Asian Journal of Engineering and Applied Technology
ISSN 2249-068X Vol. 7 No. 2, 2018, pp.52-57

Mechanical and Biodegradation Behaviour of Jute/ Polylactic Acid Green Composites

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***Abstract* - Global warming, diminution of fossil fuels, escalating oil price's are the major reasons which forces the researchers to develop green products for the sustainable development. In this research work, green composites have been developed with jute fibers as reinforcement and poly lactic acid as matrix material using compression moulding technique. All composites were developed with maintaining the reinforcement as 30% fiber volume fraction. The influence of curing temperature with the range of 160°C, 170°C and 180°C was investigated for various mechanical properties of developed composites and degradation behaviour of developed composites were analysed using soil burial test. Results acquired from the tests specify that the tensile and flexural strength decreases with upsurge in curing temperature. Morphology study using scanning electron microscopy is further justified the findings obtained from mechanical tests. Biodegradation study was done on the all the three different composites under the soil burial conditions for 9 months and results indicate that composites developed at 160°C degrade faster as compared to others. This study also gives an optimum curing temperature for the development of jute/PLA composites.**

***Keywords:* Jute Fibers, Polylactic Acid (PLA), Curing Temperature, Mechanical Characterization, Green Composites, Soil Burial, Bio Degradation**

I. INTRODUCTION

Glass fiber reinforced plastics (GFRPs) have not only higher strength to weight ratio, outstanding durability, corrosion resistance, but also have electromagnetic neutrality, lox axial coefficient of thermal expansion. However, sometimes, the advantages become trouble because of the waste FRP remains in the ground during rejection. In addition to that the supply of raw material in FRP leads to depletion of fossil fuels in the near future, and during the recycling of FRP, toxic gasses lead's to the increase the effect of global warming also. Considering all these issues, nowadays researchers are focusing on the development of a new type of material with good mechanical characteristics and can be biodegradable in nature [1]. Green composites are biodegradable composites consists of natural fibers which acts as reinforcing material and natural polymer which acts as matrix material. One of the important characteristics of green composites that they can be designed and tailored to meet the desired requirements [2]. The exponential growth in the bio composites is indicative of their wider application in future. Flax, hemp, jute, sisal, bamboo and kenaf are the popular reinforcement materials because of their low density with high specific strength and stiffness [3], cost effective and readily available. Lot of researchers reported the innovative work related to the use of natural fibers to improve the mechanical properties of polymer composites [4]. Dhawan *et al.* [5] investigated the effect of natural fillers on the mechanical characteristics of GFRPs and found coconut coir shows better mechanical properties as compared to the other fillers in glass/epoxy composites. Dhawan *et al.* [6] discussed novel approach for the prediction of forces during the drilling of composite laminates using artificial neural network. Jai Inder *et al.* [7] studied the effect of curing temperature on mechanical properties of jute fibers/epoxy based green composites and found that tensile and flexural strength are maximum when curing is done at 100°C. Gomes [8] studied the effect of high concentration alkali treatment on mechanical properties of curaua fibers/corn starch based green composites and the results proved that the appropriate alkali treatment is the key technology for improving the mechanical properties of green composites. Later on lot many researchers have proposed various surface treatment techniques to improve the mechanical properties of polymer composites.

Goriparthi *et al.* [9] studied the effect of fiber surface treatments on mechanical and abrasive wear performance of polylactic acid/jute composites and results of thermo-gravimetric analysis showed a higher thermal stability for silane treated composites. T. Lee *et al.* [10] investigated the interfacial adhesion of ramie/acetylated epoxidized soybean oil based green composites and found that silane treated fibers improve the interfacial property. Jai Inder *et al.* [11] investigated the effect of alkali treatment on mechanical properties of jute/epoxy based green composites and found that 5% concentrated NaOH solution shows the best mechanical properties as compared to untreated jute/epoxy green composites. Martucci *et al.* [12] studied the biodegradation behaviour of gelatin and PLA. During the investigation, it has been found that 20% of weight loss upon 120 days of indoor soil burial.

This paper describes the research in which composites were developed with the use of woven jute fibers and polylactic acid (PLA). Mechanical properties were investigated for jute/PLA composites which were developed at 30% fiber volume fraction with curing temperature ranging from 160°C to 180°C. In addition to that, this research also leads to analyses the bio-degradation of developed composites under soil burial experiments. Biodegradation behaviour of jute/PLA composites were investigated using scanning electron microscopy analysis.

II. EXPERIMENTAL PROCEDURE

A. Materials and Processing

The PLA used in this work was commercial grade of 3052D in the granular form and purchased from Natur Tec India Pvt Ltd. Jute fibers used in the form of woven mat and purchased from local market. Properties of PLA and jute fiber are mentioned in the table 1 [13] and 2. Jute fibers were composed of alpha cellulose (60%), Hemicellulose (21%), Lignin (12%), Fats & waxes (0.4%), pectin (0.2%), Proteins/Nitrogenous matter etc. (0.80%) and Ash (0.7%) [1]. Figure 1 and 2 shows the PLA and Jute fiber used in the study

TABLE I PROPERTIES OF PLA

Properties	Values
Specific Gravity	1.24
Crystalline Melt Temp (°C)	145-160
Glass Transition Temp (°C)	55-60
Tensile Yield Strength (MPa)	62
Tensile Elongation (%)	3.5
Flexural Strength (MPa)	108
Flexural Modulus (MPa)	3600

TABLE II MECHANICAL PROPERTIES OF JUTE FIBER

Properties	Values
Density (g/cm^3)	1.3
Tensile strength (MPa)	393-773
E-modulus (GPa)	26.5
Elongation (%)	1.5-1.8

Fig. 1 Woven Jute Mat

Fig. 2 Optical View of PLA in granular form

Development of the composites was done with the help of compression molding technique. The complete set up comprises of sub components as metallic upper and lower die, heaters in the form of rods, temperature sensors and a control unit to ensure the required mold temperature of the die. The dimensions of the die are selected in such a way so that developed composite should have dimensions as 320 mm x 120 mm x 4 mm. In the first stage, three layers of woven jute mats were cut as per the required size of the die cavity and the calculated amount of PLA by weight were placed in the oven along with the fibers to remove the moisture content for 4 hours at 80°C. Meanwhile, metallic die was set to heat at the specified temperature, which was further controlled by the thermocouples and control units. To avoid the sticking of PLA with metallic die, Teflon sheet was used which act as a releasing agent.

Fiber content in terms of volume has been calculated as equation 1[8] .

$$V_f = 1 - \frac{W - W_f}{\rho_m V} \quad (1)$$

Where (Vf) = Fiber volume fraction, (Wf) is the fibers weight in composite, (W) is the weight of the developed composite, (V) is the volume of the developed composite and (ρm) is the density of matrix material.

To calculate the fiber volume fraction, weight of the fibers were calculated with the help of weighing machine. From the initial trails it has decided to keep three layers of jute fibers, for maintaining the 4 mm thickness of the composite. In this study, jute/PLA composites were developed with the fiber volume fraction as 30%. Firstly, tefloan sheet was placed in the cavity and on the top of that defined quantity of PLA granuals were spread over it. Distribution of PLA granuals should be uniform in the complete cavity. After that three layers of woven jute fibers were placed above the PLA layer. Again defined amount of PLA granuals were spread over the jute fibers and then cover the complete sandwitch like structure with tefloan sheet and finally closed the die with the help of an upper metallic die. The complete die which was at set temperature from the given range (160°C, 170°C and 180°C) was located under the press, and 4MPa of pressure was applied for 4 min, thereafter pressure was amplified to 6MPa for 2 min at constant temperature and kept the die under the load for 15 minutes curing. The composites were air cooled under the load. At 80°C temperature, the developed composite was removed out from the die and weight of laminate was recorded to get the final fiber volume fraction using equation no 1.

All composite were developed using same procedure as mentioned above, with 30% fiber volume fraction at different curing temperature range from (160°C, 170°C and 180°C). Curing temperature range was selected on the basis of trail runs and DSC test of pure PLA. The melting temperature of pure PLA was recorded as 160°C which was considered as the lower range and the upper range of the 180°C temperature was finalized based on the trials conducted and found fibers losses its strength because of burning at higher temperature of 190°C. Figure 3 shows the procedure of developing jute/PLA composites using compression molding process.

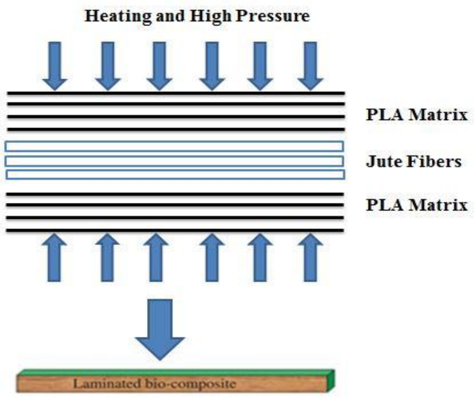


Fig. 3 Procedure for the development of green composites

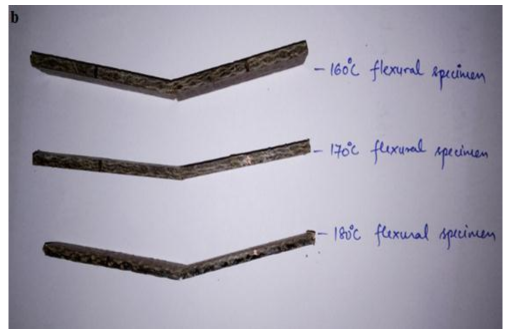
Laminates were further cut as per the ASTM standards for further testing. Figure 4 shows the developed jute fiber reinforced polylactic acid composites.

Fig. 4 Optical view of Jute/PLA composite

B. Tensile Test

The tensile test was carried on U.T.M Machine to find out the tensile strength and young’s modulus as per the ASTM standards D3039 [14]. Specimen dimensions were taken as 250 mm x 25 mm x 4 mm and were cut from the developed laminate with the help of cutter. The cross speed of universal testing machine while performing the tensile test was taken as 2mm/min. Figure 5a,b shows the ruptured specimens of tensile and flexural test respectively.

(a)



(b)

Fig. 5 Fractured tensile and flexural specimens

C. Flexural Test

3 point bend test was conducted on universal testing machine to find the flexural strength of developed jute/PLA composite as per ASTM standard D790-02 [15] with the specimen dimensions as 120 mm x 15 mm x 4 mm. Flexural strength of jute/PLA composites in comparison with neat PLA material was recorded and analysed.

D. Soil Burial Test

The soil degradation of samples prepared at 160°C, 170°C and 180°C curing temperature with 30% fiber volume fraction was buried in a fertile soil for up to 9 months. The sample size was kept 150 mm x 25 mm x 4mm. The samples were removed after every 5th week till the 40th week. The recovered samples were cleaned and dried in vacuum oven before analyze the degradation in terms of weight calculation. Percentage of weight loss has been calculated using equation 2.

$$\%WL = \frac{Wt-Wo}{Wo} \; X \; 100 \qquad (2)$$

Where Wo and Wt were the initial and final weight after dried.

E. Scanning Electron Microscopy

Morphological analysis was used to analyze the interfacial adhesion between the PLA and jute fibers and also to understand the failure mechanism of composite under the tensile load. The degradation behaviour of soil buried samples was also analysed using scanning electron microscopy.

III. RESULTS AND DISCUSSION

A. Mechanical Characterization of the Composites

1. Tensile Strength

Test results of tensile strength and youngs modulus for jute/PLA based composites are shown in figure 6 and 7. Results of tensile strength show that a maximum of 64.133 MPa was recorded at 160°C. It has also observed that with an increase in the temperature, tensile strength decreases. The decrease in the tensile strength at higher temperature is, because of fiber burn out.

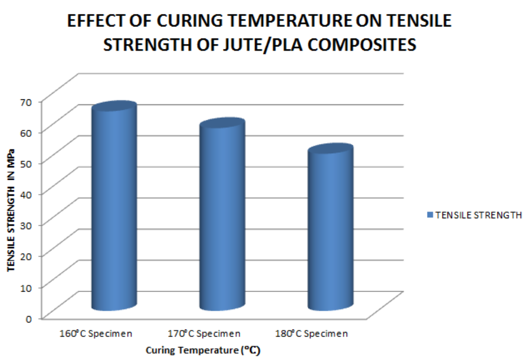


Fig. 6 Tensile strength of Jute/PLA composites

Figure 7 shows the influence of curing temperature on young's modulus of jute/PLA composites. It was observed that with increase in curing temperature, young's modulus decrease. Maximum value of young's modulus recorded as 3.393GPa 30% fiber volume fraction at 160°C as compared to other temperature ranges.

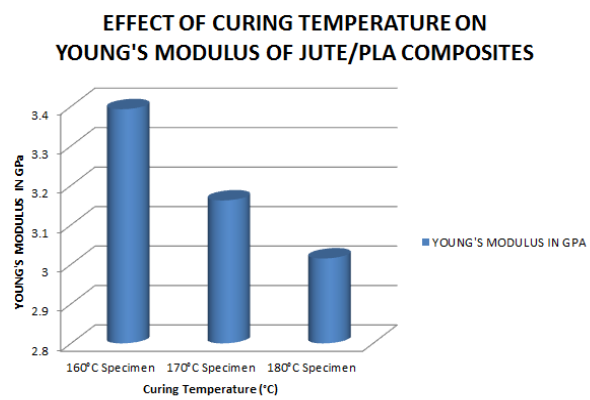


Fig. 7 Young's Modulus of jute/PLA composites

Figure 8a, b and 8c, shows the SEM micrographs of fractured tensile specimens of composite with 30% fiber volume fraction developed at 160°C, 170°C and 180°C curing temperature. From the figure, brittle failure of tensile specimens recorded, as some cracks were developed in the matrix. Figure 8a, b and c clearly show the comparison of interfacial bonding between fiber and matrix developed at different curing temperature. Poor interfacial bonding has been observed for the composites developed at 180°C curing temperature. The major reason is because of thermal degradation of fibers at the higher temperature.

Tensile strength of jute/PLA composites developed at 180°C was recorded as 50.495 MPa. From the SEM micrographs it was also observed that fibers were completely pulled out from the matrix during the tensile load. Fiber pull out was observed because of poor interfacial bonding between the fiber and the matrix.

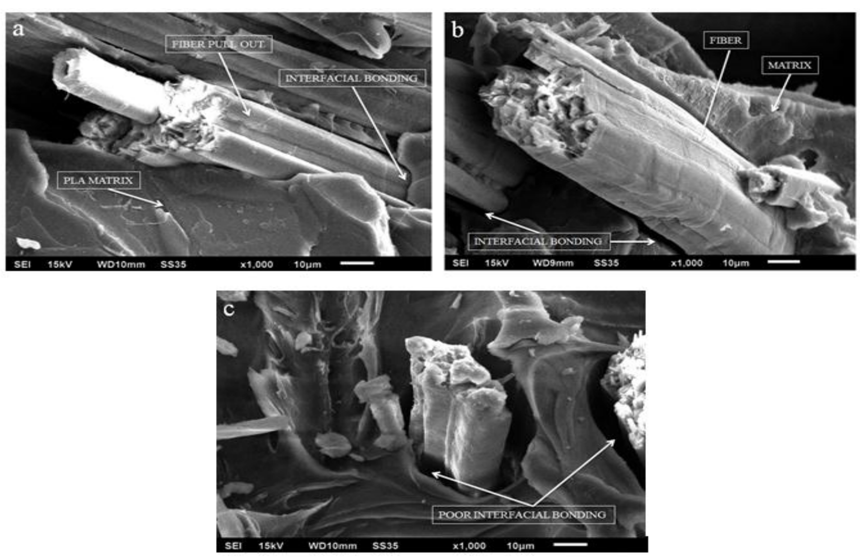


Fig. 8 SEM Images of tensile fractured specimens developed at 160°C, 170°C and 180°C curing temperature

2. Flexural Strength

Figure 9 shows the variation of flexural strength of developed composites at different curing temperatures. Results revel that flexural strength decreases with increase in curing temperature. Maximum flexural strength of 97.741 MPa was recorded at 160°C curing temperature and minimum flexural strength of 82.724 MPa at 180°C. Fiber burn out and fiber pull out are the main parameters effecting the flexural strength of jute/PLA composites.

Figure 10 shows the flexural modulus of jute/PLA composites. Maximum flexural modulus was recorded as 8.694 GPa at 30% fiber volume fraction and at 160°C curing temperature. Similar type of trend of flexural modulus and strength has been reported by other researcher also. The flexural strength is mainly depends upon percentage of reinforcement [6], type of surface treatment of fibers [2] and processing techniques used to develop these green composites.

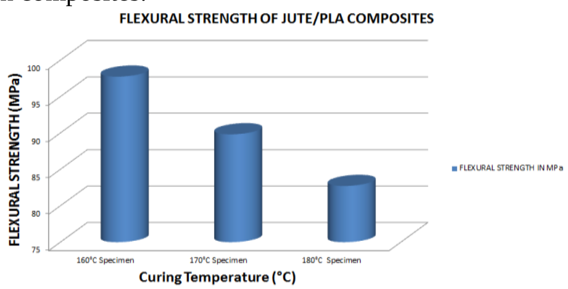


Fig. 9 Effect of curing temperature on the flexural strength of Jute/PLA composite

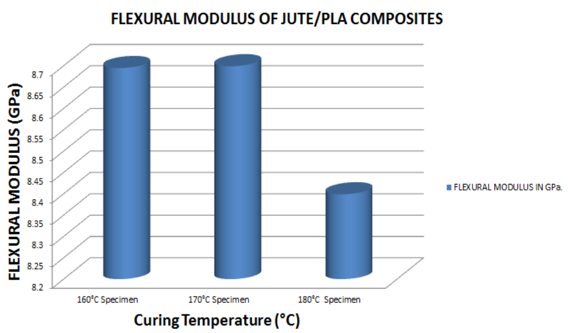


Fig. 10 Effect of curing temperature on flexural modulus of Jute/PLA composite

3. Soil Burial

Weight loss of specimens exposed to soil environment was considered as the indicator of degradation. Figure 11 shows the percentage degradation of jute/PLA composites developed with 30% fiber volume fraction with three temperature ranges. Selection of 30% fiber volume fraction was based on the composite showed maximum mechanical properties among other composites. The degradation period of the composites was accompanied by macroscopic changes of their physical appearance, as revealed by visual inspection and also shown in the figure 11.

Figure 12 a, b, c, d, e and f are the images of the physical appearance of the composites after degradation at 5th , 15th, 20th, 30th, 35th and 42th week of their incubation period. At the beginning of the incubation period, moisture was absorbed by all the specimens and during the 5th week of period, small cavities were developed in jute/PLA composites which were developed at 160°C, 170°C and 180°C. First crack was initiated and observed during the 5th week of incubation period. At the 15th week of degradation, maximum of 12% reduction in weight was observed in jute/PLA composites developed at 160°C curing temperature.

It was observed that the rate of degradation of composite developed at 160°C was increased drastically and at 42th week, 36% reduction in the weight was observed.

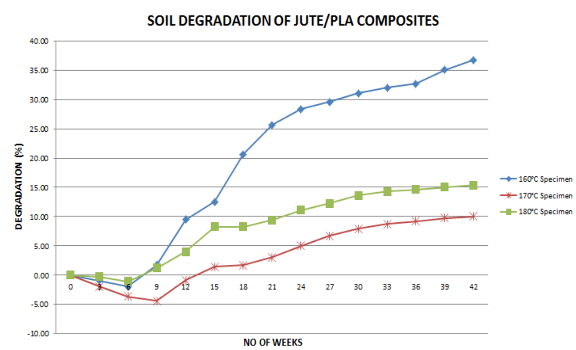


Fig. 11 Degradation of Jute/PLA composites

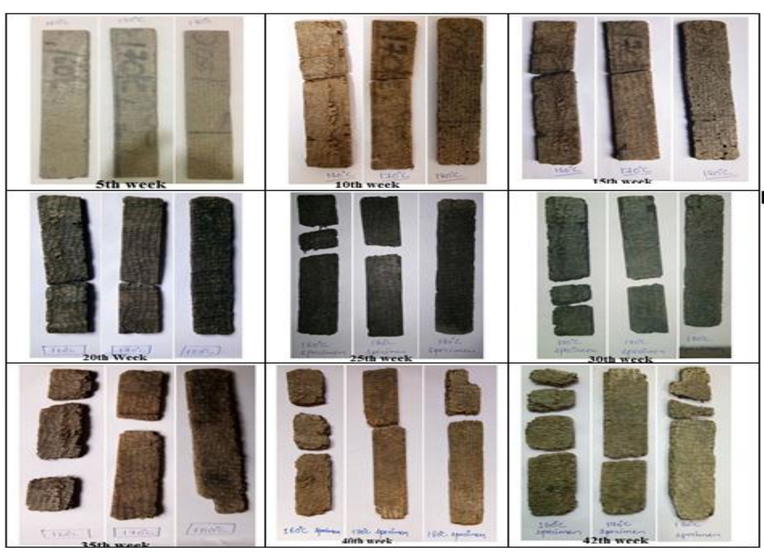


Fig. 12 Physical representation of degradation of jute/PLA composites in soil

Figure 13 shows the SEM images of degraded sample developed at 160°C curing temperature. It has been observed that small holes were generated in the matrix material and fibers were degraded first as compare to matrix.

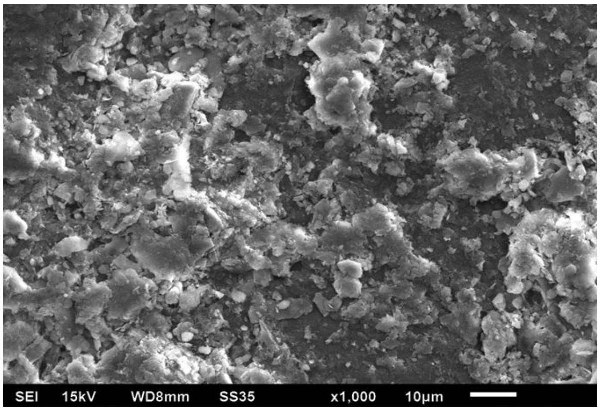


Fig. 13 SEM Images of degraded surfaces of jute/PLA composite

IV. CONCLUSION

The following conclusions have been drawn from this research

1. The result shows that mechanical properties were improved with incorporation of jute fiber as reinforcement in the PLA matrix.
2. Testing results show that with an upsurge in the curing temperature from 160°C to 180°C, tensile and flexural strength decreases. The highest value of tensile strength as 64.133 MPa was calculated at a 160°C curing temperature and a minimum of 50.495 MPa was recorded at 180°C. Decrease in tensile and flexural strength, is mainly due to the fiber burn out at higher temperatures which leads to the more fiber pull.
3. Maximum flexural strength of 97.741 MPa was obtained for jute/PLA composite developed at 160°C curing temperature.
4. Scanning electron microscopy of samples shows the brittle failure of jute/PLA composites and interfacial bonding between fiber and matrix decreases with upsurge in curing temperature from 160°C to 180°C. Decrease in the interfacial bonding may be because of thermal degradation of fibers at higher temperature.
5. The soil burial test show that the degradation of jute/PLA composite caused 36.72% weight loss after 42 weeks of soil burial.
6. It clearly signifies that green composites are the future materials for industry and are eco-friendly in nature.

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