Effect of Volume Fraction and Heating Temperature on Hybrid Natural Fibre Composites Developed Through the Die Moulding Process

Guravtar Singh Mann, Lakhwinder Pal Singh, Pramod Kumar

Abstract: Green composites are the materials which are made up of natural fibers and biodegradable matrix materials, which have the ability to replace the non-biodegradable, petroleum-based products. In this study, the focus is to develop the fully biodegradable green composites in which matrix material is selected as Polylactic Acid (PLA) reinforcement with jute and sisal fiber using hand layup followed by a compression molding technique. Composites are developed with different volume fraction from 25% to 50% and different temperature from 165°C to 195°C. Furthermore, the study of the failure mechanism of the tested specimens will be done with the help of a scanning electron microscope (SEM).

Keywords: Hybrid composites, Sisal, Jute, Volume fraction, Heating temperature

I. INTRODUCTION

The high consumption of the petroleum based products shows a negative impact to the environment while the natural fibers are categorized under environment friendly materials which contain many good properties as compared to the synthetic fibers [1]. Conserving the environment using natural raw materials has helped boost interest in biomaterial growth and use therefore lot of research is going on the filed of green composites[2]. As these materials are totally environmentally friendly and biodegradable[3]-[4]. Not only are the materials used in the packaging, automotive, power, sports and recreation industries submitted, but they also have sustainability for biomedical applications in implants and medical devices [5]-[6]. Known as the green composites and bio composites, the reinforcements of these materials are extracted obtained from natural resources [7]-[8]. To enhance mechanical properties of the composites lot of work is also going on in the field of hybrid composites. The Composites in which both the matrix and the fibers of the constituent are derived from natural resources are fully biodegradable green composites and composites in which one of the components is derived from natural resources are partly biodegradable green composites are known as hybrid composites [9]. Hybrid composites have recently received considerable attention from the scientific community due to their ability to give designers a new freedom to tailor composites, thus achieving properties that cannot be achieved in binary systems composed of one sort of filler / fibre spread in a matrix [10].

The features of hybrid composites rely on the single components that more constructively balance the intrinsic advantages and drawbacks. One of the drawbacks of filler / fiber might be ignored by the benefits of the other filler. In addition, the advantages of one type of filler / fiber could also be complemented by the advantages of the other hybrid composite filler containing two or more fibers / fillers [11]. Zhao et al.[12] used needle-punched method for fabrication of hybrid jute / glass composites in which jute mat was mounted on glass sheet. After a needle-punched process, the hand lay-up method was used to manufacture the composites with unsaturated polyester resin. Moreover the three-point bending tests were also carried to obtain flexural properties of different composites. The results showed that there was significant increase in mechanical properties because of hybridization. The effect of Nano titanium oxide fillers on the properties of hybrid jute-glass FRP composites was studied by Seshanandan et al.[13] nano titanium oxide particles-hybrid jute-glass FRP composites were produced using glass fiber chopped strand sheet, woven jute mat, polyester resin and Nano titanium oxide fillers using manual design process. It was observed that tensile strength, flexural strength and shear strength of the hybrid fiber reinforced plastics improved with addition of nano TiO2 filler particles. Hybrid composite of jute & carbon fiber reinforced epoxy composite was manufactured and compared to composites of jute-epoxy and wood-epoxy by Ramana et al.[14] Mechanical properties such as tensile strength, friction modulus, flexural strength, flexural modulus and impact strength were also tested. The findings showed that jute & carbon-epoxy hybrid composite could substitute carbon-epoxy composite without significant loss of tensile strength, flexural strength and flexural frame, as well as enhanced ductility and impact strength. Maleque et al.[15] Evaluated the flexural and impact properties of kenaf-glass (KG) fibers by reinforcing unsaturated polyester (UPE) hybrid composites. Composites were manufactured using the process of sheet moulding compound (SMC). The results showed that there was significant improvement in the mechanical properties. Ahmed et al.[16] Manufactured isenthalpic polyester composites woven jute and glass fabric through hand lay-up method and examined the effect of hybridization on mechanical properties. Results indicated that mechanical properties such as tensile strength and impact strength showed significant improvements with the incorporation of glass fiber in addition to improved composite water absorption with glass fiber. Khalil et al.[17] manufactured hybrid composites utilizing oil palm fiber and glass fiber as polyester reinforcement fibers and the hybrid effect of glass and EFB fibers on the tensile, flexural, impact and hardness of the composites were also evaluated.
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The results showed that hybrid composites exhibited good properties compared to the EFB/polyester composites. Chaudhari et al. [18] assessed the mechanical properties of jute, hemp, flax and its hybrid composites based on epoxy and proposed good mechanical properties for hybrid composites. Sreekala et al. [19] The impact of glass fibre introduction on the mechanical properties of phenol-formaldehyde composite reinforced oil palm fiber was also evaluated. The inclusion of glass fibers has been stated to have improved the mechanical properties. Ahmed et al. [20] examined the impact of glass fibres hybridization on mechanical properties of jute fibre reinforced polyester composite, and noted an improvement in mechanical properties of jute composite. In this experimental study Hybrid composites of Jute and sisal composites are developed with different volume fraction from 25% to 50% and different temperature from 165°C to 195°C. Scanning electron microscopy (SEM) analysis of tensile fractured specimens was carried out to understand the adhesion between fibers and matrix.

II. EXPERIMENT SETUP

The experimental setup for fabrication of green composites consist of compression molding machine. It consists of an upper part (punch plate) and the lower part (die block). There are different types of strips used for making different samples. The die is made of EN31 steel material which achieves very high compressive strength with high hardness value. First and foremost, consideration is the ASTM standard, for that the thickness of the composite maintained at 4mm and the dimension of the die is 320x120mm and thickness can be varied with the help of strips used in the die. The maintained thickness of die is 4mm for the fabrication of composite material. Before starting the experiment, the die washed with diesel. Secondary the die to accommodate the heaters are inserted in the upper as well as the lower plate.

III. MATERIAL AND METHODS

Natural fiber (jute and sisal) used as the reinforcement and PLA is used as the matrix material to make the green composite. PLA is used as matrix material. This is purchased from NaturTech India Pvt.Ltd. Chennai, India. The grade mentioned is 3052D. The selection of the material is done by observing the applications such as Candy twist wrap, Salad and vegetable bags, Window Envelop Film, Lidding Film, Label film, and other packing applications. The former line describes the application shows the 3052D is an ideal product for practical application. The density of the material is 1.24g/cc.

Going through the intensive literature survey, it has been observed that the volume fraction and applied pressure are the important parameters for the development of biodegradable green composites. During the literature, it has been found that temperature is one of the parameters can be considered which the research gap is. So, it has been decided that volume fraction and temperature are the operating parameters for the development of the composites.

Curing temperature of 165°C, 175°C and 185°C have been finalized after conducting the different trails at different temperatures till 200°C. During the trails, it has been observed that fibers get burned at a higher temperature (more than 195°C), so as per the observations come from trails, it has decided to develop the composites with a maximum curing temperature of 185°C. PLA is having a melting point of 160°C, thus curing temperature range was selected as 165°C, 175°C and 185°C.

As per the literature, it has observed that fibers are the load bearing members in the composites and plays an important role in defining the strength of composite material. After an intensive literature survey, it has decided to select the volume fraction as 25% to 50%. As after increasing the volume fraction more than 50%, it has been found from the literature that wetting of fiber gets decreased which leads to a decrease in the strength.

The other parameter was to find out the effect of curing, which is the amount of time required to reach an optimum viscosity or modulus at a certain temperature for an adhesive to cure fully for the fabrication of composites to protect from the breakage in bonding between fiber and matrix. Basically, the curing time is useful for observing the durability of composites. After conducting the number of trails on the basis of curing time for 10, 15, 20 and 25 minutes, it has been observed that the 15 minutes curing time is the best curing time in which the fabricated composites give the best results and the fiber volume fraction is the quantity of fiber present in the composite material. The fiber volume fraction obtained after fabrication of composite is cross verified with the formula

\[ V_f = 1 - (W_c - W_f) / (\rho_m V) \]

Where,

- \( V_f \) is the volume fraction of fiber
- \( W_f \) is the weight of the fabric
- \( W_c \) is the weight of the fabricated composite
- \( \rho_m \) is the density of the resin
- \( V \) is the volume of the fabricated composite.

For the experimental purpose hot pressing & compression molding techniques adopted through Literature Review. According to the selection of experiment the working procedure changed for the convenience of the work; it is divided into several steps. The development procedure of jute fabric composites is explained step by step given below:

**Step 1**
Cut the jute mat and sisal fiber fabrics into the particular size of the heating die and measure the required amount of PLA granules and placed in an oven for the preheating at 90°C temperature for the duration of 3 hours as per the researchers.

**Step 2**
Clean the die and heat it up to the required temperature at which the composite is going to be made.

**Step 3**
Once the temperature of the die has reached the required temperature, fix the Teflon sheet and the strips. Divide the PLA granules into two halves for the fabrication process. Firstly, the first half of the granules spread uniformly into the cavity and then measured the weight of catted jute mat placed over the PLA granules in to the cavity and then weighted sisal fiber placed over the jute mat layer and...
then another jute layer placed over the sisal fiber, finally the rest PLA granules are spread over the placed jute fabrics into the cavity. At last the Teflon sheet is placed on the lower die, the later the die has closed and cut pieces of Teflon sheets are placed in between the upper and lower dies. Finally, the whole arrangement of the die is kept in the CTM machine.

**Step 4**
The temperature is maintained as per the requirement and load starts to apply. Initially, 40 KN load is applied for the duration of 45 seconds then 60 KN, 90 KN, 110 KN loads are applied for the duration of 2.20, 3.50 and 8 min respectively. Later, the power supply has switched off and die kept under the same load for cooling at room temperature.

**Step 5**
Cooling time was limited to 80°C to open the die the composite has removed manually. Finally, the composite sheet has made to a thickness of 4mm. The diagram of the fabricated composite sheet as shown below:

**IV. MECHANICAL CHARACTERIZATION**

**A. Tensile test**
The tensile testing is conducted on Computerized Twin Screw UTM machine as per ASTM D3039 standard. The dimension of the specimen is 250x25x4mm. The model number of the machine is LSU-13 having a maximum load limit of 1000KG. The specimen is fixed between two jaws and axially load is applied by the machine which is parallel to the fiber direction. The longitudinal unidirectional fiber embeds in the matrix experiences the axial load and fracture takes place when material loses its elastic nature and extends plastically. Fig.1 and Fig.2 shows tensile specimens before and after fracture.

**B. Flexural test**
The flexural test is also done in the same UTM machine according to the ASTM standard D790-02. It is a three-point bending test. The specification of samples for flexure test is as per the ASTM standard is 120x15x4mm. Fig. 3 and Fig. 4 Shows flexural strength specimens before and after fracture.

**C. Impact test**
The impact test is a method for evaluating the toughness, impact strength and notch sensitivity of engineering materials. It is divided into two types Izod and Charpy test.

**D. Izod test**
The Izod test is done in the Izod testing machine according to the ASTM standard 256A. The specimen samples for Izod test is as per ASTM standard is 63.5x12.7x4mm. Create V notch on the specimen of depth and width 2.54mm and 10.16mm respectively and place the specimen at the position of cantilever beam on the bed of the machine. Fig.5 shows fractured Izod specimens.
E. Charpy test

The Charpy test is done in the Charpy testing machine according to the ASTM standard 256A. The specimen samples for Charpy test is as per ASTM standard is 127x12.7x4mm. Create V notch on the specimen of depth and width 2.54mm and 10.16mm respectively and place the specimen at the position of the simply supported beam on the bed of the machine.

D. Morphological characterization

The structural analysis will be done by Scanning electron microscopy (SEM). The pictures of samples are produced with an electron microscope by keeping it in a focused electron beam. The surface of the test piece is firstly cleaned with acetone and kept in vacuum for approximately 15 minutes to completely dry the surface. Scanning electron microscopy (SEM) was used to observe the surfaces and fractured surfaces of composites by using an accelerating voltage of 10 kV. The fractured surfaces were observed directly after mechanical testing and the specimens were coated with gold to facilitate the SEM observation. After this, the piece is kept on the microscope for the proceedings for SEM. The SEM results are taken at different magnifications and then analyzed.

V. RESULT AND DISCUSSION

According to the operating parameters all the composites were fabricated at 165°C, 175°C, 185°C and 195°C curing temperatures in volume fraction ranges are 25%, 30%, 35%, 40%, 45% and 50%. All the specimens were prepared according to the ASTM standards for the mechanical testing. The results of Mechanical Properties of PLA/Hybrid composites fabricated at 165°C and 175°C and 185°C and 195°C curing temperature are given below in the Table I, Table II, Table III and Table VI respectively.

A. Tensile strength

Experimental results of tensile strength for the Hybrid composites are shown in Fig. 6. Results shows that by adding the jute and sisal fabric, tensile properties of sisal composite have been improved. The tensile strength of sisal composite is 68.56 MPa at 165°C. Which shows that 165°C curing temperature gives the maximum tensile strength of sisal composite. Results shows the significant effect of Heating temperature on the tensile properties of developed jute and sisal fabric composites at 165°C, 175°C and 185°C and 195°C curing temperature are given below in the Table I, Table II, Table III and Table VI respectively.

It has been observed that with increase in the fiber content (volume fraction) in the jute and sisal fabric composites the tensile strengths were increased up to 30% volume fraction and then gradually decreased up to 50% volume fraction. It has been also investigated that with increase in the temperature from 165°C to 195°C the tensile strengths are also decreased respectively. As a result, it was found that at 165°C temperature with 30% volume fraction gives the maximum tensile strength was 89.43 MPa. Fiber pullouts, fiber burning, fiber breakages, pores in the matrix, unevenness in lignin content, debris, de-bonding between fabric and matrix, crack propagation are the main parameters affecting the tensile strength at different temperatures. Fiber pullouts and burring of fibers are more at 185°C as compare to 175°C and 165°C. It has investigated that the de-bonding between fiber and matrix, fiber breakages, pores in the matrix, unevenness in matrix and debris are increases as the volume fraction increases from 25% to 50%. At less volume fraction the material shows a brittle in nature because of proper bonding between fabric matrix interface.

At 30% volume fraction, the presence of proper matrix coverage on the fiber surfaces and fiber breakages indicates good interaction between fiber and matrix which results in better interfacial adhesion. SEM images of fractured composites at 165°C to 195°C at 30% maximum tensile and flexural strength and 50% minimum tensile and flexural strength.

Fig. 6. Effect of volume fraction and curing temperature on tensile strength of developed composites.

![Fig. 6. Effect of volume fraction and curing temperature on tensile strength of developed composites.](image)

Fig. 8. SEM images of fabricated composite

![Fig. 8. SEM images of fabricated composite](image)

Fig. 9. SEM images of fractured surface

![Fig. 9. SEM images of fractured surface](image)

B. Flexural strength

The flexural strength of sisal composite was recorded as 95.531 MPa at 165°C. Which shows that 165°C Heating temperature gives the maximum flexural strength of sisal composite. Results shows the significant effect of curing temperature on the flexural properties of developed hybrid composites at 165°C, 175°C,
185°C and 195°C composites. Experimental results of flexural strength for the jute fabric composites are shown in Fig.7

The outcomes of flexural strength of developed hybrid composites showed an increase in temperature from 165°C to 185°C after the temperature at 195°C the flexural strength is decreased. It has been observed that with increase in the fiber content (volume fraction) in the hybrid composites the flexural strengths were increased up to 30% volume fraction and then gradually decreased up to 50% volume fraction. As a result, it was found that at 165°C temperature with 30% volume fraction the maximum flexural strength was 126.81 MPa and 68.238 MPa was recorded at 165°C with volume fraction of 50%. It has been observed that with increase in volume fraction the flexural strengths are gradually decreased. For the better flexural strength, the bonding between fiber and matrix should be strong. The decrement in flexural strength with respect to volume fraction it is concluded that due to bad embedment between the fiber matrix interface.

![Graph showing flexural strength for composite developed at 165°C to 195°C temperature](image)

Fig. 7. Effect of volume fraction and curing temperature on Flexural strength of developed composites.

**Table-I: Heating at 165°C temperature**

| Sr. No. | Volume Fraction (%) | Tensile Strength (MPa) | Flexural Strength (MPa) |
|---------|---------------------|------------------------|------------------------|
| 1.      | Sisal composite 33.7| 68.56                  | 95.531                 |
| 2.      | 25%                 | 66.95                  | 104.524                |
| 3.      | 30%                 | 78.52                  | 115.473                |
| 4.      | 35%                 | 72.34                  | 103.337                |
| 5.      | 40%                 | 65.18                  | 114.63                 |
| 6.      | 45%                 | 56.85                  | 98.214                 |
| 7.      | 50%                 | 45.71                  | 85.118                 |

**Table-II: Heating at 175°C temperature**

| Sr. No. | Volume Fraction (%) | Tensile Strength (MPa) | Flexural Strength (MPa) |
|---------|---------------------|------------------------|------------------------|
| 1.      | Sisal comp (33.7)   | 68.56                  | 95.531                 |
| 2.      | 25%                 | 45.5                   | 75.543                 |
| 3.      | 30%                 | 54.36                  | 85.081                 |
| 4.      | 35%                 | 45.22                  | 78.847                 |
| 5.      | 40%                 | 42.74                  | 61.325                 |
| 6.      | 45%                 | 31.89                  | 50.854                 |
| 7.      | 50%                 | 29.34                  | 34.625                 |

**Table-III: Heating at 185°C temperature**

| Sr. No. | Volume Fraction (%) | Tensile Strength (MPa) | Flexural Strength (MPa) |
|---------|---------------------|------------------------|------------------------|
| 1.      | Sisal comp (33.7)   | 68.56                  | 95.531                 |
| 2.      | 25%                 | 45.5                   | 75.543                 |
| 3.      | 30%                 | 54.36                  | 85.081                 |
| 4.      | 35%                 | 45.22                  | 78.847                 |
| 5.      | 40%                 | 42.74                  | 61.325                 |
| 6.      | 45%                 | 31.89                  | 50.854                 |
| 7.      | 50%                 | 29.34                  | 34.625                 |

**C. Impact test (Izode and charpy test)**

We are performing the test of those samples which gives the highest results of tensile and flexural strength of different temperature. At 165°C give the highest result of both Izod Charpy test, and 195°C give the lowest result of both Izod and Charpy test. Because in 165°C atomic structure is good and atomic bonding is proper that why in 165°C the result will be highest. And we are increasing the temperature.
than the result will decease. The results are as given below in Table IV.

Table IV: Heating at 195°C temperature

| Temperature (°C) | Volume fraction (%) | Izod impact test (kJ/m²) | Charpy impact test (kJ/m²) |
|------------------|----------------------|--------------------------|---------------------------|
| 165              | 33.7                 | 72.32                    | 79.38                     |
| 175              | 31.43                | 39.39                    | 44.42                     |
| 185              | 32.5                 | 24.81                    | 41.93                     |
| 195              | 29.75                | 16.30                    | 19.07                     |

VI. CONCLUSION

This study is about the development of green composites with poly lactic acid as matrix material and natural fibers as reinforcement. Woven Jute and sisal fibers are used as reinforcement which is purchased from the local market. Hand layup followed by compression molding technique has been used for the development of green composites. This research throws the light on study the effect of curing temperature on mechanical properties of PLA/natural jute and sisal fiber developed composites. Jute and sisal fiber reinforced PLA based composites were developed at different curing temperature with different fiber volume fraction ranging from 25%, 30%, 35%, 40%, 45% and 50% and their mechanical properties were investigated. Following are the conclusions drawn from the study as:

1. Successfully developed the fully biodegradable green composites with the help hand layup followed by the compression molding technique.
2. With the incorporation of jute and sisal fibers, tensile and flexural strength improved.
3. Tensile testing result shows that with increase in fiber volume fraction, tensile strength of all the composites increases, but then decreases thereafter attaining the maximum strength at 30% volume fraction. It has been found that for composites developed at 165°C Heating temperature gives the best results of tensile strength of 89.43 MPa as compared to other composites.
4. Flexural testing results exhibit that the maximum flexural strength of 126.81 MPa with 30% fiber volume fraction for the composites developed at 165°C. It has been also observed that with increase in fiber volume fraction, flexural strength of all the composites increases, but then decreases thereafter attaining the maximum strength at 30% volume fraction.
5. Morphological study of the fractured surfaces by mechanical testing as observed by the SEM as shown in Fig.8 analysis indicated that the good adhesion between fiber and matrix Heated at 165°C with 30% fiber volume fraction as compared to the 50% fiber volume fraction. Because of weak adhesion between fiber and matrix for the composites developed at 50% fiber volume fraction, complete fiber pulls out has been seen during SEM as shown in Fig.9

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AUTHORS PROFILE

**First Author**: Guravtar Singh Mann  
**Designation**: PhD Research Scholar at NIT Jalandhar, and Assistant Professor, Lovely Professional University, Jalandhar.  
**Department**: Industrial and production engineering  
**Qualification**: M.Tech Industrial and production engineering (Dr. B R Ambedkar NIT Jalandhar), B.Tech Mechanical (PTU, Jalandhar).  
**Research interests**: Green composites, Biomaterials.

**Second Author**: Dr. Lakhwinder Pal Singh  
**Designation**: Associate Professor  
**Department**: Industrial and production engineering  
**Qualification**: PhD Industrial Engineering (NIT Jalandhar), M.Tech Industrial and production engineering (Dr. B R Ambedkar NIT Jalandhar), B.Tech Industrial Engineering (REC Jalandhar, PTU)  
**Research interests**: Human Factors Engineering, Ergonomics, Occupational Health and Safety, Operations and Supply Chain Management

**Third Author**: Dr. Pramod Kumar  
**Designation**: Associate Professor  
**Department**: Mechanical engineering  
**Qualification**: PhD Industrial Engineering (NIT Jalandhar), M. Tech Mechanical (CIDM) (NIT Jamshedpur), B. Tech Mechanical (NIT Jamshedpur)  
**Research interests**: Renewable Energy, Composite Materials  
**Research interests**: Human Factors Engineering, Ergonomics, Occupational Health and Safety, Operations and Supply Chain Management