

Fabrication and Characterization of Almond Skin Powder Reinforced PLA Based Composite Filaments for Fused Filament Fabrication

Ravi Kumar ¹, Kapil Chawla ², Jaspreet Singh ^{3,*}

Abstract

Researchers around the globe are increasingly utilizing recycling methods to address waste generated from polymers, spurred by the dire need to deal with environmental challenges promoting sustainable practices. Polymers are having highly-stable long-chain polymer structure which makes it difficult to degrade easily posing many environmental and health challenges, hence, recycling of the polymers is the sole method to mitigate harmful effects. However, there has been relatively little focus on producing fused filaments using secondary recycled Polylactic acid (2° PLA) reinforced with industrial waste of almond skin. The goal of this study is to fabricate fused filaments for fused filament fabrication (FFF) in additive manufacturing. The process involves controlling parameters such as torque (0.1 Nm, 0.2 Nm, 0.3 Nm), load (7.5 kg, 10 kg, 12.5 kg), temperature (175°C, 185°C, 195°C) of a twin-screw extruder using 2° PLA reinforced with almond skin. Taguchi's orthogonal array has been applied to prepare composite filaments. Total 9-composite filaments have been prepared with a composition of 94% 2° PLA and 6% almond skin by %wt. The highest peak strength (43.85 MPa) of the 2° PLA + almond skin composite filament was achieved at 0.1 Nm torque, 12.5 kg load and 195°C extrusion temperature. The morphological and thermal characteristics of the fused filaments were examined through scanning electron microscopy and differential scanning calorimetry. Further, porosity analysis indicates that lower porosity percentages in the filaments lead to improved mechanical properties.

Keywords: Polylactic acid, almond skin, twin screw extruder (TSE), scanning electron microscopy (SEM), differential scanning calorimetry (DSC)

INTRODUCTION

Polymers has become indispensable in modern life for its ease of shaping, ability to create intricate designs, and affordability. Polymer finds extensive use in a wide range of applications including household items, packaging, container for fluids, shopping bags, industrial goods, automobile interiors,

aircraft components, and many more. Despite its versatility and widespread utility, the global community grapples with the significant challenge of managing polymer solid waste effectively [1]. Indeed, global production of solid waste has exceeded 150 million tons annually and continues to grow rapidly. Due to their long chain polymer structure, polymers are highly stable and degrade over hundreds of years under natural conditions, posing significant environmental challenges [2]. Traditional disposal methods like landfilling generate methane gas, which contributes to global warming 21-times higher than carbon dioxide. Therefore, recycled polymer solid waste is seen as the sole viable solution to mitigate detrimental environmental effects [3].

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In recent decades, there has been a notable rise in the demand for products featuring improved characteristics. This heightened demand and competitive pressure have compelled companies to introduce new or innovatively designed products within short span of time [4]. However, conventional manufacturing methods often cannot meet these requirements due to the extensive time needed for design and production activities [5-6]. Also many researchers now a days doing extensive research on utilizing waste from industries like acrylonitrile butadiene styrene (ABS), polycarbonate, PLA, polyamide (PA) as raw materials. These materials are used either alone or with reinforcement from other substances to enhance different properties of components printed using 3D manufacturing technology [7-8]. Zhang et al., focused on investigating mechanical performance of composites made from cellulose nanofiber (CNF) reinforced with polylactic acid (PLA). It was found that tensile modulus of the composite increased by 18.4% compared to pure PLA, whereas strength of prepared composite has been decreased by 7.4% due to inadequate bonding between CNF and PLA. [9]. Santos et al. investigated the mechanical properties of polymer composites manufactured via fused filament fabrication (FFF). The composites were prepared using PLA filament as the matrix material and vegetable yarns such as jute, ramie, and sisal as reinforcements, which were printed simultaneously. The authors observed 28.9% increase in stiffness and 28.6% increase in strength compared to pure PLA matrix materials [10]. Cao et al. determined the impact of reinforcing 30 vol% carbon fiber (CF) on the tensile properties of PLA parts manufactured via FDM. Their study revealed that reinforcing with CF led to decrease in tensile strength of prepared PLA specimens from 54.51 MPa to 49.41 MPa. However, both Young's modulus and elongation-at-break improved from 1.04 GPa to 1.26 GPa, and 6.26% to 7.81% as compared to non-reinforced PLA specimen [11]. Zhao et al. investigated the tensile properties of pure PLA, short glass fiber (SGF)/PLA composites, and carbon nanotube (CNT)-SGF/PLA composites processed via FDM 3D printing. According to their findings, the composites containing SGF/PLA and CNT-SGF/PLA shows higher young's modulus and tensile strength due to improved interface adhesion. The authors also noted that varying FDM raster angles did not significantly influence the tensile properties of the samples [12]. Some researchers found that thermal conductivity of a poly vinyl chloride was enhanced through the addition of corn straw and wood fibers, which facilitated effective interlock bonding between the particles [13]. Ryder et al. investigated mechanical and physical properties of ABS reinforced with (10,15,23) percent by weight of Stainless steel 420 powder. The authors developed new methodology to fabricate composite by chemical mixing of ABS and SS in acetone solution. They concluded that mechanical properties of fabricated filaments has been enhanced with addition of reinforced material to base matrix [14]. Makannavar et al. has carried out the study to examine the tensile properties of graphite reinforced Acrylonitrile Butadiene Styrene (ABS) composites filament fabricated by twin screw extrusion for 3D printing. Authors observed that filaments prepared via reinforcing graphite in ABS exhibit superior properties as compared non reinforced fused filament. [15]. Kumar et al. developed composite by reinforcing Multi- Walled- CNT in High Density Polyethylene (HDPE) for investigating their different mechanical properties. They also developed different composites by varying different weight proportions i.e. 0.5, 1, 3, and 5 by %wt. They observed that with increase in weight % of reinforcement both tensile and flexural properties has been improved significantly [16]. The mechanical and thermal properties of ABS composites matrix showed improvement when reinforced with fine powder of iron compared to coarse iron powder [17]. Kartal et al. developed PLA-based composite filaments by incorporating waste beach sawdust (WBS) using a single screw extruder to examine various mechanical properties, including flexural, tensile, and impact strength. The WBS was added at different weight percentages—5%, 10%, 15%, and 20%—to the PLA matrix for filament fabrication. Tensile testing was performed using a universal testing machine. The authors found that when the WBS content increased from 0% to 5%, the tensile strength decreased slightly from 56.03 MPa to 52.78 MPa. However, with further increases in WBS percentage, the tensile strength dropped more significantly [18]. Rodriguez-Pacheco et al. developed filaments for FDM by reinforcing zinc carbonate basic (ZCB) in PLA matrix via extrusion. Author has used ZCB in various weight fractions like 10%, 20%, 30%, 40% and 50% to reinforce in PLA matrix. Author observed nozzle clogging at different weight %ages except 10% ZCB. The 10% ratio was chosen as it represents the highest percentage of ZCB that could be extruded [19].

Wang et al. has prepared PLA composite filaments by reinforcing carbon fiber (CF) in ratio of 100 wt% PLA, 5 wt% CF and 95 wt% PLA, 10 wt% CF and 90 wt% PLA using twin screw extruder for use in 3D printing. Author investigated mechanical properties such as impact strength, bending strength, tensile strength and fracture elongation which were measured as 3.27 kJ/m², 65.7 MPa, 56.06 MPa and 6.16% respectively which was found to be declining upon further increase in CF content to 10 wt% [20]. Shubmugam et al. has fabricated PLA based composite filaments by reinforcing 25 wt.% orange peel powder (OPP) using twin screw extruder at 160°C temperature for use in 3D printing. Author examined thermal properties of the composite filaments and found that adding OPP in PLA matrix increases thermal stability at higher temperatures [21]. Kalimuthu et al. has created composite filaments using PLA matrix by reinforcing 5% and 10% loading of tamarind shell powder (TSP) using twin screw extruder at 190°C temperature, 40mm/s speed and 1.74mm diameter. Author has observed that tensile strength of PLA composite filaments by reinforcing 10 wt% TSP was higher than tensile strength of 5 wt% reinforcement of TSP. Further, author concluded that PLA/TSP composites may be used in biomedical application [22]. From the literature review, it has been figured out that many investigations were carried out on thermal & mechanical properties of polymers with reinforcement of carbon fiber, CNF, wood fibers, almond flour, kenaf fiber, glass fiber, MWCNT, sisal fiber, jute, TiN and SS but hitherto very less has been explored on printing of recycled thermoplastic composites using industrial waste as a reinforcement in different wt%. Many researchers have used virgin PLA as a matrix material with reinforcement of CNTs, graphene, jute, glass fiber, graphite & metallic powders but so far very less work has been done on 2° PLA. In the present research work, 2° PLA will be used as base material and almond skin as reinforcement to fabricate fused filaments using TSE. As prepared composite filament will be used for 3D printing of car dashboards, car bumpers, and car exterior panels. With the addition of different reinforcement in base matrix mechanical, rheological, and morphological properties has been improved significantly.

MATERIALS AND METHODOLOGY

In the current research, 2° PLA procured from local market area (Batra Polymers, Ludhiana, Punjab, India) was chosen as base material to fabricate fused filaments. Almond skin was selected as reinforcement materials to prepare fused filaments. The waste almond skin was taken from almond oil manufacturing industry and placed in oven at 50°C to dry for 5 hrs. in order to remove moisture. After collecting reinforcement and base material, MFR has been calculated by varying different weight proportions of almond skin from 0% wt. to 9% wt. Further, TSE has been used in order to prepared fused filament by varying different parameters such as torque, temperature and load which were finalized entirely based upon the pilot experimentation according to Taguchi's L9 orthogonal array as shown in Table 1. After preparing filaments, mechanical properties like strength, toughness, modulus of elasticity have been examined using UTM with load capacity of 500kgf. Analysis of variance (ANOVA) was applied using Minitab as ANOVA results highlights the most significant parameters and their %age contributions which affects the PS of the fused filaments. Further DSC and SEM has been done on prepared filaments in order to determine their thermal and morphological properties. Figure 1 showing flow process for the fabrication and testing of fused filament.

RESULTS AND DISCUSSIONS

Melt flow rate (MFR) depicts flow rate of any given thermoplastic polymer usually measured in gm/10min which assures the smooth flow of thermoplastic polymers. The results of melt flow rate performed according to ASTM D 1238 standard with varying percentage of reinforcement materials were shown in Table 2. Initially MFI of 2° PLA was 33.85 gm/10min (average of three repetitions). Then MFR of all reinforcements were taken according to ASTM D1238 standard and different observations mentioned in Table 2 given below.

From MFR observations, it was found that with rise in reinforcement %age from 3% wt. to 9% wt., MFR starts decreasing. It has been found that with rise in reinforcement %age, most of the heat was absorbed by reinforcing material instead of base matrix which resists the flow from orifice. Also it has been found that on further increasing reinforcement %age beyond 9% wt. lead to nozzle clogging [23]. The MFI of 2° PLA is nearby equal to MFI of 2° PLA + 6% almond skin, therefore, in order of effective utilization of industrial waste 6% wt. proportion has been selected.

Next step was to prepare fused filaments by applying L9 (O. A) on TSE in order to avoid repetition as shown in Table 1 for determining various mechanical and thermal properties. Total 9 filaments have been prepared by reinforcing 6% wt. Almond skin in 2° PLA by varying different parameters such as torque, load and temperature of TSE. To investigate various mechanical properties such as peak strength (PS), peak elongation, modulus of elasticity and modulus of toughness of fused filaments, tensile testing was carried out on UTM and Stress-Strain diagram has been drawn based on different observations as shown in Figure 2. During the analysis of stress-strain diagram, no negative strain was found which ensures that there was slippage between jaws of UTM while testing.

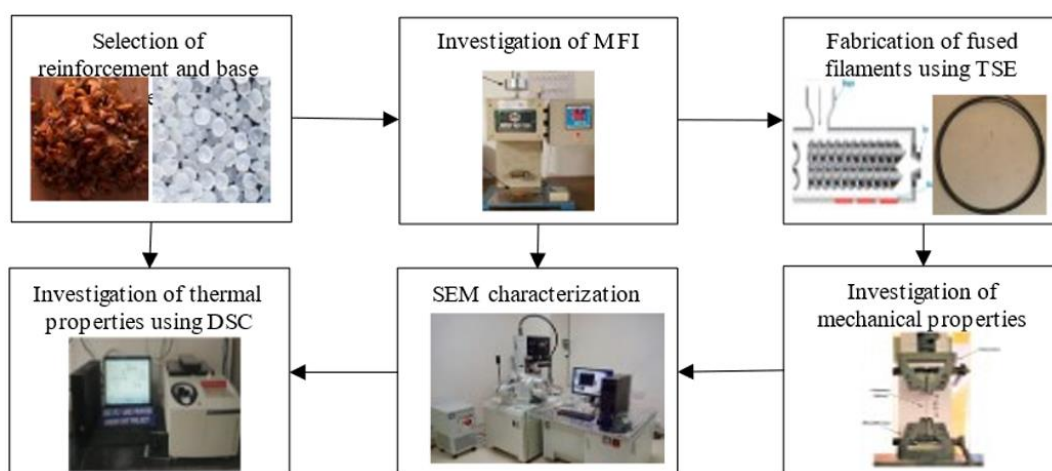


Figure 1. Methodology employed in the present study.

Table 1. Control log to prepare fused filaments.

Sample No.	Torque (Nm)	Load (Kg)	Temperature (°C)
1	0.1	7.5	175
2	0.1	10	185
3	0.1	12.5	195
4	0.2	7.5	185
5	0.2	10	195
6	0.2	12.5	175
7	0.3	7.5	195
8	0.3	10	175
9	0.3	12.5	185

Table 2. MFR of 2° PLA and Almond Skin in Varying Proportions.

S N	Specimen	MFR (gm/10min)			Average MFI (gm/10min)
1	2° PLA	33.75	33.85	33.95	33.85
2	2° PLA + 3%wt. almond skin	29.4	30.7	31.1	30.40

3	2° PLA + 6%wt. almond skin	22.2	24.5	23.8	23.50
4	2° PLA + 9%wt. almond skin	14.9	17	16.1	16

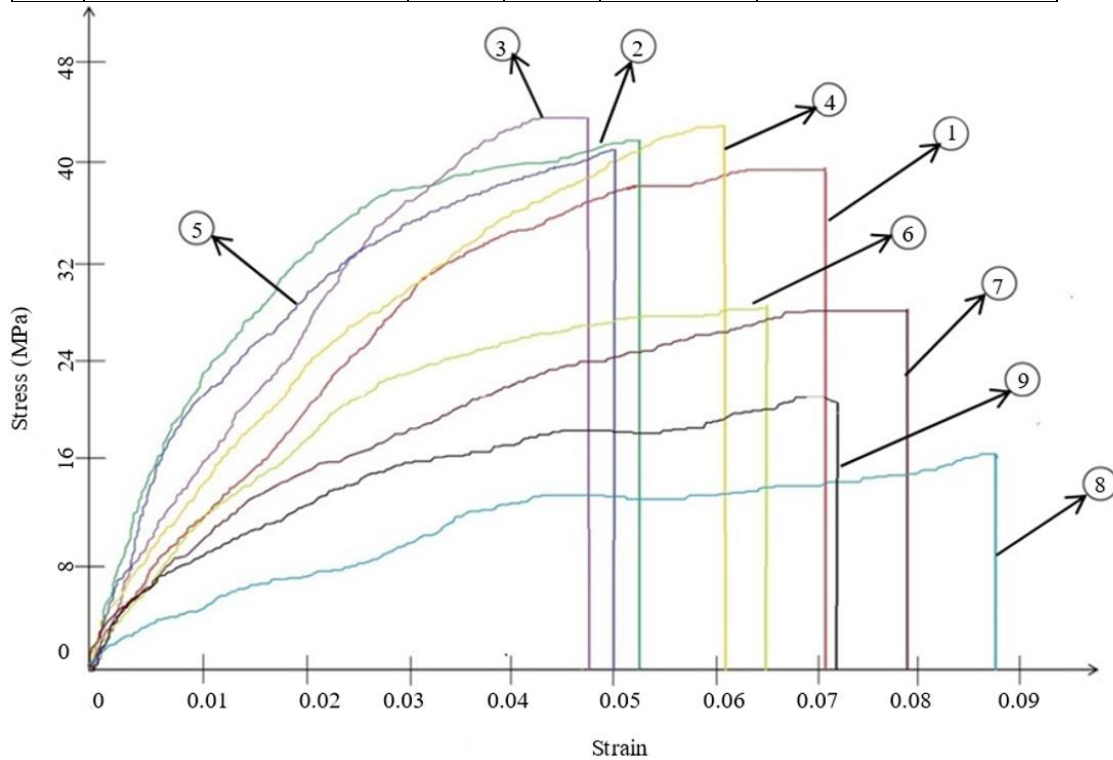


Figure 2. Stress-strain diagram.

Tensile testing of the fused filaments has been carried out on UTM and the results obtained have been shown in Table 3. It has been observed from the Table 3 that, at low torque (0.1 Nm) and high temperature (195 °C) conditions, sample 3 has highest peak strength (43.85 Mpa) amongst all the fabricated filaments which may be due to strong intermolecular molecular bond between reinforcement and base matrix whereas at high torque (0.3 Nm) and least temperature (175 °C) conditions, sample 8 possesses the minimum peak strength (16.82 Mpa). Further from Table 1 and Table 3, it was observed that upon increasing torque (Nm), peak strength is decreasing for all the specimens because of improper bonding between reinforcement material and the matrix material at higher load (kg) conditions. Whereas at low torque conditions, composite material gets sufficient time for proper bonding between reinforcement and matrix material which further helps in exhibiting ductile behaviour of the fused filaments.

Further signal-to-noise ratio (S/N) was investigated via Minitab software by assuming larger is better approach & mentioned in the Table 4 given below. Based on mean S/N ratio values, mean effect plots for PS for fused filaments have been obtained using Minitab to investigate effect of individual parameters such as torque, load & temperature on the PS of fused filaments shown in Fig. 3. From Fig. 3, it has been found that PS enhanced with rise in temperature from 175°C to 195°C. Further, with an increase in torque from 0.1 Nm to 0.3 Nm, PS decreased sharply and PS decreases upon increasing load from 7.5 kg to 12.5 kg. Parametric conditions for maximum PS for almond skin reinforced composite were 0.1 Nm torque, 7.5 kg load and 195°C temperature.

Further, to find most dominant parameter affecting PS, analysis of variance (ANOVA) was used to find S/N ratios and results obtained are mentioned in the Table 5. The results of ANOVA shows that temperature and torque both have remarkable effect on PS as P-values were very close or less than 0.05.

As PS were important mechanical characteristics of the almond skin reinforced composite filament, %age contribution using S/N ratios of the factors by applying ANOVA were also determined as shown in Table 5.

Table 3. Mechanical Properties of the Fused Filaments.

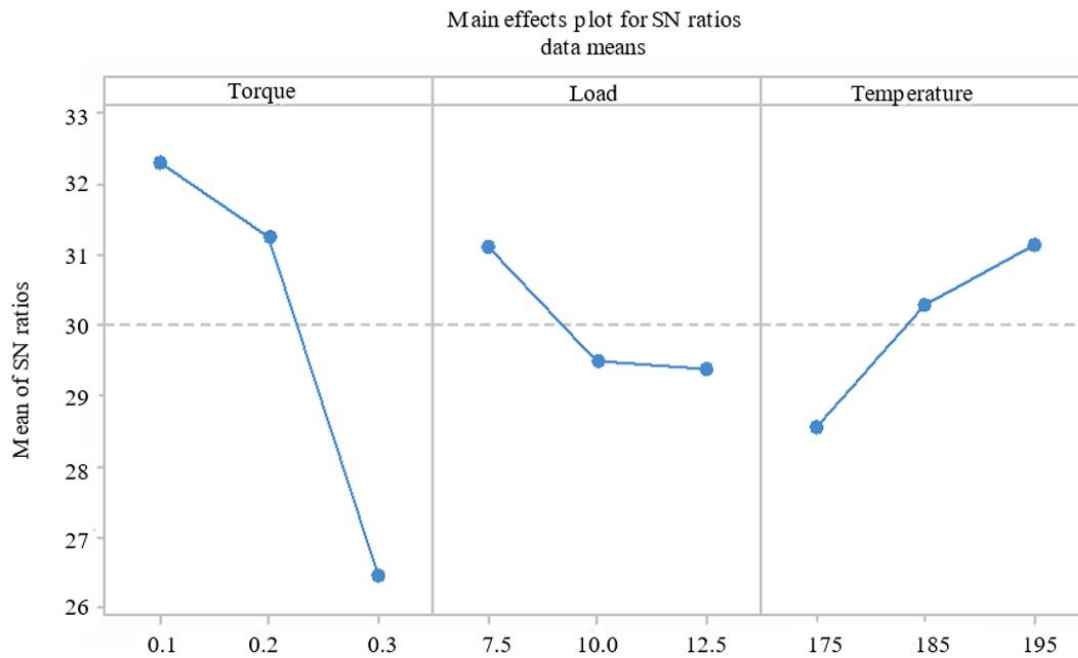
Sample no.	Peak strength (Mpa)	Modulus of elasticity (Gpa)	Modulus of toughness (Mpa)
1	39.82	0.6423	1.2248
2	40.36	0.9138	1.0181
3	43.85	0.6464	0.9333
4	43.1	0.8081	1.2814
5	39.33	0.8740	0.9280
6	28.8	0.5538	0.8132
7	27.2	0.3831	0.9298
8	16.82	0.2713	0.6491
9	20.23	0.3915	0.6853

Table 4. S/N Ratio for PS.

Sample. no.	1	2	3	4	5	6	7	8	9
S/N ratio for PS (dB)	32.00	32.12	32.84	32.69	31.89	29.19	28.69	24.52	26.12

Table 5. ANOVA for PS of Fused Filaments.

Source	DOF	Adj SS	Adj MS	F-value	P-value	%age Contribution
Temperature	2	51.818	51.818	30.26	0.003	69.203
Load	2	4.569	4.569	2.67	0.163	6.10
Torque	2	9.930	9.930	5.80	0.061	13.26
Error	2	8.561	1.712			11.43
Total	8	74.878				100



Signal-to-noise: Larger is better

Figure 3. Mean S/N Ratio Plot for PS.

SEM analysis has been performed for characterization of the prepared filaments as shown in Figure 4 and Figure 5. It has been found that filament fabricated at high torque 0.3Nm, temperature 175°C and load 10kg depicts poor mechanical and thermal properties due the presence of voids as shown in Fig. 5. Further, porosity analysis of sample as shown in Figure 6 having maximum PS (sample 3) and minimum PS (sample 8) at 100X zoom via toolmaker software according to ASTM B267 standard, it has been found that sample 3 has lower porosity i.e. 6.1% as compared to sample 8 which was 34%. This shows that less porosity %age is responsible for better mechanical strength.

In the present study, DSC set-up was utilized to investigate exothermic and endothermic reactions. For the analysis of thermal stability of selection compositions according to Table 1, air flow rate of 50ml/min with exothermic and endothermic reactions at -10°C/min and 10°C/min respectively has been retained. Thermal analysis as shown in Fig. 7 depicts that with change in percentage of reinforcement from 0wt% to 3wt%, glass transition temperature was decreased from 58.31 °C to 57.52 °C while upon further increase in %wt. of almond skin to 6%wt., glass transition temperature (T_g) rises to 59.72 °C during endothermic reaction. While investigating the results of crystallinity, better result has been found in case of 3% wt. 2° PLA reinforced with almond skin composites as compared to 0%wt. & 6%wt. almond skin reinforced 2° PLA and further similar results were obtained while observing heat capacity as in case of crystallinity.

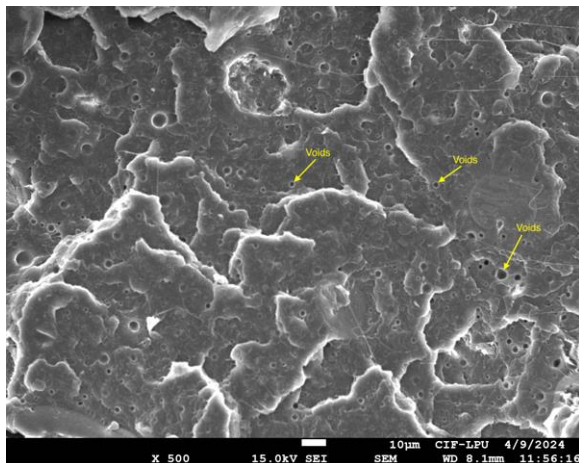


Figure 4. SEM of fused filament (at ×500) having maximum peak strength (PS)

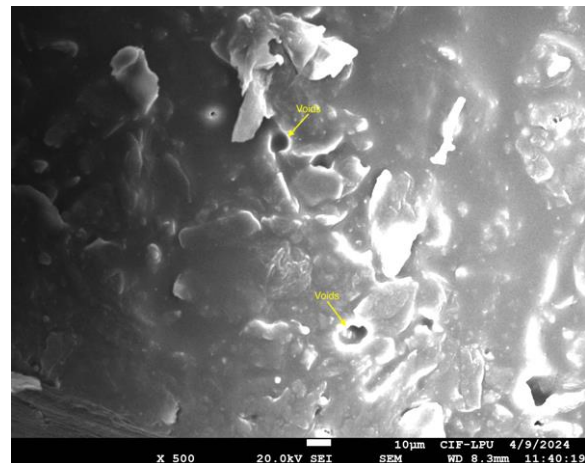


Figure 5. SEM of Fused Filament (at ×500) Having Minimum Peak Strength (PS)

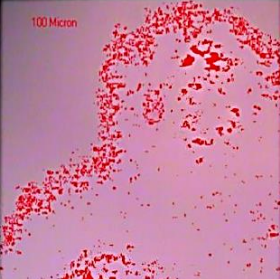
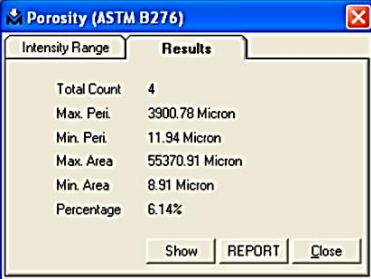
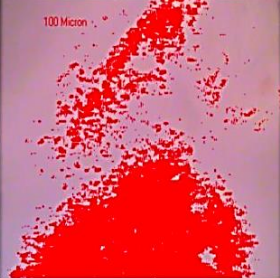
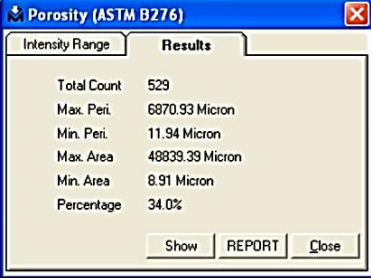
Specimen No.	Porosity	Porosity value
1.		
2.		

Figure 6. Porosity %age of sample having minimum and maximum porosity.

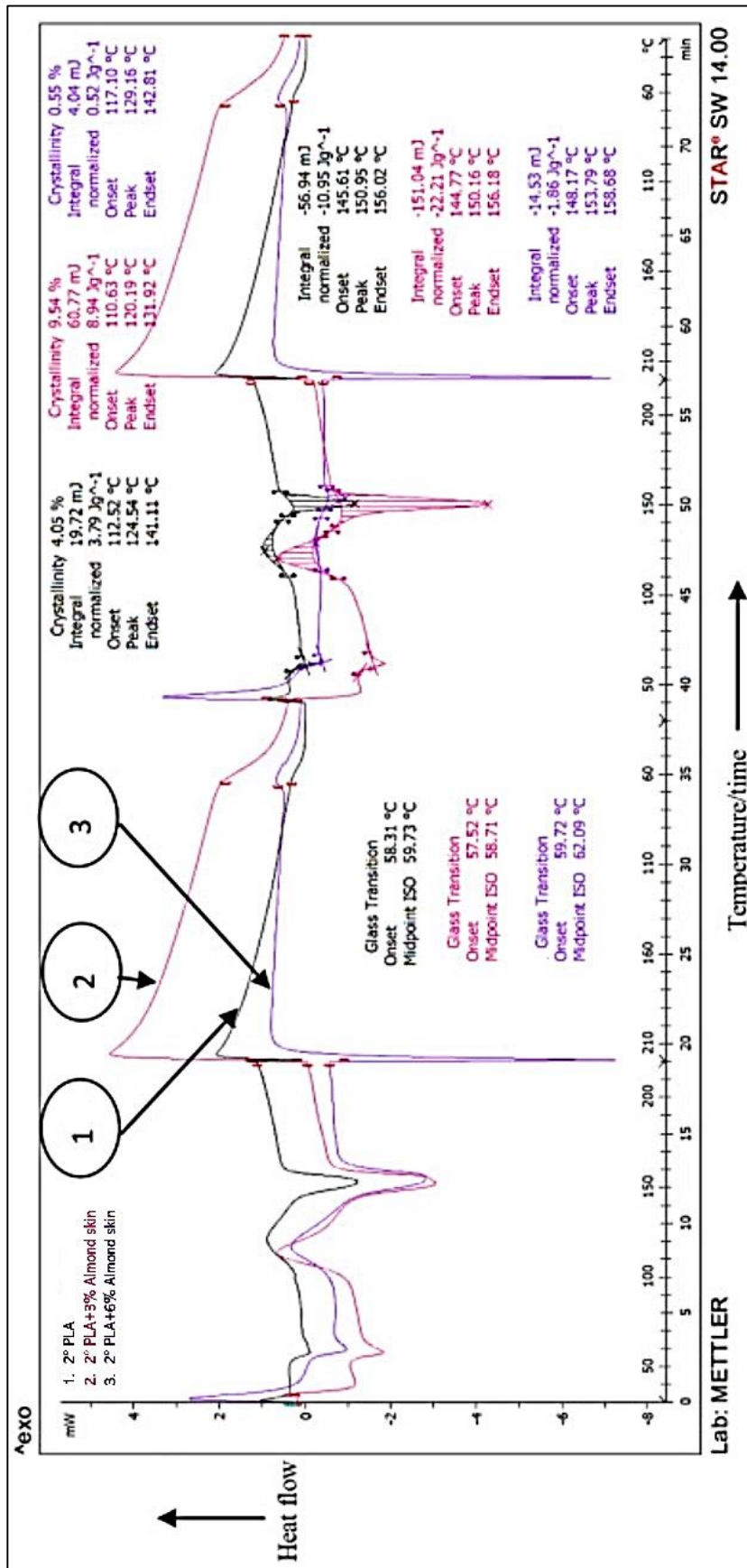


Figure 7. DSC Analysis for varying almond skin proportions

CONCLUSION

The current study has yielded the following findings:

- Rheological analysis indicates that as the reinforcement percentage of almond skin in 2°PLA increases, the melt flow index (MFI) of the filaments decreases. Furthermore, when the reinforcement exceeds 9 wt%, nozzle clogging occurs.
- Reinforcement of almond skin powder into secondary recycled PLA has successfully enhanced the mechanical properties of the filaments to achieve desired properties.
- The mechanical properties of the filaments are directly influenced by torque, external load and porosity percentage. Filaments extruded at higher temperature exhibit increased porosity, resulting in diminished mechanical properties.
- The 2° PLA+ almond skin powder composite filament achieved its maximum peak strength (43.85 MPa) under conditions of 12.5kg load, 0.1Nm torque and 195°C extrusion temperature.
- Porosity analysis shows that less porosity %age in the filaments results in better mechanical properties.
- Analysis of thermal properties shows that the Glass transition temperature (T_g) of 3% wt. almond skin in 2° PLA was higher suggesting that the reinforced filament is suitable for high-heat resistance applications.

Present study aims at reinforcing only waste almond skin in recycled PLA for the investigation of various properties. Other waste industrial materials such as wood dust, thermosetting polymers, iron powder etc can also be used as reinforcement in recycled PLA matrix to carry out the comparative study. Future research should focus on identifying the feasibility of employing fabricated composite filaments in various 3D printing applications and to analyse their properties/performance.

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