Effect of Process Parameters on Water Absorption and Impact Strength of Hybrid PLA Composites

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Abstract In the present research work, an effort has been made to study the impact strength and water absorption ability, also the consequences, of the hybrid poly lactic acid composites prepared by using sisal and jute fibers through compression die process. The input process parameters such as compaction pressure (CP), molding temperature (MT), and curing time (CT) have been studied in the response of the observed outcomes through the application of Taguchi-based design of experimentation approach. Further, the surface morphology of the samples after impact and water absorption tests has been studied to understand the mechanism of failure. From the analysis of variance, it has been found that molding temperature (MT) acted as the most influential parameter affecting the observed properties at 95% confidence level.

Keywords Poly lactic acid · Water absorption · Impact strength · Taguchi · Sisal · Jute

1 Introduction

Bio-based polymers have recently been used as sustainable products to replace certain composites arising from petroleum products because of their environmental friendly prospects [1]. These types of materials can replace the non-degradable petroleum-based polymer with degradable biopolymer based on renewable resources, considerably reducing the emissions of hazardous waste and carbon dioxide (CO_2) [2]. Not

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only environmental issues but biocomposites can also to some extent solve the issue of the packaging industry once they are strengthened with distinct grades of polymers to improve their distinct characteristics and can be utilized in the automotive industry for sustainable construction [3]. Biocomposites when combined with natural/synthetic fibers/fillers have potential to substitute conventional polyolefin/glass fiber composites because they deliver diverse benefits such as recyclability, light weight, and low cost [4]. These composites have fascinating responsiveness in the sector as well as in academia, as they could allow thorough soil degradation through composting processes and make any toxic parts noticeable [5]. Moreover, to investigate, these fibers are combined with other fibers which are also known as hybrid fibers for improving their mechanical properties. These are the composites where both the matrix and the functional fibers are extracted from agricultural resources and are entirely degradable green composites and the other may or may not be biodegradable [6]. These composites have received substantial responsiveness because of their capability to give engineers new liberty to customize composites, thus achieving properties that cannot be achieved in binary systems consisting of a single type of filler/fiber spread in a matrix [7].

It also makes the use of expensive fillers more cost-effective by partly replacing them with inexpensive fibers. Though it is possible to combine diverse fillers into the hybrid system, it would be more advantageous to mix only two kinds of fillers. The performance characteristics of the subsequent composite can be significantly enhanced by a vigilant assortment of the reinforcing fillers [8]. The characteristics of hybrid composites are based on the single constituent, which balances the intrinsic advantages and disadvantages more constructively. The weaknesses of the one filler/fiber could be overlooked by the other filler's advantages [9]. The suitable material structure could achieve a hybrid composite with cost and efficiency balance [10]. Masoodi and Pillai [11] investigated the conduct of moisture and swelling in bio-based jute-epoxy composites and observed the rate of moisture diffusion in composites improved with a rise in the proportion of jute fiber to epoxy. Sanjay and Yogesha [12] studied the impact of hybridization and loading sequences on the various mechanical characteristics of composites of jute/kenaf/glass and epoxy and observed the enhancement of these properties. Chaudhari et al. [13] evaluated the various characteristics of jute, hemp, and flax composites based on epoxy and observed better mechanical characteristics of hybrid composites.

Attempts have been made in the present investigation to develop hybrid composite using jute and sisal fiber, whereas poly lactic acid (PLA) a highly versatile biodegradable polymers obtained from 100% renewable materials is used as resin [14]. The enhancement in mechanical properties like tensile strength and flexural strength has been examined experimentally using Taguchi method. The fiber matrix morphology of the interface region was explored using SEM analysis.

2 Materials and Methods

Poly lactic acid (PLA) was used as matrix material. It was supplied by Nature Works India. It is biodegradable material extracted from corn starch. The tensile strength of the smooth PLA was examined with universal testing machine with 1 mm/min cross velocity and 150 mm gage length. Woven jute and sisal were supplied by Chandra Parkash & Co. located in Jaipur India. Table 1 shows the mechanical properties of jute and Sisal.

2.1 *Fabrication of Composites*

The surface treatment of the fibers is done before the fabrication of the fibers which is also necessary for improved wettability (adhesion) between the fiber and the matrix as this process eliminates the hemicellulose layer leading to higher crystallinity in fibers. Then, the fabrication of composites was carried using compression molding method. The full setup was fitted with the molding machine for metal die, heating elements, thermocouples, and compression. The metal die was intended to create laminate of 4 mm thick. Four layers of jute fibers have to be used to get 4 mm dense laminate after pilot runs. Treated jute and sisal fibers were cut to the length of 120 mm and were kept in an oven for preheating at 90 °C temperatures for 3 h to remove the moisture. Compression molding die was cleaned and heated to the temperature provided. The Teflon sheet was set once the die temperature exceeded the appropriate temperature. First, the PLA granules were spread evenly into the cavity and afterward, the jute fibers were put in the cavity over the PLA granules and then, the other jute layer was positioned over the sisal fibers. Eventually, remaining PLA granules were scattered over the jute materials contained in the cavity. Finally, pressure is applied. The temperature will be maintained according to the requirement and the load will begin to apply.

Table 1 Properties of sisal and jute

Properties	Sisal	Jute
	Values	Values
Density (g/cm^3)	1.4–1.5	1.3–1.5
Length (mm)	1.5–120	900
Failure strain (%)	1.5–1.8	2.0–2.5
Tensile strength (MPa)	393–800	507–855
Stiffness/Young’s modulus (GPa)	10–55	9.4–28
Specific tensile strength (MPa/g cm^3)	300–610	362–610
Specific Young’s modulus (GPa/g cm^3)	7.1–39	6.7–20

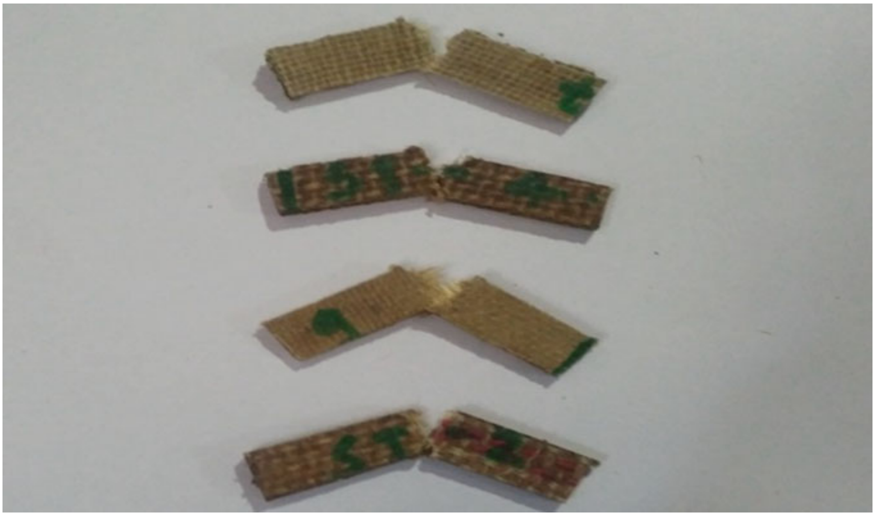


Fig. 1 Fractured Izod specimens

2.2 Impact Test

The Izod test was carried as per ASTM standard D256. The cutting of the samples was as per ASTM specifications 63.5 × 12.7 × 4 mm, and for Charpy test, the specimen samples with 127 lengths × 12.7 width × 4 mm were prepared. Figure 1 shows the fractured Izod specimens.

2.3 Water Absorption

In water absorption test, specimen was prepared 38 mm^2 with a 0.5 mm clearance. The weight of the specimens were measured first in air (WI) and then dipped in distilled water for a while of 24 ± 1 h. After completion of time, the specimens get out from the water and measured the weight ($W2$) within 2 min.

$$\text{Water absorption } (\%) = (W2 - W1)/W1 \times 100$$

$W1$ is the weight of specimen before dipped in distilled water and $W2$ is the weight of specimen after 24 h dipped in water.

3 Optimization Using Taguchi Methodology

Taguchi method provides information on the full set of possibilities for experiments that cover various applications. Moreover, this method reveals the outcome of different parameters on the mean and variance for process parameters. Moreover, experimental designs by Taguchi method helps to implement orthogonal arrays and organizes process parameters which affect the process, and helps in a recommendation of various levels and their variation for achieving the optimized results. Therefore, the number of experiments can be arranged with minimum means and lesser time without affecting the quality of the product [15–17]. For the implementation of the Taguchi method, the objective function is established along with the identification of process parameters with their levels as shown in Table 2. Secondly, the appropriate orthogonal array (OA) is selected for experimentation as shown in Table 3, and Table 4 shows the values obtained. At last confirmation, the experiment is done for the obtained data. The major aim of this technique is to produce high-quality products at minimum cost. Therefore, in the present research is to find the optimal set of parameters for maximum impact test and water absorption test during the fabrication of natural hybrid composites. Taguchi methodology organizes control factors and noise factors which affect the process quality. While noise is the uncontrollable factor, causes variations in the output, contributed due to the experimental environment like the ambient temperature, humidity, etc. Control variables are the most significant variables in determining the characteristics of the product [18]. Therefore, for the

Table 2 Levels of the variables used in the experiment

Levels	Variables		
	A:MT	B:CP	C:CT
1	160	250	30
2	185	300	60
3	210	350	90

Table 3 L_9 orthogonal array

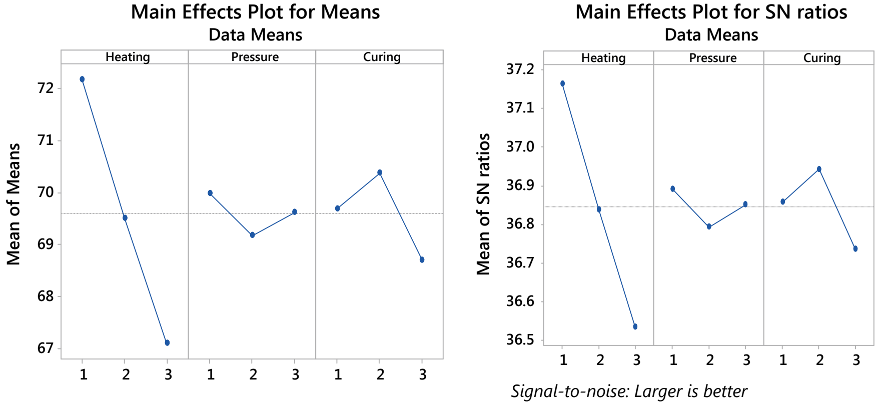
Exp. No.	MT	CP	CT	*S*/*N*
1	1	1	1	S/N_1
2	1	2	2	S/N_2
3	1	3	3	S/N_3
4	2	1	2	S/N_4
5	2	2	3	S/N_5
6	2	3	1	S/N_6
7	3	1	3	S/N_7
8	3	2	1	S/N_8
9	3	3	2	S/N_9

Table 4 Results of the L_9 orthogonal array experimental design for flexural strength

Trail	Process parameters			Impact strength (N/mm)				Water absorption (%)			
	MT	CP	CT								
	Trail conditions			*R*1	*R*2	*R*3	*S*/*N* (dB)	*R*1	*R*2	*R*3	*S*/*N* (dB)
1	1	1	1	72.23	71.44	73.33	37.185	22.61	23.25	24.25	27.31
2	1	2	2	72.62	73.51	71.22	37.199	26.43	25.21	26.52	28.31
3	1	3	3	71.42	72.33	71.41	37.112	25.81	24.13	24.33	27.86
4	2	1	2	70.24	72.91	70.12	37.032	23.04	22.04	24.04	27.23
5	2	2	3	67.52	68.84	67.33	36.636	23.34	25.52	22.66	27.51
6	2	3	1	68.62	69.32	70.72	36.844	24.25	23.29	24.28	27.57
7	3	1	3	66.22	66.11	67.23	36.458	20.64	20.43	19.64	26.11
8	3	2	1	68.33	67.14	66.12	36.545	19.81	18.13	19.21	25.58
9	3	3	2	67.12	67.32	68.35	36.598	19.04	20.04	20.64	25.96

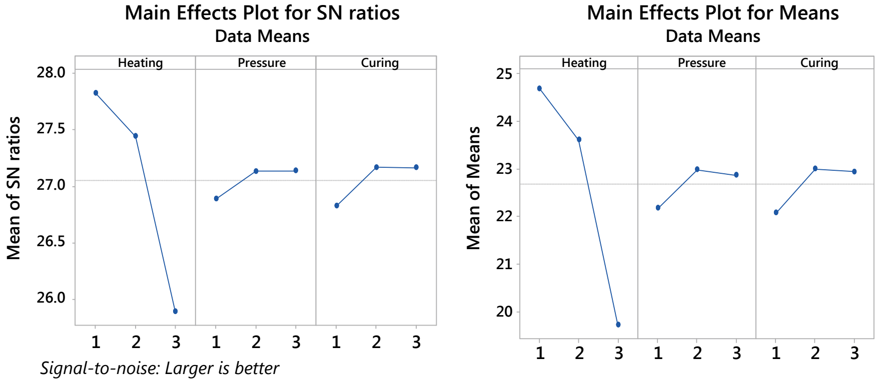
manufacturing of hybrid composite, the typical control factors include the heating temperature of the die. Taguchi method utilizes *S*/*N* ratio to express scatter around the target value. There are three possible categories for quality characteristics (1) smaller is better (2) nominal is better (3) larger is better. The main objective of the current research is to maximize the composite's impact strength and water adsorption behavior.

Therefore, larger quality features are chosen which is given as It can be observed from Fig. 2 that parameter values at levels A_1, B_2, and C_3 are the best choice in terms of the impact strength and for water absorption values at levels $A_{1,}$ B_3, and C_2 are the best in terms of optimum levels. These values are plotted in Fig. 3. Table 5 shows the mean values and Table 6 shows ANOVA values.



(a) (b)

Fig. 2 Mean (**a**) and *S*/*N* plot (**b**) for Izod strength



(a) (b)

Fig. 3 Mean (**a**) and *S*/*N* plot (**b**) for water absorption

Table 5 Response table for means

Level	Impact strength		CT	Water absorption		CT
	MT	CP		MT	CP	
1	24.69	22.18	22.08	72.17	69.98	69.69
2	23.61	22.98	23	69.51	69.18	70.38
3	19.73	22.87	22.94	67.1	69.62	68.71
Delta	4.96	0.81	0.92	5.06	0.8	1.67
Rank	1	3	2	1	3	2

$$^{S}/_{N} = -10\log\left\lfloor {}^{1}/_{R}\sum_{i=0}^{R} {}^{1}/_{y_i^2}\right\rfloor \tag{1}$$

$$\boldsymbol{\mu_{Ts} = \overline{A_1} + \overline{B_2} + \overline{C_1} - 2\overline{I_{TS}}} \tag{2}$$

where μ_{Ts} is mean value impact strength, $I_{\mathrm{IS}} = 71.67$ N/mm^2 (Table 6), and $A_1 + B_2 + C_1$ are average values of tensile strength. Therefore, $\mu_{\mathrm{Ts}} = 74.36 \pm 10.84$ N/mm^2. The confidence interval (CI) for the anticipated result can be calculated as:

$$\boldsymbol{CI = \sqrt{F_\alpha(1, f_e)V_e\left[\frac{1}{n_{eff}} + \frac{1}{R}\right]}} \tag{3}$$

where $\boldsymbol{F_\alpha}$ $(1,f_{\mathrm{e}})$ F is-ratio at the confidence level of $(1-\alpha)$ against DOF 1 and error DOF $f_{\mathrm{e},}$ v_{e} = error variance, η_{eff} is the effective number of repetitions.

$$\mathbf{n_{eff}} = \frac{\mathbf{1}}{\mathbf{1 + \{Total\ DOF\ in\ the\ estimation\ of\ mean\}}} \tag{4}$$

N = Total number of test outcomes (9 * 27) and R is sample size = 3 using the values, $v_{\mathrm{e}} = 18.6$, Table 4, Total DOF in estimation of mean is = 6 and $\eta_{\mathrm{eff}} = 3.87$, $F_{0.05}$ (1, 9) = 5.11 (The tabulated value). Therefore, from the above calculations for the interval at 95% is ±5.31 and the predicted optimum value for impact strength is $67.05 < \mu_{\mathrm{Ts}} < 81.67$ N/mm^2.

Similarly, by using Eqs. 2, 3, and 4, the calculations of the interval at 95% is ±01.84 which resulted in the predicted optimum value water absorption is $23.32 < \mu_{\mathrm{Ts}} < 44$.

4 Results and Discussion

It is observed that although all three parameters, i.e., heating, pressure, and curing play an integral role in the fabrication of hybrid fibers, the adhesion between the

Table 6 Analysis of variance for impact strength and water absorption

Source	DF	SS: I	SS: WA	V: I	V: WA	F-value: I	F-value: WA	P: I	P: WA
MT	2	115.458	122.232	57.729	61.1158	57.16	54.39	0	0
CP	2	2.891	3.423	1.4453	1.7114	1.43	1.52	0.263	0.242
CT	2	12.633	4.79	6.3165	2.3951	6.25	2.13	0.008	0.145
Error	20	20.201	22.474	1.01	1.1237				
Lack-of-fit	2	2.479	7.308	1.2397	3.654	1.26	4.34	0.308	0.029
Pure error	18	17.721	15.166	0.9845	0.8426				
Total	26	151.183	152.919						

where, DF, SS, V, F, P, I, and WA are the degree of freedom, a sum of a square, variance, probability, impact, and water absorption, respectively fibers is influenced mostly by the heating temperature of the die. When the fibers are fabricated at 160° temperature and 250 kN pressure is applied, minimum impact strength is observed as it can be seen SEM image 4a from the fiber pull out that fracture during the impact test is brittle which makes it more weak in case of impact strength because at this temperature, the PLA does not flow smoothly in all directions and there is no proper adhesion between the fibers and resin. When the temperature is increased to 185 °C and pressure of 300 kN is applied with curing at 60°. The flow of matrix material flows in all directions due to which better bonding takes place which influences the impact behavior of the material and maximum is obtained at these parameters but when the temperature is increased to 210 °C and pressure of 350 kN is applied at 90° curing, the internal fibers get burnt due to which the minimum values of impact strength are obtained, it can be observed from Fig. 4c that voids are seen in samples. These pores and voids can lead to early failure of these composites during loading. Another study which is related to outdoor applications of the natural fibers is water absorption tests. Figure 5 shows SEM images after moisture absorption tests. Initially, the water absorption does not affect the hydrophobic PLA matrix as shown in Fig. 5a–d. Before moisture absorption, the SEM images for hybrid composites display rougher fracture but while SEM images during moisture absorption show smoother fractured surface as shown in Fig. 5e which shows good adhesion between the surface as shown in Fig. 5f but when the temperature and pressure are increased to maximum 210 °C with 350 KN the fibers are burnt due to which poor fiber/matrix interface bonding. This indicates poor fiber/matrix interface

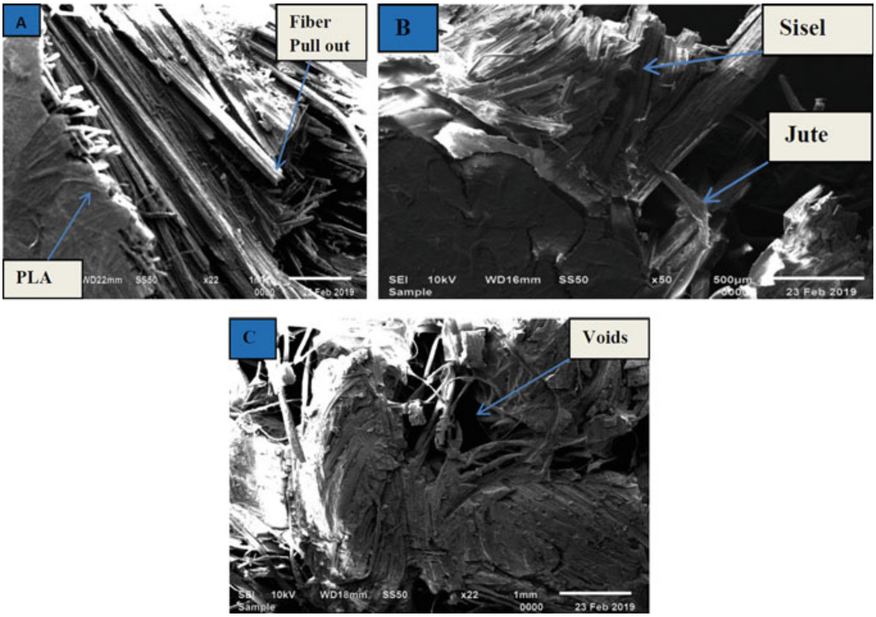


Fig. 4 SEM images of hybrid fibers after impact failure

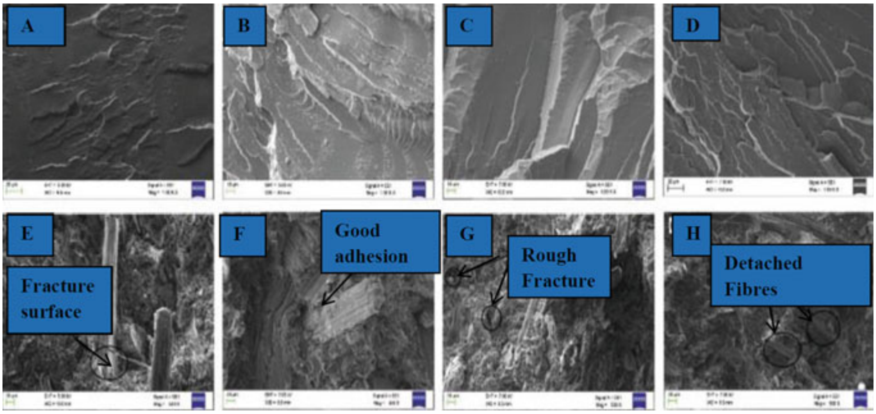


Fig. 5 Hybrid fiber SEM pictures after water absorption experiment

bonding there and therefore poor adhesion is observed as PLA comes out from the die due to high pressure and temperature and fibers are also detached easily during the impact loading as shown in SEM image in 5h. Therefore, optimum parameters where impact strength is maximum are at 185 °C temperature, the pressure of 300 kN, and curing at 60°.

5 Conclusion

Using the Taguchi technique, the impact strength and water absorption behavior of manufactured hybrid sisal and jute composites were explored. Three important parameters, that is the heating temperature, pressure, and curing temperature have been studied. It is possible to draw the following findings from the inquiry: To obtain maximum tensile and flexural strength, optimum heating temperature, pressure, and heating temperature were maintained. The maximum impact strength was observed at the temperature 185 °C, pressure of 300 kN, and curing at 60 °C.

Confirmation tests have been performed to confirm the optimum circumstances predicted. Estimate gain and confirmation gain values have been discovered close to each other. The moisture absorption was maximum when parameters were temperature 185 °C pressure of 300 kN, and curing at 60° when compared to dry samples because of the plasticization effect of diffused water molecules.

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