defects such as problems with bed adhesion and warping.

This experimental research examines effective techniques for enhancing the adhesive properties of a 3D model to the hot bed surface, as well as approaches for reducing internal stress within the 3D model to avoid warping in an FDM process (Singh 2018). A detailed study of the printed sample will be reported in a future study.

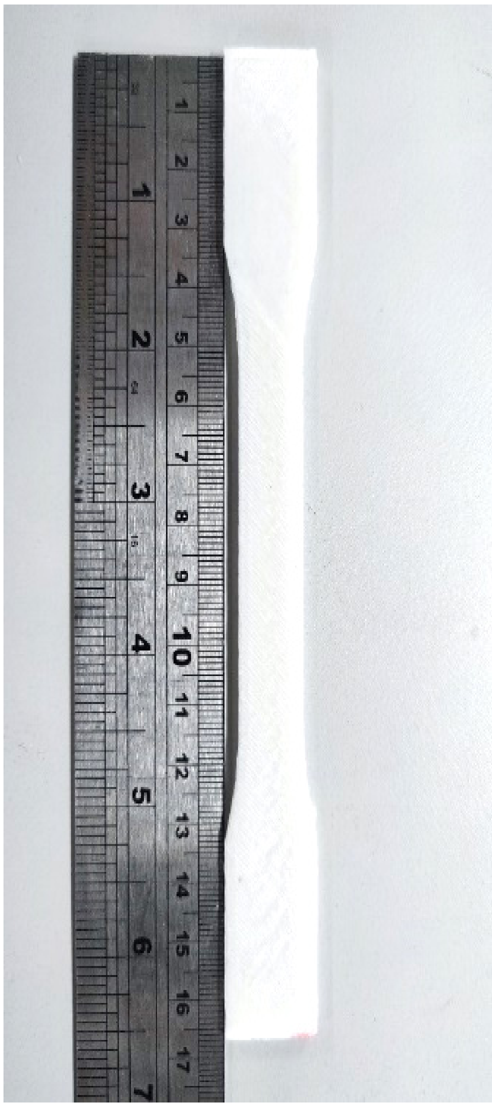

FIGURE 5. 3D printed of ASTM D638 tensile bar shape PLA/WA composite

CONCLUSION

The objective of this study is to develop PLA/WA filament using the extrusion process for application in field of 3D printing, which is now in its preliminary research phase. A feedstock comprising 90 wt.% of PLA and 10 wt.% of WA was successfully manufactured. The feedstock exhibits pseudoplastic flow behaviour, characterised by a reduction in viscosity as the shear rate increases. The viscosity value obtained is 333.06 Pa.s, with the shear rate ranging from 20 s^{-1} to 6382.52 s^{-1}. The flowability index, measured at all temperatures, consistently demonstrates a value below 1 (n<1), indicating that the feedstock is indeed appropriate for use in the filament extrusion process. The feedstock has modest sensitivity to temperature fluctuations, as indicated by the lower activation energy value of 49.54 kJ/mol. PLA/WA filament has been successfully created by the extrusion process, with optimal parameters determined. Specifically, the temperature of Zone 1 (T1) at 210 °C, while the temperature of Zone 2 (T2) at 190 °C. The rotational speed of the screw is configured to 5 rpm, while the spool speed is set to 40 mm/min. To summarise, the results of this study suggest that PLA/WA filament composites can be manufactured using the extrusion method. However, further investigation, specifically into the mechanical properties of PLA/WA filament, is needed to reinforce these initial findings.

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DECLARATION OF COMPETING INTEREST

None.

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