Chapter 05. Characterization Techniques

5.1 Preamble

Different characterization techniques were utilized in this to study the CFF/PLA composite filaments and samples prepared by extrusion and the sandwich method. The flow chart shown in Figure 5-1 depicts different characterization techniques which have been utilized in the present study.

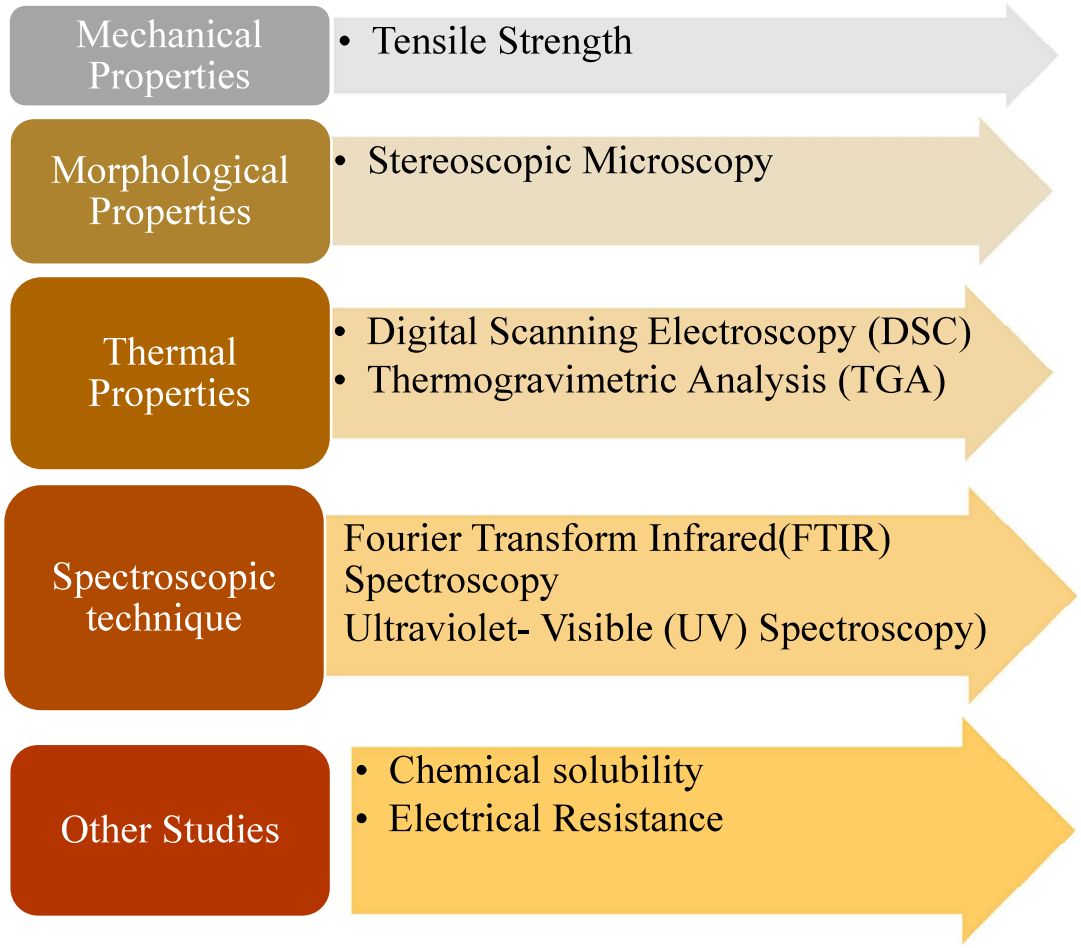

Figure 5-1 Flow chart of different characterization techniques.

5.2 Mechanical Properties

The mechanical properties of samples are one of the most important properties required to be known before selecting them for any application. In the subsequent section, details of the measurement of tensile strength are described.

5.2.1 Tensile test

The composite filaments were tested on Tinius Olsen 10ST Electromechanical Universal Testing Machine (UTM) (as shown in Figure 5-2) as per the standard - ASTM D638 for its tensile properties.

Figure 5-2 Tinius Olsen 10ST Electromechanical Universal Testing Machine (UTM).

The tensile test of the CFF/PLA composite samples prepared by the sandwich method was tested following the ASTM 3034 standard. Figure 5-3 shows the shape and size of the samples prepared for the tensile testing

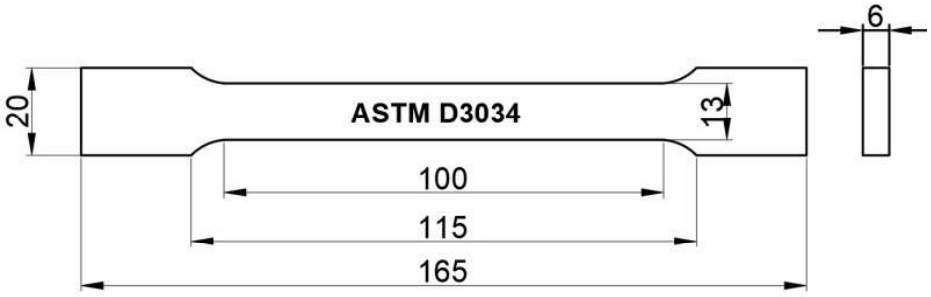

Figure 5-3 Shape and Dimension of the tensile sample as per ASTM D3034 (all dimensions are in mm).

5.3 Stereoscopic Microscopy

For carrying out the morphological studies of the CFF/PLA composite filament, it was observed under a stereo microscope by NIKON SMZ 1000 (Figure 5-4) with a maximum of 100x magnification. The filament diameter was also measured using the MetaVue software which is coupled with the stereo microscope. The tensile tested samples were observed under the microscope for their surface condition and observation of pinholes, distribution of fiber inside the filament and any other changes.

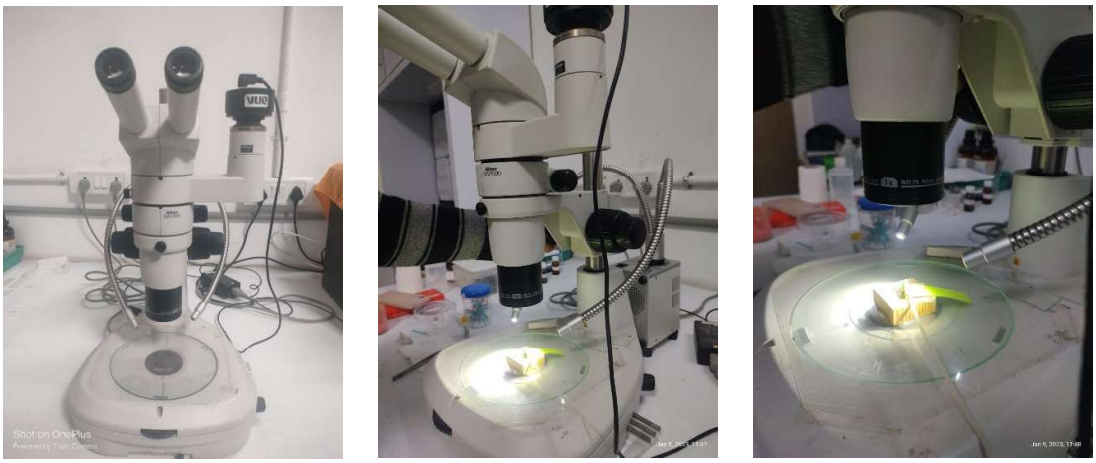

Figure 5-4 Samples observed under a stereo microscope by NIKON SMZ 1000.

These observations have laid the foundation for carrying out further investigations of thermal properties and spectroscopy.

5.4 Thermal Properties

Observations of the thermal properties indicate the behavior of the composite over a temperature range. The DSC measures the heat flow in a sample while TGA is used to observe the weight change over a temperature range.

5.4.1 Digital Scanning Electroscopy (DSC)

Digital Scanning Electroscopy (DSC) was employed to measure the thermal transition of the CFF/PLA composite filament in terms of heat flow over a temperature range. The DSC was carried out on DSC-60 Plus Make: Shimadzu, Japan at the Applied Chemistry Department of The MSU of Baroda, Vadodara.

Figure 5-5 DSC-60 Plus, Make: Shimadzu.

The following parameters were set for the instrument to carry out the DSC analysis:

- Atmosphere : Nitrogen
- Flow Rate : 100[ml/min]
- Cell : Aluminum
- Sample Weight : 6.000[mg]
- Sampling Time [sec]: 0.1
- Temperature gradient : 15.00 °C/min
- Max Temperature : 400.0 °C

5.4.2 Thermogravimetric Analysis (TGA)

The analytical technique of Thermogravimetric analysis (TGA) was employed for the CFF/PLA composite filament to determine the thermal stability by obtaining the weight loss over time when subjected to heating at a controlled temperature gradient. TGA is used to analyze the changes in the physical and chemical properties of the samples. Phenomena such as second order phase transitions, decomposition and dehydration, and degradation of the samples are studied. The TG analysis was carried out on TGA-50 Make: Shimadzu, Japan (shown in Figure 5-6) at the Applied Chemistry Department of The Maharaja Sayajirao University of Baroda, Vadodara.

Figure 5-6 TGA-50 Make: Shimadzu, Japan.

The analysis was carried out with the following parameters:

- Atmosphere : Air
- Cell : Platinum
- Sample Weight : 22.930 mg
- Temperature gradient : 10.00 °C/min
- Max Temperature : 400.0 °C

5.4.3 Derivative Thermogravimetric Analysis (DTGA)

The first derivative of the Thermogravimetric analysis (TGA) curve is used to determine the maximum peak temperature of the mass loss and can be used as an indicator for the host materials present in the sample (Libourel et al., 2021). In the present study, OriginPro software is used to obtain the first derivative of the TGA curve.

5.5 Spectroscopic Technique

Spectroscopic techniques utilize ultraviolet, Infrared, or visible light to identify the chemical bonds and predict the molecular structure of the sample being tested.

5.5.1 Fourier Transform Infrared (FT-IR) Spectroscopy

The Fourier Transform Infrared (FT-IR) spectroscopy is utilized to analyze the interaction between the matrix (PLA) and reinforcement (CFF) at the interfaces and the formation of chemical interactions at the interfaces. The FT-IR is a plot of intensities of absorbance or transmittance versus the wave frequency (in cm-1). For the study, a PerkinElmer Frontier IR (Figure 5-7) is to carry out the FT-IR spectroscopy.

Figure 5-7 PerkinElmer Frontier IR.

The interactions between the molecules and the electromagnetic field in the IR region are studied in this technique. The sample absorbs light in regions of certain frequencies and excites at a higher vibrational state. The absorbed frequency corresponds to various chemical bonding and functional groups present in the filament sample.

5.5.2 Ultraviolet-Visible (UV) Spectroscopy

When light interacts with the molecules of the solution, its energy content increases. The electrons are excited to higher molecular orbital, resulting in a distinct spectrum. This spectrum can be used to detect certain functionalities or chemical groups. In the present study, UV/VIS spectrophotometer (as shown in Figure 5-8) was utilized.

Figure 5-8 BR Biochem UV/VIS Spectrophotometer.

For the study, first the filaments were dissolved in 5% by weight NaOH solution at 100°C. A Quartz cuvette is used to load the sample in the spectrophotometer. The visible spectrum was used for the analysis purpose.

5.6 Other Studies

Along with the mechanical, morphological, thermal, and spectroscopic characteristics it is required to check other characteristics such as chemical solubility and electrical resistance to widen the scope of application of the composite developed.

5.6.1 Chemical solubility

Biomaterials are soluble in acidic medium. (Deshayes and Kasko, 2013). CFF and PLA both being biomaterials are also soluble in acidic medium. It is not necessary for biomaterials to be soluble in an alkaline medium. Thus, the CFF/PLA composite filament was tested for solubility in an alkaline medium as shown in Figure 5-9.

5% by weight NaOH dilute solution was selected for testing (Fitriyanto et al., 2022). The weight of the filament used was 1 gm. The composite filament was also immersed in the solution and boiled till the entire water in the solution was evaporated.

Figure 5-9 Boiling of CFF/PLA filament in 5% weight NaOH solution.

5.6.2 Electrical Resistance

The filaments were tested for their electrical resistance. First direct resistance was measured using a multimeter. The filament piece was connected across AC and DC and the voltage was varied from 0 – 230V. The setup for measuring the resistance in various conditions is mentioned in Figure 5-10.

(a) (b) (c)

Figure 5-10 a) Electrical resistance measurement (b) Resistance to AC voltage (c) Resistance to DC voltage.

The samples were connected to the two terminals and supplied with AC and DC voltage. The measurement was observed using an M65 multimeter (Motwane Make).