

# An Investigation and Characterization of Monkeypod Tree Flower Particulates Filled PLA Composites

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## Research Article

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# Abstract

Samanea saman (SS) flower particulates were filled in Polylactic acid (PLA) composites were fabricated with different 0, 10, and 20 wt. % through the injection molding process. The elemental composition and morphology of SS PLA composites were studied through FESEM and Energy Dispersive X-ray analysis. Thermal stability of the SS PLA composites specimens was carried out through Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC). Crystal orientations studied through X-Ray Diffraction (XRD) showed the presence of the orthorhombic SS particulates. The properties of the composites were investigated such as tensile strength, compressive strength, flexural strength, and Shore D Hardness. It was found that 20 wt. % of SS filled PLA composites has a superior tensile strength of 43.76 MPa, the compression strength of 37.94 MPa, the flexural strength of 72.47 MPa, and Shore D Hardness of 80.1 SHN than pure PLA. SS particulates-filled PLA composites would be used for low-strength applications.

## 1. Introduction

The natural fibers filled composites were used in cosmetics, packaging, medical applications, and agriculture. PLA and fibers composites showed good processability through injection molding for 20–30% fiber content (Cinelli et. al 2021). PLA has been used for biodegradable products, such as planting cups, and plastic bags. The stiffness of the PLA composites was increased from 3.4 to 8.4 GPa with the incorporation of 30 wt. % flax fibers (Oksman et. al 2003). PLA composites were developed through reinforcing nanoparticles such as graphite, silica, inorganic metal, and metallic oxides exhibited improvement of the glass transition temperature, crystallinity, modulus, tensile strength, and antimicrobial property (Mulla et. al 2021).

The coconut shell powders blended in PLA composites decreased the tensile strength and the surface modification of filler using acrylic acid improved the modulus of elasticity, thermal stability, and tensile strength, of PLA/CSP biocomposites (Salma et. al 2013). The tensile modulus of unidirectional composites was significantly higher than that of the PLA (i.e., ~40%). Since the unidirectional composites were revealed tensile strength slightly higher than that of the neat matrix (Botta et. al 2015). 30% wt., HAp-HDPE contributed to the slight toxicity of the composite after interacting with the MG63 cell line. Such an extracted HAp would be useful as inexpensive ceramics are environmentally and biologically compatible materials (Balaji Ayyanar et. al 2020).

The 30 wt. % of cotton gin waste was incorporated in PLA composites was revealed a 42% increment in flexural modulus as compared with neat PLA (Bajracharya et. al 2017). The mechanical properties and microhardness of PLA composites were increased due to the high rigidity of calcined seashell inorganic bio-filler (Razali et. al 2021). Mussel shells filled PLA biocomposites revealed excellent mechanical properties, without significantly lowering the composite strength and improving the elastic modulus (Gigante et. al 2020). The direction fiber content in the composites was significantly increased moduli between 70% up to 6 times greater than the matrix. Young's modulus and highest tensile strength were

found in PLA self-reinforced composite than PBS composites (Jia et. al 2014). The PLA composites revealed higher deformation at break higher than 120% also when 20 wt. % of filler was filled in the composite. Differential Scanning Calorimetry analysis of the PLA composites revealed that microcrystalline cellulose fillers mostly affect the crystallinity of polybutylene adipate-co-terephthalate (Botta et. al 2021).

The modification of the properties of both PLA and flax fibers may also lead to an increase in energy absorption, impact strength, and thermal stability of the composites (Sanivada et. al 2020). Since there was no toxic in the specimens. A new composite has been (10:3 wt%) developed using 30 wt. % HAp filled in HDPE (Balaji Ayyanar et. al 2019). PLA was suitable for applications ranging from short-term packaging to biomedical applications (i.e. implants, sutures, drug encapsulation) (Fattori et. al 2011). *Syzygium cumini* particulates filled epoxy sandwich composites revealed better mechanical properties (Balaji Ayyanar et. Al 2021). The objectives of this work is to develop a new novel *Samanea Saman* filled PLA composites and to carry out XRD, FESEM, EDX, tensile strength, compressive strength, hardness, and flexural strength of the results of the composite were compared.

## 2. Experimentation

### 2.1 Materials

Matrix Thermoplastic polyester Polylactic acid was used as the matrix material. It was procured from Chennai, Tamil Nadu in pelletized form. It has a density of 1.24 g/cm<sup>3</sup>, a Melting point of 157 - 170°C, Tensile Strength of 61- 66 MPa, Flexural Strength of 48 – 110 MPa, and weight of 1.5 Kg. Properties of SS and its Scientific name: *Samanea saman* Family: Fabaceae and Kingdom: Plantae. Figures.1 (a-c) shows the SS fillers, PLA, and SS PLA composites specimen.

### 2.2 Fabrication of the Composites

To develop the composites specimens, a systemic approach was carried out. The methodology and process that was carried out to develop a SS particulate-filled PLA composite specimen as shown in Figure. 2 SS flowers were collected from the trees growing in and around our college campus. The stem of the flowers was cut down and only the head was used. The flowers were cleaned again and again with cold water to remove the impurities.

The cleaned flowers were dried to room temperature for 8-10 days until they dried completely. The 1 kg of dried flowers were grinded into powder at the speed of 1500 rpm through the floor mill. To fabricate the composites specimen mixtures of the required quantity 0, 10, and 20 wt. % SS particulates filled in PLA. Each weight percentage of mixtures was preheated at 100°C for about 10 min and introduced in the heating chamber for further heating. The temperature 110±5 °C was preferred in injection molding to control the melting of the matrix.

The manual operating pressure was adjusted to 60 – 65 bar during the injection process. PLA pellets and SS powder particulates were fed into the hopper. Once the material passes through the hopper it enters the injection barrel. The barrel consists of separately controlled heating zones. The semi-melted mixtures were injected into mold cavities in the die as per ASTM standards and removed immediately. By increasing the SS particulates by more than 20 wt. % in PLA leads to the burning of SS fillers during the molding process. Due to these limitations, the fillers were incorporated upto 20 wt. % in PLA which was affected the quality of composites.

### **3. Characterization Techniques**

i) XRD ii) Surface morphology and EDX analysis of SS particulates filled PLA composite were examined using a ZEISS Sigma 300 Scanning Electron Microscope iii) DSC analysis was carried out under flowing nitrogen (Nitrogen 50 ml/min). The sample with a mass of 9 mg was heated gradually from 10° C to 500° C at a rate of 20° C/ min and found the peak melting temperatures and thermal energy required for phase changes of the 20 wt. % SS PLA Composites. iv) Thermogravimetric Analysis (TGA) was carried out using a Q600 SDT (TA Instruments, USA) with a mass of 9 mg was heated gradually from the temperature of 50° C to 600° C at a constant heating rate of 20°C/min under flowing nitrogen (20 mL/ min).

v) Tensile testing was performed under ambient conditions on a universal tester at a speed of 2 mm/min according to ASTM D638 (span length 90 mm, gauge length 40 mm, thickness 3 mm, and width 7 mm) vii) The Shore D hardness testing was performed under ambient conditions on a Shore 'D' (length 30 mm, and width 10). viii) Flexural testing was performed under ambient conditions on a universal tester at a speed of 2 mm/min according to ASTM D790 (span length 80 mm, width 13 mm, thickness 3 mm). The same compositions of six samples were tested and the average values are reported.

## **4. Results And Discussion**

### **4.1 X-Ray Diffraction Analysis**

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. The locations (angles) and intensities of the diffracted X-rays are measured and given in Table.1. The XRD test results show a peak value between 10 to 25 degrees as shown in Figure 3. It confirmed the presence of AlPO-8 in SS Powder.

Table 1  
XRD Pattern of SS PLA Composites

Pos. [ $^{\circ}2\theta$ ]	Height[cts]	FWHM Left [ $^{\circ}2\theta$ ]	d-spacing [ $\text{\AA}$ ]	Rel. Int. [%]
14.9823	331.82	0.6140	5.91330	24.94
21.3081	1330.54	0.2047	4.16997	100.00
22.4861	972.68	0.8187	3.95411	73.10
24.2878	481.21	0.0768	3.66472	36.17
26.4475	88.36	0.3070	3.37015	6.64

The crystal structure was found to be in orthorhombic structure. Compound Name: AlPO-8, Chemical Formula: Al<sub>1</sub> O<sub>4</sub> P<sub>1</sub>, Mineral name: Berlinite, Crystal Structure: Orthorhombic is given in Table.2

Table 2  
Crystal Structure SS PLA Composites

Compound Name	Chem. Formula	Mineral Name	Cryst. Syst.
AlPO-8	Al <sub>1</sub> O <sub>4</sub> P <sub>1</sub>	AlPO-8	Orthorhombic

## 4.2 FESEM Analysis of Composite

Figure. 4 revealed the morphology of the 20 wt. % SS PLA composites through FESEM. The FESEM image shows the surface morphology of SS particulates were dispersed uniformly in the PLA matrix. The fillers were blended in PLA were identified and confirmed through FESEM. Also, SS particulates have irregular in shapes, and size was identified from the FESEM image. Further reducing the size of the dispersion of the particulate could be improved further.

The EDX analysis of 20 wt. % SS PLA composites gave an organic and inorganic composition of C, O, K, Si, and Mg was obtained as 57.23, 40.93, 1.81, 0.01, and 0.01 wt. % as shown in Figure. 5 and given in Table 3.

Table 3  
EDX Analysis

Elements	Norm. C [wt. %]
C	57.23
O	40.93
K	1.81
Si	0.01
Mg	0.01

## 4.3 DSC Analysis

Figure 6 depicts the DSC curve of 20 wt. % SS PLA Composites. 20 wt. % SS PLA composites was heated steadily from 10°C to 500°C at a rate of 20/min. The Figure. 6 shows a negative peak which means that the 20 wt. % SS PLA Composites absorbs energy due to an endothermic reaction. The peak melting temperature was found to be 312.4°C and at this transition, the material changed from being relatively hard to a rubbery material. The energy absorbed by the powder at peak temperature was found to be -196.8J/g.

## 4.4 TG Analysis

Figure. 7 depicts the TGA curve of 20 wt. % SS PLA composites. The thermal stability of PLA was evaluated by using TGA in the range of 10°C to 600°C (30°C/10.0(K/min)/550°C). The analysis measures the quantity of rapid mass degradation of the 20 wt. % SS PLA Composites. Up to 280±5° C the weight of 20 wt. % SS PLA composites was stable and there was no change in mass in that range of temperature. When the temperature was increased beyond 280°C to 300°C huge quantity of weight was decreased. On further increasing the temperature from 300°C to 547.9°C remaining 20 wt. % SS PLA composites had been decomposed and exhausted. During this most functional presence in the composites was decomposed. The residual mass was found to be 12.23%.

## 4.5 Mechanical Characterisation of the Composites

The tensile strength of PLA 37±0.5 MPa was found and it was gradually increasing by varying the particulate from 0, 10, and 20 wt. % respectively. The tensile strength was gradually increased by increasing the SS particulates contents in PLA composites which were observed in the test. The 10 and 20 wt. % of SS PLA composites exhibited a tensile strength of 41±0.5 MPa and 43±0.5 MPa respectively. Compare to pure PLA composites the 20 wt. % SS PLA composites revealed the tensile strength was more than 15% of pure PLA.

SS PLA (0, 10, and 20 wt. %) composites were carried out and found the compressive strength and results were compared. The compressive strength was found for 0, 10, and 20 wt. % SS PLA composites were

found as 33 MPa, 36 MPa, and 37 MPa respectively. The compressive strength was increased from 0 to 20 wt. % SS PLA (0, 10, and 20 wt. %). The flexural strength of the composites was carried and the results were compared. Maximum flexural strength 1.86 MPa was found at 20 wt. % SS particulates filled HDPE composites. The flexural strength was increased from 0 to 20 wt. % SS particulates filled HDPE composites after that is started decreasing strength.

The Shore D hardness values are measured at five different locations at each pure PLA and 10, 20 wt. % of SS PLA composites specimens. The highest hardness value was found to be 80.1 SHN for 20 wt. % SS PLA composites. The pure PLA, 10, and 20 wt. % of SS PLA composites exhibited Shore D hardness 76.7, 77.8, 80.1 SHN respectively. The SHN results were revealed that increasing the SS fillers in PLA enhanced the hardness of the composites. The maximum hardness of the 20 wt. % SS PLA composites were increased 4. 5% compared with pure PLA. A comparison of mechanical properties was given in Table. 3

Table 3  
Comparison of Mechanical Properties

Sl.No	Composites Specimen	Tensile Strength (MPa)	Compressive Strength (MPa)	Flexural Strength (MPa)	Shore D Hardness
1.	Pure PLA	37±0.5	33±0.5	66±0.5	76±0.5
2.	10 wt. % SS PLA	41±0.5	36±0.5	70±0.5	77±0.5
3.	20 wt. % SS PLA	43±0.5	37±0.5	72±0.5	80±0.5

## 5. Conclusion

The structural and elemental compositions of SS particulates were studied using XRD and EDX analysis. The surface morphology was studied through FESEM. SS PLA composites specimens were characterized the thermal stability using TGA and DSC. The peak melting temperature was found to be 312.4°C and at this transition, the material changed from being relatively hard to a rubbery material. The energy absorbed by the SS PLA composites at peak temperature was found to be -196.8J/g. When the temperature is increased beyond 280°C to 300°C huge quantity of weight had decreased. On further increasing the temperature from 300°C to 547.9°C remaining 20 wt. % SS PLA composites had been exhausted. Compared to pure PLA composites the 20 wt. % SS PLA composites revealed the tensile strength of more than 15% of pure PLA The flexural strength was increased from 0 to 20 wt. % SS particulates filled HDPE composites after that is started decreasing strength. The maximum hardness of the 20 wt. % SS PLA composites were increased 4. 5% compared with pure PLA.

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## Figures

Figure 1

a) Samanea Saman Fillers b) PLA c) SS PLA composites specimen 2.2 Fabrication of the Composites

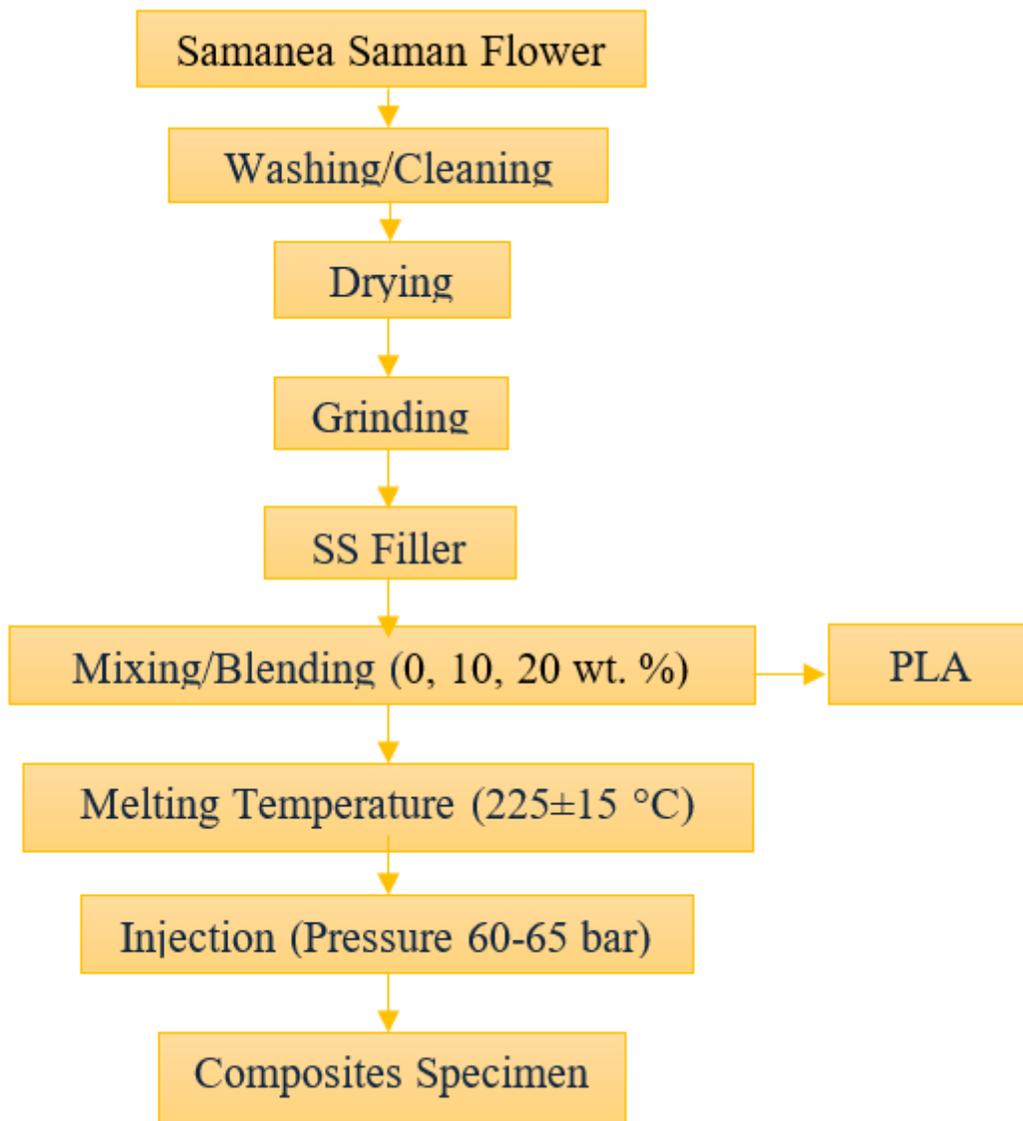
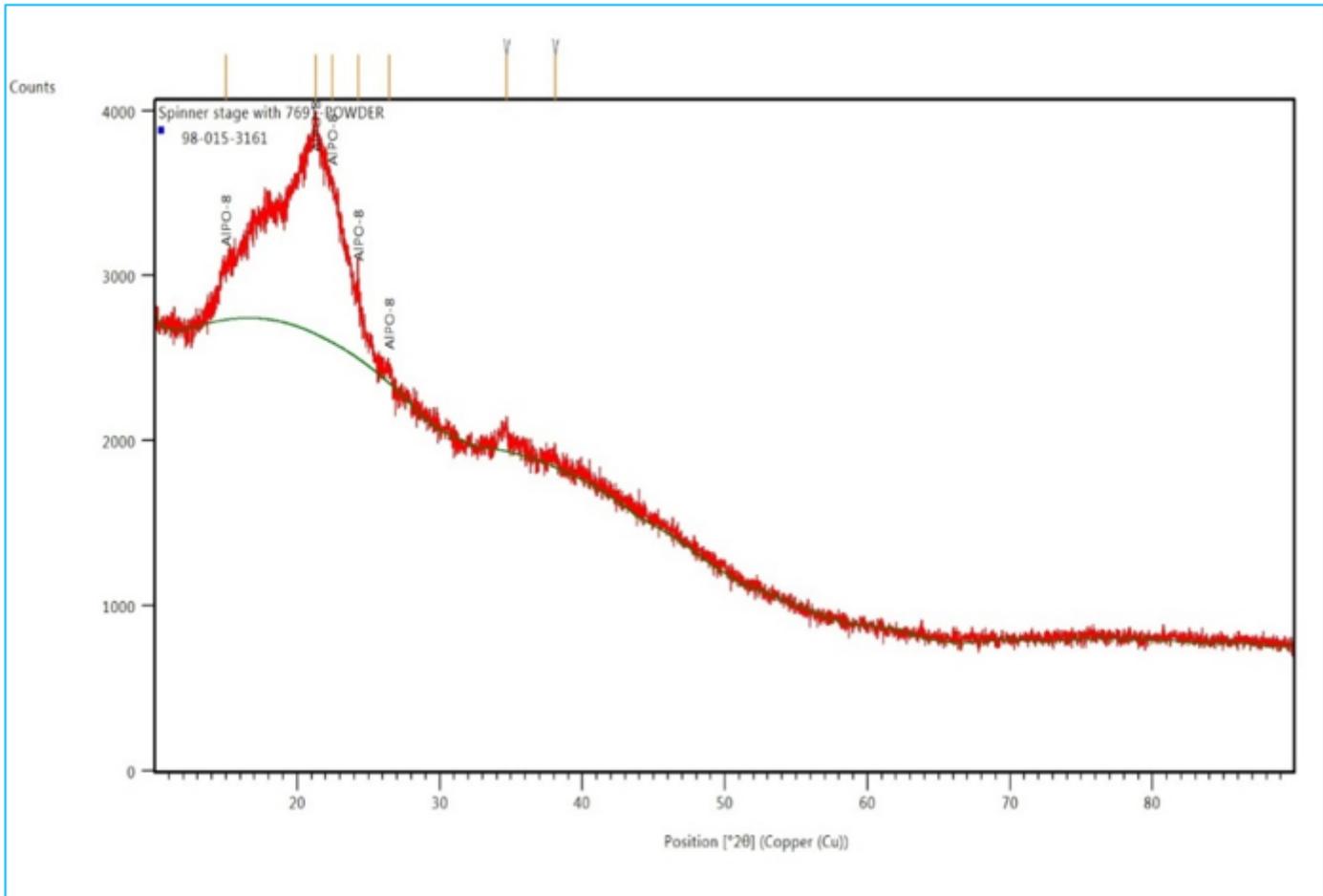


Figure 2

## Methodology



**Figure 3**

XRD Spectrum of SS PLA Composites

**Figure 4**

Surface morphology 20 wt. % SS PLA Composites

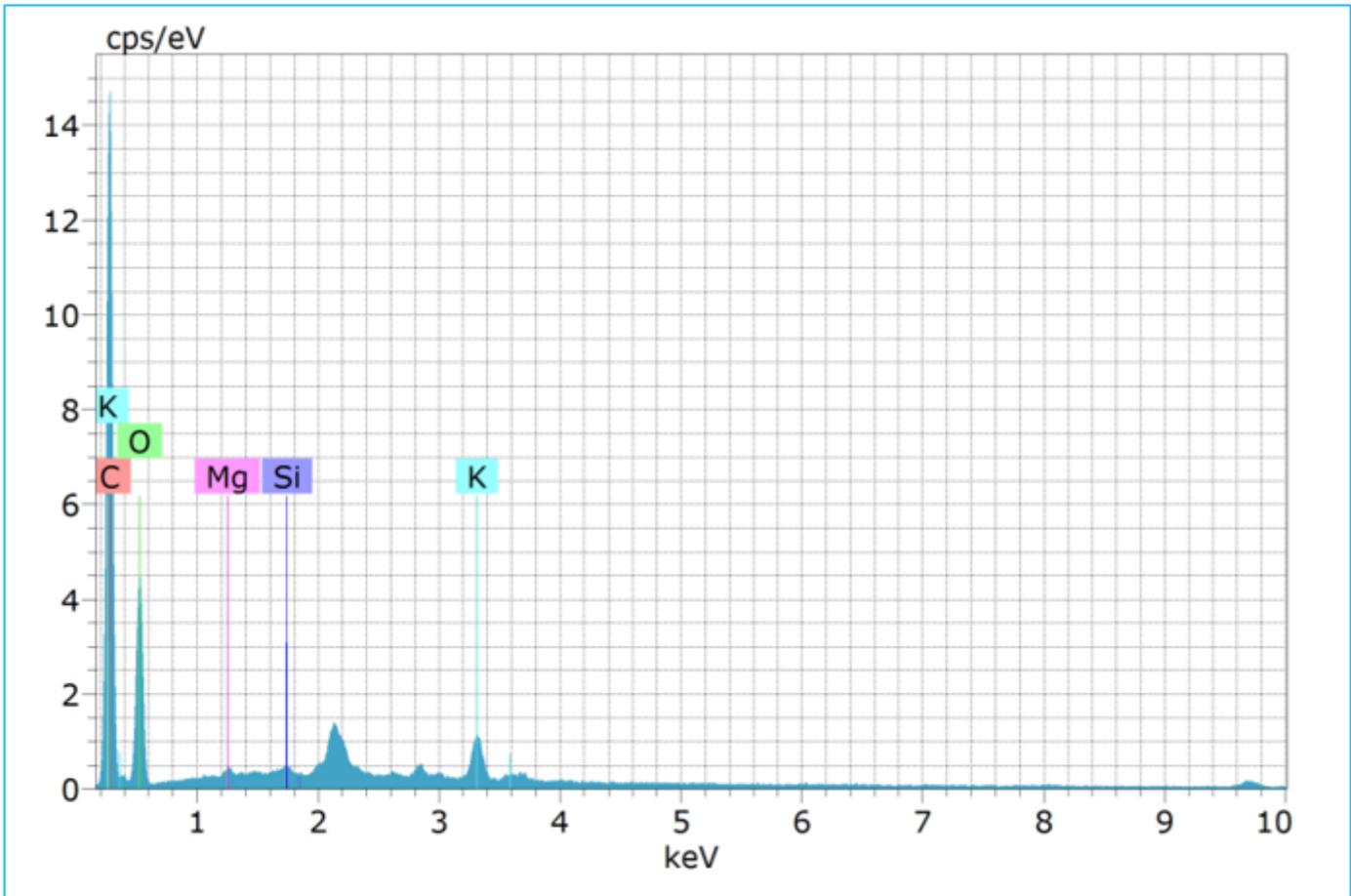


Figure 5

EDX analysis of 20 wt. % SS PLA Composites

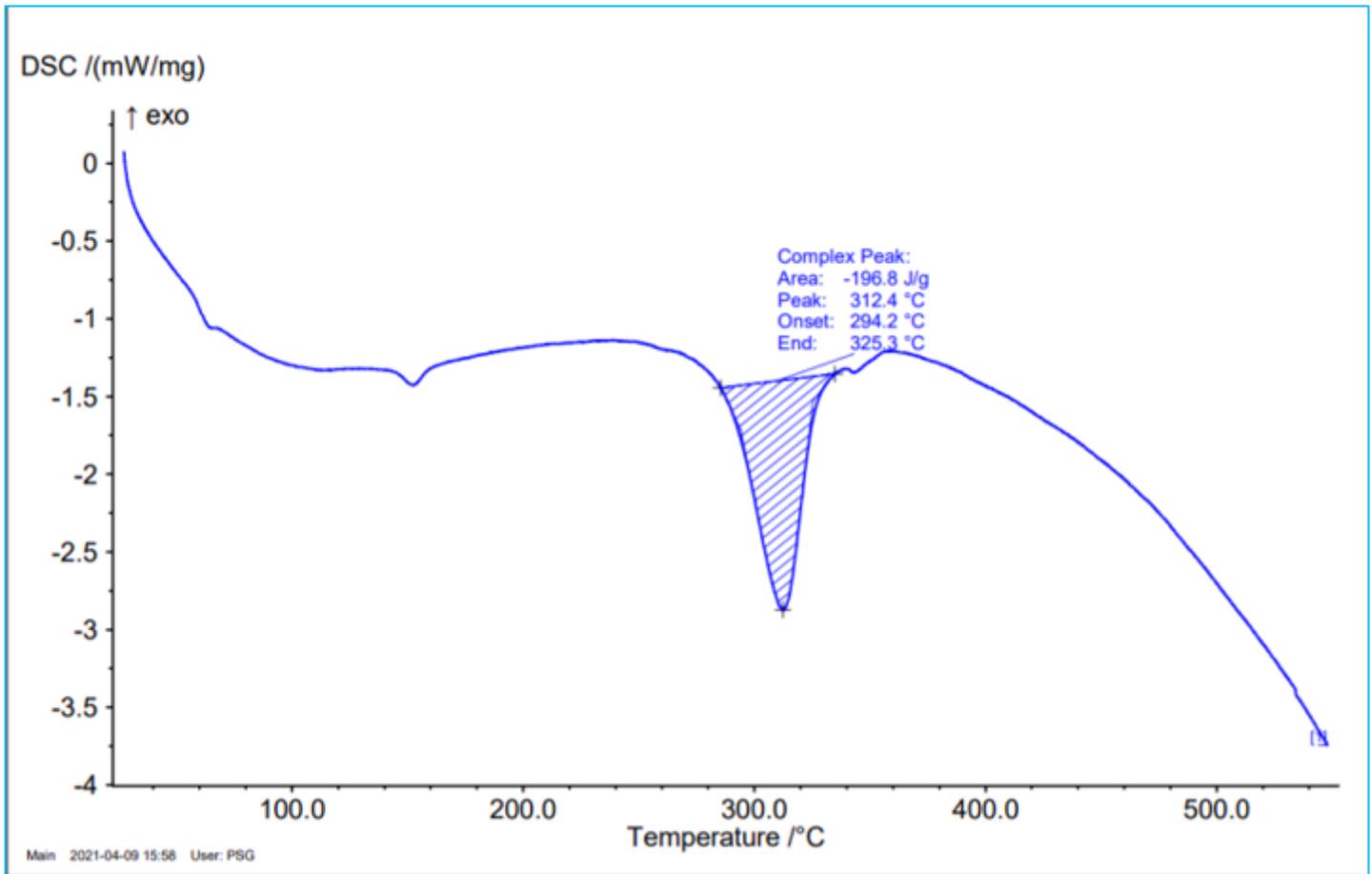


Figure 6

DSC curve of 20 wt. % SS PLA Composites

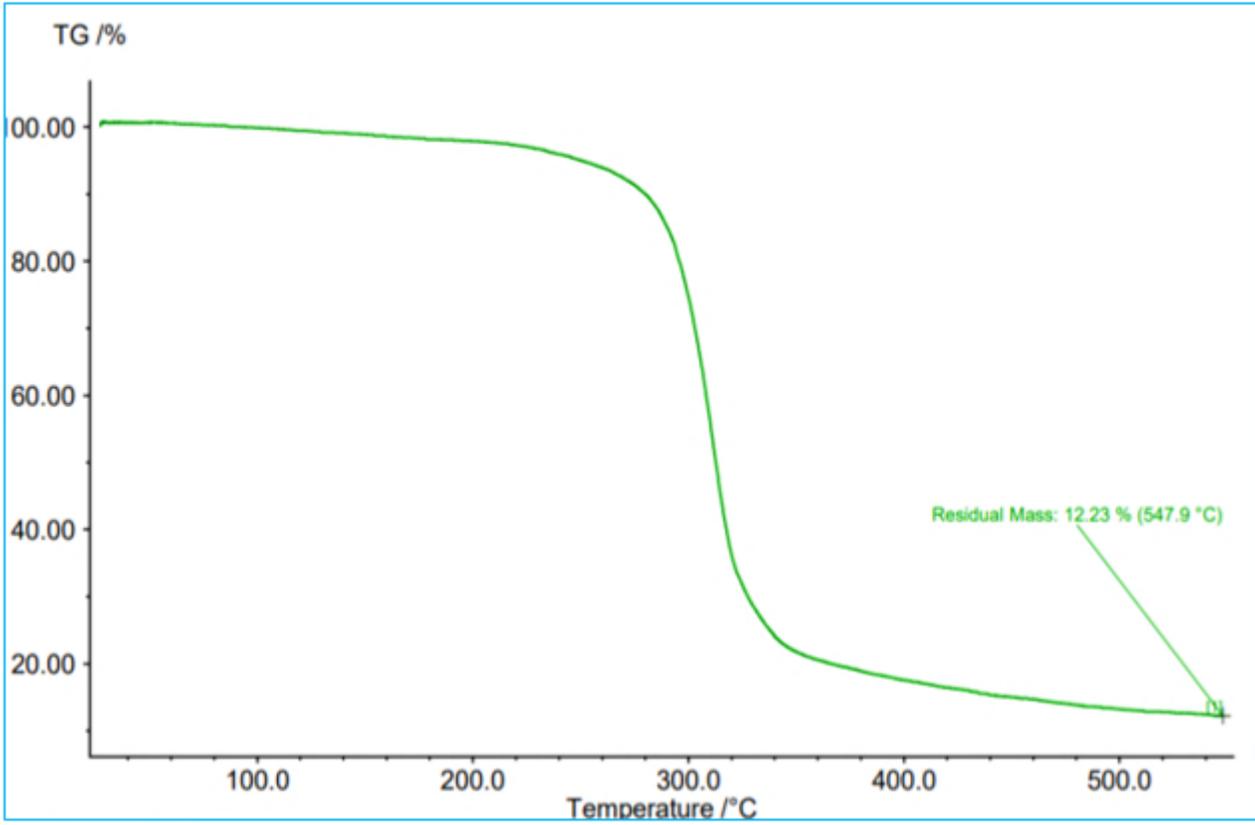


Figure 7

TGA curve of 20 wt. % SS PLA Composites