

Mechanical Properties, DMA and Structural Analysis of Cassia Auriculata/PLA Blended Hybrid bio Matrix Composite

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Abstract: In this study, hybrid composite material composed of Poly Lactic Acid (PLA) and Cassia Auriculata (CA) were fabricated and the mechanical properties were evaluated to assess the impact of bio filler loading in the bio polymer (PLA) matrix. In order to fabricate the composite specimens, the CA bio filler was added to the PLA matrix in proportions of 10%, 20%, 30%, 40% and 50% by volume. In line with ASTM specifications, experiments were conducted to establish how the hybrid bio matrix, which was composed of both PLA and CA influenced the composite's mechanical characteristics. The test results indicated that adding 30 percent CA to PLA enhanced the material's mechanical, dynamic mechanical analysis, thermogravimetric properties. The composite composition with better properties was considered for further fabrication of a colposcopy fixture, the component was fabricated using additive manufacturing methodology.

Keywords: Cassia Auriculata, PLA hybrid matrix, mechanical properties

1. Introduction

To improve the impact strength, wear resistance, thermal stability, and strength of polymer composites, a number of studies [1-5] have explored the impacts of bio and synthetic particle reinforcements and fillers on the characteristics of the polymer matrix. Some of the most widely used powdered synthetic fillers are talc, carbon nanotubes, silicon carbide, nanoclay, aluminium flakes, calcium carbonate, etc. Recent research [6-8] has placed an increased emphasis on the utilisation of naturally occurring elements as filler components for polymer matrices that are categorised as bio fillers. Examples of these materials are sawdust and powder made from ground up coconut shells [9-10]. The polymer-based composites' reduced impact on the surrounding ecosystem is a direct result of the incorporation of biofillers. When compared to polymer-based composites that only contain synthetic reinforcements, they have a significantly higher rate of biodegradability. It has been shown that the properties of composites can be altered not only by the surface treatment of the fibres but also by the amount of filler that is included in the polymer matrix. According to the findings of a study done by Sudheer et al. [11] on the tensile and flexural properties of an epoxy composite reinforced with potassium titanate whiskers, the inclusion of the filler diminishes the composite's tensile and flexural strength. The greatest percentage of loss in tensile strength occurred when the whisker loading reached 15% of the total. The addition of filler material, upto 15% reinforcement loading led to a rise in the flexural as well as the tensile moduli of the material. This is a direct consequence of the increased durability of the material. The researchers Onuegbu et. al. [12] have evaluated the impact that powdered snail shell had on polypropylene composites as part of their studies. According to the findings, an increase in tensile, flexural, and impact properties occurred as a result of the inclusion of the filler. This effect occurred regardless of the size of the filler. Regardless of whether or not the filler was recycled, this was the experimental outcomes.

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An research that was conducted by Kokta et. al. [13] on the mechanical behaviour of a polypropylene composite that included wood flour found that the addition of filler material to a composite material improved the latter's mechanical qualities. It was determined to be the case that the incorporation of filler material into a composite material results in an enhancement in the latter's mechanical properties. This was reported to be the case in their conclusion. The aforementioned research found that including filler materials into polymer matrix composites led to an improvement in the composites' overall mechanical properties.

In the present investigation, a hybrid bio matrix composite made up of CA and PLA is fabricated for obtained desired mechanical properties in biomedical applications such as colposcopy screening etc.,

The purpose of this study is to develop the modified composite material by incorporating the *Cassia Auriculata* in the neat PLA resin. Experiments to evaluate mechanical properties such as tensile, flexural and impact properties were performed to assess the impact of powdered bio filler in the PLA polymer matrix.

2. Materials and methods

2.1. Materials

Cassia Auriculata, an abundantly available natural filler material in India, was used as particulate filler in PLA matrix in this work. *Cassia Auriculata* is common throughout the southern part of India. The powdered and processed particle filler was procured from a local vendor in Kanchipuram, and the natural filler was sun-dried for two days to remove any remaining moisture content. In this work, the PLA matrix was utilized to explore the influence of adding *Cassia Auriculata* on the composite's mechanical properties. PLA was chosen as the material of choice for this study since it is the most commonly used material for 3D printing.

2.2. Preparation of CA natural filler powder

The CA plants in Chennai, India are harvested for their gum. Figure 2: CA gum is preserved in dry conditions at room temperature for 20 days and then kept in oven at 40°C for one hour. For next 20 min, coarse gum is agitated in a high-speed mechanical dynamic mixer (Sunkaier SK090) to create fine powder of particle size (10nm) [4].



Figure 1. Cassia Auriculata Gum

2.3. Preparation of specimens for PLA composites

Specimens with PLA acid solid material filament are heated to melting stage, injected via the nozzle and combined with CA natural filler powder loading (10, 20, 30, 40 and 50 vol%) are created by the process of 3D printing [20] as shown in Figure 2.

The ability to upload 3D designs to the cloud is another feature that 3D printer manufacturers offer, which will aid in the spread of 3D printing. Open-source CAD software is required to print the specification size of object (Specimens for tensile strength, flexural strength and impact strength). Powder bed fusion (PBF) uses heat energy to melt PLA composites to create hybrid composites using

CA as a natural filler. Composites are created using 3D printing in a manner similar to that shown in Table 1.

Table 1. Composition of the PLA/CA specimens

Sl. No	Composition Specimens	Matrix	Reinforcement
1.	A	PLA	10% V of CA
2.	B	PLA	20% V of CA
3.	C	PLA	30% V of CA
4.	D	PLA	40% V of CA
5.	E	PLA	50% V of CA

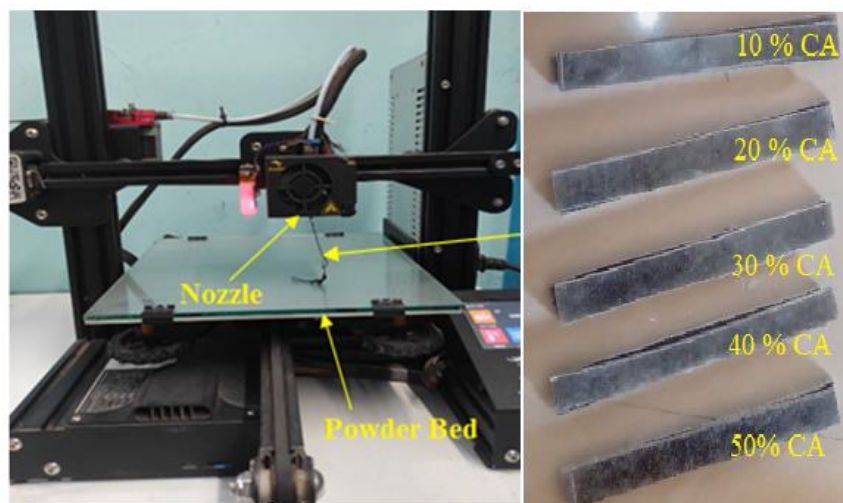


Figure 2. 3D printing machine and Photograph of PLA/CA specimens

2.4. Composites testing

The specimens of the manufactured composites are put through a variety of tests, including the tensile test, impact test and flexural test to ascertain its mechanical properties. The average values of the results are determined for each category using five composite specimens.

2.4.1. Tensile testing

The Instron Universal Testing Machine is utilized to conduct the tensile test on composite materials with a load range of between 0 and 100 kN as well as a cross head rate ranging from 5 to 250 mm/min. This test is evaluated using the ASTM D 638 standard as shown in Figure 3.

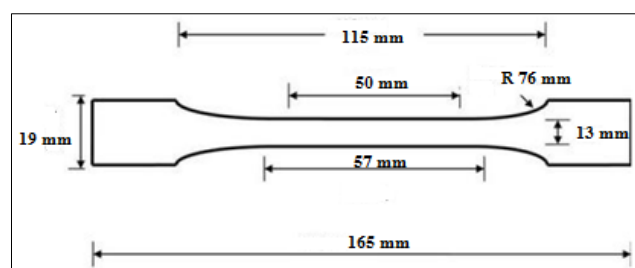


Figure 3. Specification for the tensile test specimen (ASTM D638)

2.4.2. Flexural testing

The Instron Universal Testing Machine is used to conduct flexural test on composite materials with a load range of between 0 and 100 kN as well as a cross head rate ranging from 5 to 250 mm/min. This test is evaluated using the ASTM D 790 standard as depicted in Figure 4.

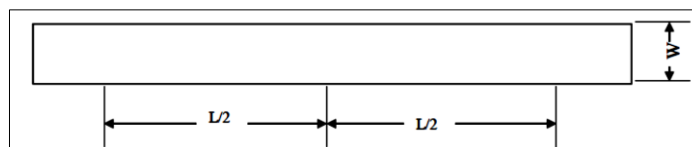


Figure 5. Specification for the flexural strength specimen (ASTM D790)

- W – Specimen's width = 13 mm
- L – Span length between two rollers = 4 X W = 52 mm
- T – Specimen's Thickness = 3mm

2.4.3. Impact testing

A pendulum machine (EMIC) is used to conduct the Izod impact test in accordance with ASTM D 256, as depicted in Figure 5. For each composite, the average outcomes from five samples are presented.

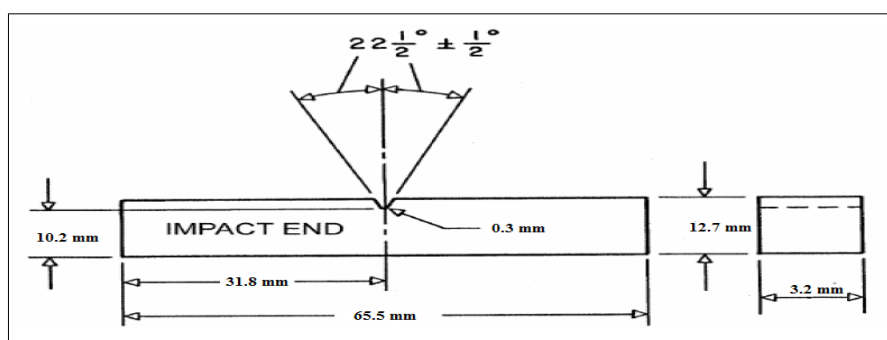


Figure 6. Specification for the impact strength specimen (ASTM D256)

2.5. Dynamic mechanical analysis (DMA) test

DMA test is carried out to assess the storage modulus and damping factor for composites under nitrogen conditions at temperatures ranging between 30 to 240°C (3 - 5°C/min) at ten Hz in the tensile mode as shown in Figure 7.



Figure 7. Dynamic mechanical analyzer

2.6. Design and fabrication of colposcopy fixture using Cassia Auriculata/PLA blended hybrid bio matrix composite

Colposcopy is an equipment used by medical professional in examining the cervix for any anomalies in the tissue or presence of Human Papilloma Virus (HPV). It was required to design and develop a

proper fixture/stand for the colposcopy in order to do the examination with ease. In this regard, a simple fixture used to hold the colposcopy was designed and developed to assist the medical professionals. The stand was developed based on the functional requirements with the dimensions to hold the colposcopy. The material which is used to produce the colposcopy fixture meant for the external usage only. Through the colposcopy unit will be used for medical applications, it doesn't require any biocompatibility as the device which is not meant for any kind of internal use either temporary or permanent. Also, the final application requires no physical contact with the end user hence, there is no possibility of any allergic reactions by the use of this material. And hence this material can be used safely for the intended purpose.

3. Results and discussions

3.1. Mechanical properties of *Cassia Auriculata*/PLA composites

Figures 8, 10 and 11 show the tensile, flexural, and impact strengths of composites made of *Cassia auriculata* bio filler and PLA bio polymer that have different volumes and percentages of *Cassia auriculata* in particulate form.

3.1.1. Tensile properties

Tensile strength and modulus of the bio composite with various bio filler loading fraction is presented in Figure 8. The results show that the mechanical characteristics of the PLA composite material are marginally enhanced by the inclusion of *Cassia Auriculata* bio filler. It is observed that the maximum value of experimental tensile strength of the bio composite material made of PLA polymer and *Cassia auriculata* was obtained for the specimen with 30% v/v bio filler. The material's capacity to withstand the stress was investigated. The composite specimen with a 30% filler content had a maximum tensile strength of 15.2 MPa during testing. With further addition of bio filler (40 and 50 % v/v) the tensile strength of the composite reduced indicating unfavorable filler percentage. Researchers had noted the various competitive benefits in natural filler substances that helped to increase the endurance of hybrid composites such as fracture sealing and crack stopping [14].

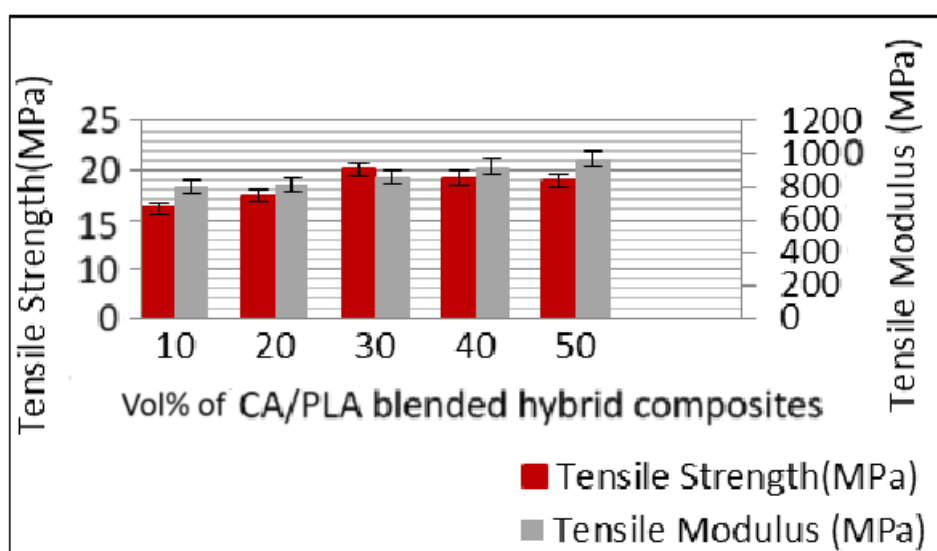


Figure 8. Tensile Properties of CA bio filler/PLA composites

3.1.2. Fractography observation

SEM images of (a) 10 vol%, (b) 30 vol% and (c) 40 vol% of *Cassia Auriculata*/PLA blended hybrid bio matrix composites as shown in Figure 9a, b, c. The enhancement in the tensile strength is attributable to the uniform distribution of the filler particles and the thorough saturation of the filler material that happens when the matrix mixed with the filler as shown in Figure 9b. Images of the scanning electron micrograph (SEM) images provide evidence that the filler was dispersed uniformly. In addition, the SEM

images reveals that the bonding between the composite of *Cassia Auriculata* and PLA and the clean PLA is superior [15]. Figure 9a, c showing the SEM image of the bio composite containing 10 and 40% filler reveals that the *Cassia auriculata*/PLA combination are agglomerated and clustered. As a result of the filler material accumulation, the composite turns into an amorphous condition.

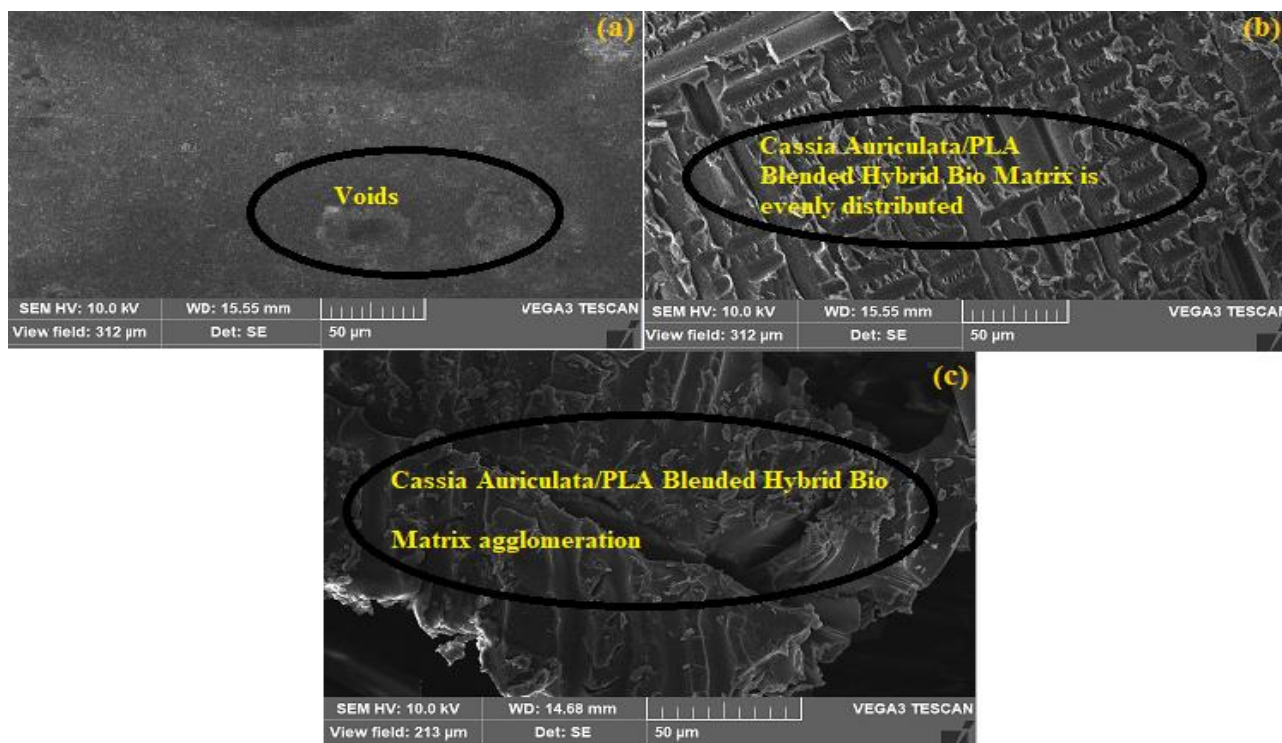


Figure 9. SEM images of (a) 10 vol%, (b) 30 vol% and (c) 40 vol% of CA/PLA blended hybrid bio matrix composites

3.1.3. Flexural properties

Figure 10 depicts the influence of the *Cassia Auriculata*/PLA composite reinforcement on the flexural strength and flexural modulus of the bio composite. Similar to the tensile qualities, the flexural strength and modulus of the composite material revealed that the addition of bio filler enhanced the flexural properties up to 30% addition of bio filler. When more than 30% v/v of *Cassia Auriculata* is added to a composite material, the flexural characteristics of the material are decreased. The decrease in flexural characteristics caused by an increase in the volume-to-volume ratio is attributable to two factors: agglomeration and porous contact between the filler and matrix [16].

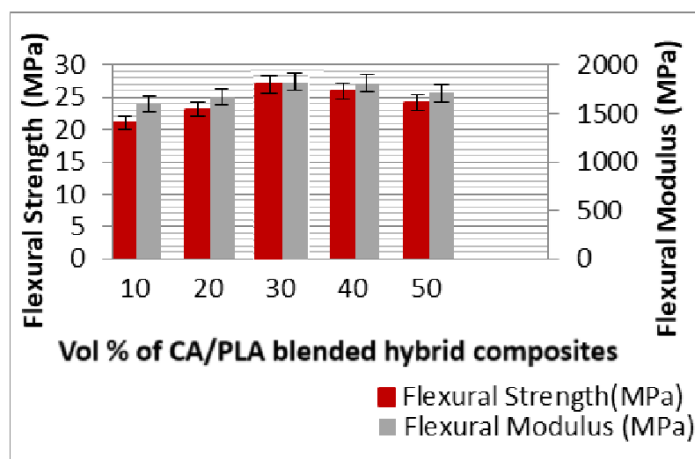


Figure 10. Flexural Properties of CA bio filler/PLA composites

3.1.4. Impact strength

The impact of *Cassia Auriculata* incorporation with PLA bio polymer on the impact strength is presented. It is noted from the Figure 11 that the incorporation of the bio filler up to 30% marginally improved the impact property which is similar to that of tensile and flexural properties. In all the cases the mechanical properties were enhanced by the incorporation of bio filler up to 30% v/v, and with additional filler content the properties decrease due to filler agglomeration and poor bonding between the filler and matrix [17].

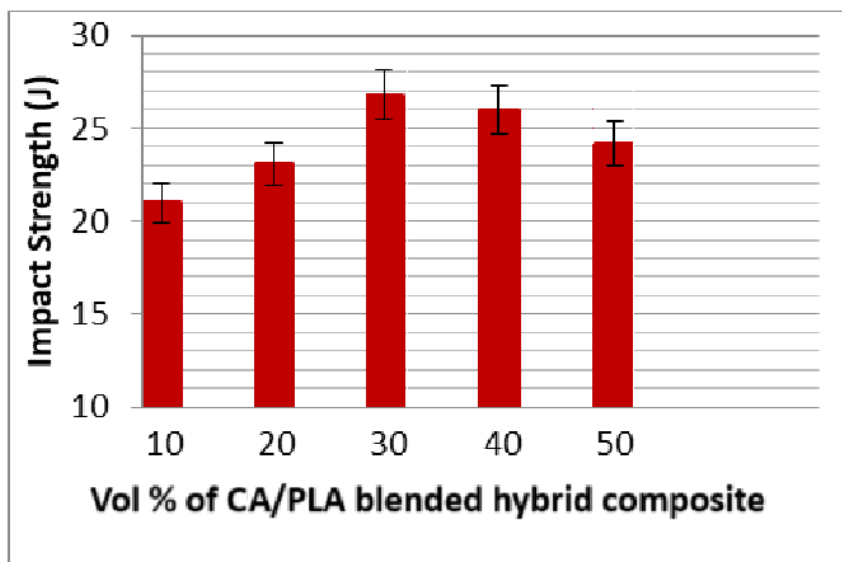


Figure 11. Impact Strength of CA bio filler/PLA composites

3.2. Visco elastic properties

The impact of bio filler loading and the temperature on the viscoelastic behavior of the bio composite was assessed at a frequency of 10 Hz is shown in Figure 12. Storage modulus, loss modulus and glass transition temperature, were studied in this analysis. The findings of the experimental investigation show that the energy storage capacity of the bio composite material can be improved by including *Cassia Auriculata* in the PLA bio polymer matrix. Due to the intermolecular mobility between the PLA matrix and the particulate filler material used in its retention, the PLA matrix was able to withstand cyclic loading at a frequency of 10 Hz. Figures 12 and 13 imply that PLA-resin has a higher tensile strength due to a lower storage modulus. Also it was noted that there had been an increase in the glass transition temperature (T_g) of the composite from 90 to 100°C as a result of the polymer chain's free molecular mobility. According to the findings of several researchers, adding filler or fibre to the polymer matrix can increase the stiffness of composite materials. The pictures obtained by the SEM clearly show situations that are conducive to bonding as well as those that are not. The inclusion of filler particles raises the storage modulus, denoted by the symbol "E," in both the glassy and rubbery sections of the DMA plot, as seen in Figures 12 and 13. The storage modulus values that are obtained are larger when the polymer composite is exposed for a shorter period of time (at a higher frequency). On the other hand, extended exposure to the composite results in lower storage modulus values (low frequency). To lessen the effects of localised stresses, the polymer's molecules were reorganised, leading to this phenomenon. The damping factor which is the ratio of a material's loss modulus to its storage modulus indicates the energy that the substance loses during loading as well as the mobility of molecular interactions between the polymer chains of the bio matrix.

The CA/PLA composite material's energy dissipation is improved because of the enhanced interface adhesion between the PLA matrix and the particulate bio filler as shown in Figure 13. Despite this, the energy loss is cut down if the filler content is more than 20% volume by volume. When compared to composite materials containing filler, PLA that does not include any additives has a lower peak height

for $\tan \delta$ (the damping factor). It may be deduced from this that elevating the filler loading will result in an increase in the composite material's stiffness. Studies demonstrated that incorporating natural filler to the PLA matrix had increased the rigidity of the hybridization composite material in both brittle as well as ductile regions. Experiments conducted by Khalil et al [2, 6] employing clay particles on the nanoscale yielded equivalent findings.

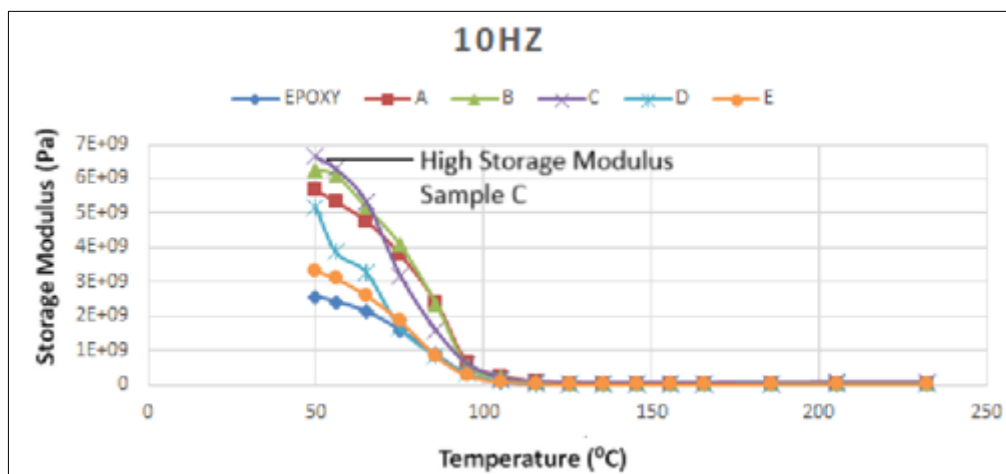


Figure 12. Impact of CA bio filler on storage modulus of PLA

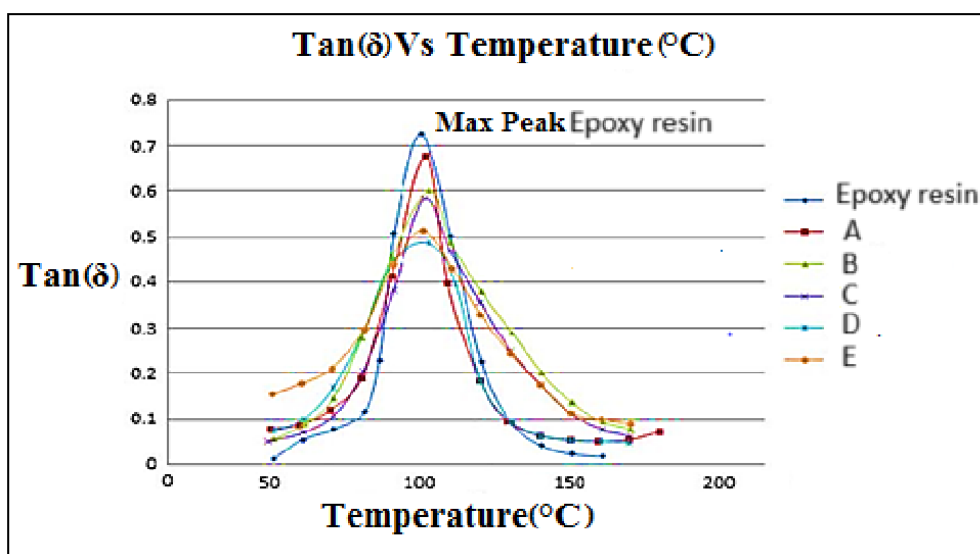


Figure 13. Impact of CA bio filler on damping factor of PLA

3.3. Structural analysis of the model using FEM

The schematic representation of the stand is shown in Figure 14. The stand was modeled in CAD software and it was fabricated using 3D printing technology. One of the key advantages of digital fabrication is that it permits the integration of CAD software tools into the fabrication of a product. Since PLA material is most widely used in 3D printing, an attempt is made to fabricate the model using the modified PLA incorporated with the natural filler in this work. A simple filament extruder setup was used to produce the modified PLA filament. The fabricated model is shown in Figure 15. The part was fabricated by elevating the melting point of the PLA filament and layer-by-layer extrusion. Using finite element method, it is possible to examine the deformations, and residual stresses of the final product. The purpose of this study is to perform preliminary investigation into the structural rigidity of the 3D fabricated part using modified PLA material. The material properties are fed as per the experimental

observations. Boundary conditions were given based on the actual usage requirement as shown in the Figure 16. The analysis was done by automatic meshing of the part and element size was selected by the software by default. The stress analysis results had revealed that the maximum stress is within the experimental strength and hence the design can also be considered safe and the composite material can be further explored for more testing and fabrication of working models [18].

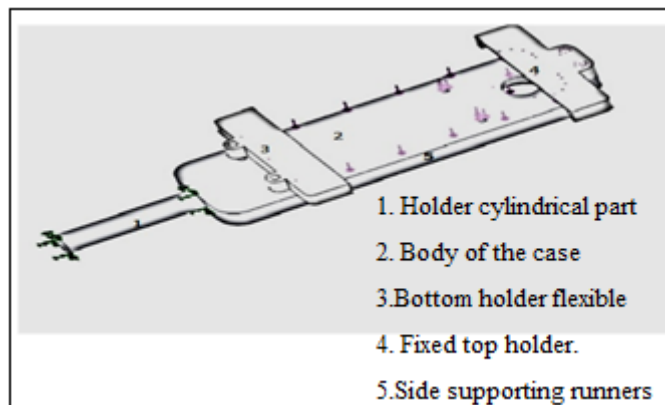


Figure 14. Schematic of the Colposcopy Stand

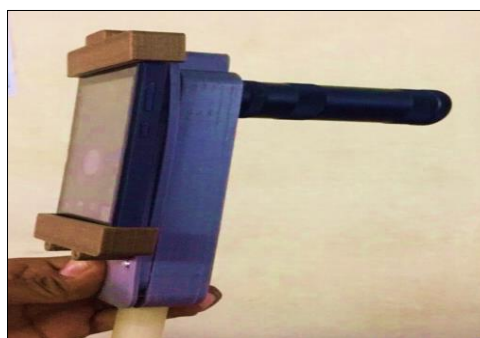


Figure 15. Fabricated prototype of the colposcopy using the hybrid PLA composite material

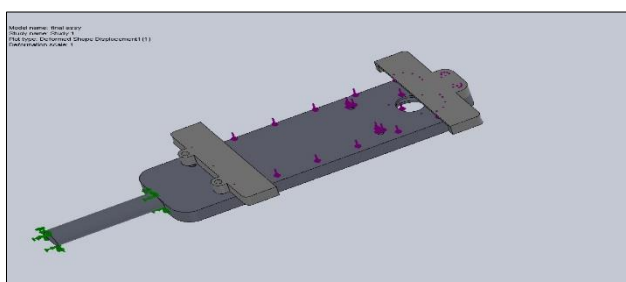


Figure 16. Boundary conditions for the FEA model

4. Conclusions

The findings of the mechanical strength revealed that the better mechanical properties are attained at 30% loading of *Cassia auriculata* in the PLA matrix. Further addition of *Cassia auriculata* diminishes the overall quality of the material owing to a variety of issues, one of which is the accumulation of the filler particles at higher volume fraction and poor surface wetting of the filler by the matrix leading to improper adhesion and inferior quality, as evidenced from the SEM images.

The modified PLA material was used to fabricate a fixture for colposcopy instrument. With the aid of additive manufacturing, the prototype was fabricated using the modified resin as the filament.



The model was successfully fabricated and FEM analysis was also performed to examine the structural rigidity of the model.

The test results had revealed that the deformations and stresses in the model were well within the safe limits. This study has shown that such kinds of 3D printed components can be fabricated using modified PLA resin which consists of natural filler.

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