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EFFECT OF INJECTION PARAMETERS ON TENSILE AND FLEXURAL PROPERTIES OF GREEN COMPOSITES

Natural fibres are used to develop green composites due to their environmentally friendly nature, ease of availability, low cost, higher strength, as well as good thermal, acoustic, and insulating properties. In this study, jute fibre (JF) and sisal fibre (SF) were considered as reinforcement and a biodegradable polymer, namely polylactic acid (PLA), was selected to fabricate the composites by the injection moulding process. The fibres were chemically treated with sodium hydroxide (NaOH) at a concentration of 2% to improve the characteristics of the fibre. The effect of the injection moulding parameters like injection pressure (bars), injection speed (mm/s), and melting temperature (°C) on the tensile and flexural properties of the sisal fibre/polylactic acid (SF/PLA) and jute fibre/polylactic acid (JF/PLA) composites were investigated. Taguchi's L_9 orthogonal array was chosen for the design of experiments, and analysis of variance (ANOVA) was performed to find the significance and contribution of the selected parameters. The optimum levels from the main plots of both the tensile and flexural strength of the JF/PLA composite were found to be the injection pressure of 90 bars, injection speed of 60 mm/s, and injection temperature of 165°C. Meanwhile, the optimum level of tensile strength for SF/PLA-based composite was recorded as the injection pressure of 70 bars, injection speed of 40 mm/s, and temperature of 165°C. For the flexural strength, the optimum level was determined as the injection pressure of 90 bars, injection speed of 60 mm/s, and temperature of 165°C.

Keywords: **natural fibre, biodegradable polymer, green composites, injection moulding, mechanical properties**

INTRODUCTION

The large-scale commercial applications of composites started during World War II with marine applications for the military, but today, composite products are manufactured by different industries including sport, aerospace, automotive, packaging, and structural applications. Due to increased understanding of sustainable resources, significant efforts are being made to develop eco-friendly and biodegradable materials. Natural fibres are less expensive, low in density and readily available. Moreover, natural fibres are gaining more attention in a wide variety of industrial applications due to their multifunctional properties. The depletion of natural resources and increasing environmental awareness have resulted in the development of green composites for various applications. The increasing use of natural fibres in composites has resulted in a reduction of greenhouse emissions and carbon footprint.

Gholampour and Ozbakkaloglu [1] presented a review based on 322 studies published since 1978. The research discussed the growing need for eco-friendly materials in many applications and provided an up-to-date review on natural fibre and resin types as well as sources, processing techniques, physical and mechanical behaviours, applications, life-cycle assessment, and other properties. Besides the many advantages of green composites, there are certain drawbacks such as (a) poor compatibility between the reinforcing natural fibre and matrix, (b) the tendency of high moisture absorption, (c) poor fire resistance, (d) low durability and impact strength.

Young et al. [2] reported that bamboo fibre (BF) is a suitable candidate that can be used as a reinforcement for the development of green composites. BF has been studied extensively for use as fabric or structural reinforcement. The authors fabricated the composites by converting the bamboo strips into fibre bundles. The alkali treated BF was delignified adequately at 100°C for 12 hours. Morales et al. [3] used a BF/PLA composite for structural applications. Bamboo strips were glued together using a natural thermoplastic (PLA) to fulfil the biodegradability standards. The extraction of bamboo strips is inexpensive and also has no harmful effect on the environment. The characteristics of the composite were further studied under ageing conditions to confirm its structural uses. The developed material met the criteria for sufficient mechanical qualities and life cycle costs in the energy and automotive industries.

Ma and Joo [4] fabricated green composites based on jute fibre (JF) and PLA by the film-stacking process. The impact of the fibre content, processing temperature, and alkali treatment on the structural and mechanical characteristics was studied. The interfacial structure between JF and PLA was also studied using SEM. The JF/PLA composites with 15 wt.% fibre and produced at the temperature of 210°C had the best tensile properties, whereas the maximum flexural properties were reached using the production temperature of 220°C and 15 wt.% JF. The crystallinity of JF after alkali treatment shifted from Cellulose I to Cellulose II, which was observed by X-ray diffraction (XRD) analysis. The tensile modulus of JF and JF/PLA composites was enhanced by 29 and 76%, respectively. Arao et al. [5] investigated the properties of green composites based on JF and PLA.

A long fibre pellet was designed to achieve a high aspect ratio of the residual fibres after injection moulding. Shorter fibre pellets were produced by different screw configurations utilizing a twin-screw extruder. The fibre shape, dispersion condition, and fibre fracture surfaces were investigated after tensile testing. The short fibre pellet composites having a high compound intensity had the best mechanical properties. Compounding using a twin-screw extruder reduced the overall aspect ratio of the residual fibres, which considerably aided both the jute yarn dispersion and jute bundle decohesion into elementary fibres. In addition, high intensity mixing promoted fibre separation, resulting in efficient load transmission from the matrix to the fibre and enhanced the interfacial strength.

Samouh et al. [6] studied the mechanical, thermal, and dynamic mechanical properties of biocomposites. The biocomposites were extruded and then injection moulded with varying amounts of sisal fibres (SF) (5, 10, and 15 wt.%) and bio-sourced polymer PLA. The findings reveal that increasing the weight percentage of reinforcement enhances the characteristics of biocomposites. The degree of crystallinity of the matrix was enhanced from 47 to 61% due to an increase in the fibre content as the fibre (sisal) worked as a nucleating agent for PLA. Kassegn et al. [7] fabricated a fully biodegradable short SF/PLA composite using an injection moulding process. The weight fractions of the used fibre and matrix were 15 and 85 wt.%, respectively. A tensile strength of 44.23 MPa, strain at failure of 1.57%, and an elastic modulus of 4.32 GPa were recorded during the investigations. The properties attained under flexural loading were a flexural strength of 74.98 MPa, strain at failure of 2.7%, and a flexural modulus of 3.29 GPa. Radzi et al. [8] studied the mechanical properties of a kenaf fiber (KF)/PP composite by optimizing the injection moulding parameters. The optimum parameters to achieve better mechanical properties were 190ºC (injection temperature), 1300 bars (injection pressure), 1900 bars (holding pressure), and 20 cm^3/s (injection speed). Wang et al. [9] studied the injection parameters affecting the tensile properties of a PP fabric/PP composite. The optimum injection conditions were determined as 260ºC (barrel temperature), 127.6 MPa (injection pressure), 0.18 m/s (injection speed), and 60 s (holding time) to achieve the maximum tensile strength of 120 MPa. Sasimowski et al. [10] evaluated the mechanical properties of an injection moulded wheat bran/PBS biocomposite. The maximum tensile strength, tensile modulus, and elongation of the biocomposite were recorded as 31.96±±0.05 MPa, 830±3, and 26.6±7.4%. The authors noticed high densities in the injection-moulded samples as compared to the raw polymer owing to the high injection pressure and cooling rate. Cao et al. [11] studied the effect of injection parameters on the mechanical properties of a bagasse/PP-PCL composite. The optimum conditions to attain the maximum flexural strength were the mould temperature of 90ºC, cooling time 30 s, and melting temperature 165ºC. The authors reported that the properties deteriorated with the cylinder temperature because of thermal degradation of the bagasse fibre. Mohamed et al. [12] studied the effect of the injection temperature, injection speed, and screw speed on the properties of a flax/HDPE composite. It was reported that the maximum strength can be achieved by maintaining a high injection speed and screw speed with a low melting temperature.

Hussain et al. [13] studied the effect of chemical treatment on the properties of coconut fibre (CF)/HDPE composite by considering the fibre weight fraction and fibre length. The optimum levels for tensile and flexural strength were registered as (a) 40% fibre weight and 3 mm fibre length, (b) 30% fibre weight and 6 mm fibre length. Fracz et al. [14] investigated the effect of alkali treatment on the properties of a flax fiber (FF)/hemp fiber (HF)/PHBV composite. The authors noticed a reduction in the diameters of the flax and hemp fibres as a consequence of chemical treatment. It was also reported that it is difficult to finalize the chemical concentration for flax fibre. Balaji et al. [15] studied the properties of an injection-moulded sugarcane bagasse (SB)/cardanol resin biocomposite. The chemical treatment reduced the hydrophilic nature of fibre, which resulted in an increase in the surface area of the fibre. An NaOH treated bagasse fibre of a 10-mm length exhibited a peak tensile strength of 28 MPa. Manjula et al. [16] studied the properties of coir fibre after chemical treatment (NaOH) for 2 h. The treatment increased the stability of the fibre by removing the impurities on the fibre surface; as a result, enhancement of the tensile properties was noticed. Campos et al. [17] researched the mechanical properties of a starch cellulose acetate/PLA composite using different fibre contents and chemical treatment. A tensile strength and tensile modulus of 70 MPa and 6 GPa were obtained with 30 wt.% treated cellulose fibre.

It is clear from the above literature survey that the mechanical properties of JF/PLA and SF/PLA composites have not been thoroughly studied. The effect of the injection moulding process parameters on the properties of JF/PLA and SF/PLA composites has not extensively investigated either. Thus, in this study the effect of the injection parameters on the tensile and flexural properties of JF/PLA and SF/PLA composites was investigated. ANOVA was performed to find the significance and contribution of the injection moulding parameters.

METHODOLOGY

Materials used

Bio-resourced jute fiber (JF) and sisal fibre (SF) were selectedas the reinforcements and were supplied by Vruksha Composites Pvt. Ltd., India (Fig. 1). Both JF and SF are inexpensive, low in density, possess excellent strength and tensile modulus, pose minimal health risks and have renewable characteristics. PLA is a compostable bioplastic derived from plant sugar, often corn starch, cassava, and sugar cane, and was chosen as the matrix material. The PLA used in this study was supplied by Natur Tec India Pvt. Ltd. The melting temperature of the PLA is in the range of 145-180°C and its density is 1.24 g/cm^3. The sodium hydroxide was provided by Greenergy Lab Chemicals, India.

Fig. 1. Natural fibres (a) sisal fibre (SF) and (b) jute fibre (JF)

Alkaline treatment of fibre

Alkaline treatment, also known as mercerization, is a chemical treatment process where natural fibres are immersed in a known concentration of an aqueous sodium hydroxide (NaOH) solution for a specific duration. The alkaline treatment strengthens the fibres by removing a certain amount of hemicellulose, lignin, wax, and oils that often cover the surface of natural fibres [18]. The partially removed lignin, hemicellulose, and wax improve the bonding characteristics of the fibres. Alkaline treatment also increases the fibre density by removing non-cellulosic components that are less dense. During the alkaline treatment, the surface roughness of the fibre is improved, which increases the surface area and subsequently improves the wetting characteristics. In addition, the treatment modifies the crystallinity and unit cell structure of natural fibres [19]. The crystallinity of cellulose is increased owing to alkali treatment. Overall, the alkaline treatment of natural fibres enhances the mechanical performance of the resultant composite by improving the stress transfer between the fibre and matrix. After the alkali treatment, the fibres are washed using distilled water to neutralize the reaction. In this work, the treatment was done in two steps as shown in Figure 2.

Step 1 consisted in detergent washing of the fibres (10 gm of detergent in 21 l of water): (a) immersing the fibres in the detergent solution for 20 minutes, (b) washing the fibre with clean water, and (c) drying the fibre at room temperature. Step 2 involved NaOH treatment of the fibre (the 0.5 NaOH solution was prepared by adding 20 gm of NaOH pellets in 1 L of distilled water): (a) soaking the fibre in the solution for 4 hours, (b) washing the fibre with distilled water, (c) drying at room temperature, and (d) cutting the fibre to the desired length of 4 mm.

Fig. 2. Processing of chosen natural fibres

Composite fabrication and mechanical testing

The injection moulding process was employed to fabricate the SF/PLA and JF/PLA green composites. The composites were developed by using fibres 4 mm in length and a 10 wt.% fibre content. The specimens were fabricated according to (a) ASTM D638 for tensile testing and (b) ASTM D790 for flexural testing. The steps involved in fabricating the green composites were (a) heating the PLA pellets to remove moisture, (b) mixing the PLA pellets and natural fibre (10 gm of fibre was mixed with 100 gm of PLA pellets), (c) feeding the fibre-polymer mixture into the hopper, and (d) setting the injection moulding parameters according to the design of experiments. The injection moulding machine (Electronica E50 Servo, India) used to fabricate the composite specimens is shown in Figure 3. A universal testing machine (Unitek 9450, India) was used to perform uniaxial tensile testing and three-point bending testing. The test speed was maintained at 2 mm/min.

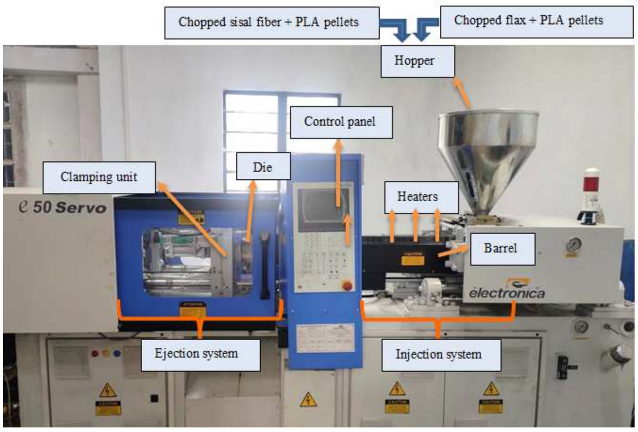

Fig. 3. Injection moulding machine used to fabricate the test specimens

RESULTS AND DISCUSSION

The Taguchi method was used for the purpose of designing the experiments. The design of the experiments (DOE) was generated using Minitab software. The main factors (injection pressure, injection speed, and injection temperature) and their levels, which affect the mechanical properties of an injection-moulded composite, were studied and selected. A low injection pressure and speed result in a poor surface finish, void formation, and warpage. On the other hand, a high injection pressure and speed may cause flash formation. Thus, the range of these two parameters was judiciously selected. The melting temperature of the chosen PLA is 165°C. Therefore, an injection temperature above the melting temperature of PLA was selected. A low injection temperature may cause inadequate bonding between the fibre and polymer and a poor surface finish. In contrast, a high injection temperature may cause voids to form in the moulded sample. The injection parameters, i.e. the injection pressure, injection speed, and injection temperature selected for this experiment are presented in Table 1. All the produced specimens and their properties are presented in Tables 2 and 3, respectively.

TABLE 1. **L_9 orthogonal array according to Taguchi method**

Run No.	Injection pressure [bars]	Injection speed [mm/s]	Injection temperature [°C]
1	70	40	165
2	70	50	175
3	70	60	185
4	90	40	175
5	90	50	185
6	90	60	165
7	110	40	185
8	110	50	165
9	110	60	175

TABLE 2. **Mechanical properties of JF/PLA composites**

Specimen No.	TS [MPa]	TM [GPa]	FS [MPa]	FM [GPa]
1	31.47	0.9	80.74	6.37
2	26.02	1.48	76.12	6.11
3	40.84	1.76	144.17	8.05
4	31.83	1.56	122.83	6.8
5	33.02	0.85	117.07	6.57
6	44.01	1.89	143.02	8.02
7	29.33	1.5	131.49	7.57
8	28.83	1.27	76.70	6.12
9	38.57	1.73	131.49	7.64

TABLE 3. **Mechanical properties of SF/PLA composites**

Specimen No.	TS [MPa]	TM [GPa]	FS [MPa]	FM [GPa]
1	45.56	4.16	144.18	8.34
2	37.91	2.3	117.07	7.89
3	17.40	1.58	96.89	7.42
4	29.52	2.69	131.49	7.64
5	10.82	0.98	94.58	7.46
6	25.22	3.46	190.31	8.78
7	24.99	2.92	95.96	7.96
8	43.88	4.01	163.21	8.6
9	26.44	2.14	140.14	7.79

Mechanical properties of JF/PLA composite

The mechanical properties of the JF/PLA composites were evaluated, which showed that the tensile strength is in the range of 26.02 to 44.01 MPa, whereas the tensile modulus is in the range of 0.85 to 1.89 GPa. The highest tensile strength was 44.01 MPa, which was obtained at an injection pressure of 90 bars, injection speed of 60 mm/s, and injection temperature of 165°C. The specimen having the highest modulus of 1.89 GPa was also obtained under the same injection conditions.

The flexural strength of the JF/PLA composites was in the range of 76.12 to 144.17 MPa. The highest flexural strength of 144.17 MPa was obtained at 70 bars, 60 mm/s, and 185°C. The flexural modulus was in the range of 6.11 to 8.05 GPa. The specimen had the highest flexural modulus of 8.05 GPa. It can be inferred that at the injection speed of 60 mm/sec, the tensile and flexural properties of the JF/PLA composite are the maximum.

The properties of the JF/PLA composite were analysed using ANOVA considering a 95% confidence level. The larger-is-better characteristic was selected to find the significance and contribution of the process parameters. The main effect plot of the tensile and flexural strength of the JF/PLA composite is presented in Figure 4. The optimal level for tensile strength from the means plot was found to be the injection pressure of 90 bars, injection speed of 60 mm/s, and temperature of 165°C. The sum of squares, F-value, p-value, and % contribution of the process parameters of the JF/PLA composite are shown in Tables 4 and 5. For the tensile strength, the p-values for the injection pressure, injection speed, and temperature were 0.074, 0.009, and 0.159. Among the process parameters, injection speed was significant as the p-value is less than 0.05. The injection speed contributed more (85.11%) as compared to the injection pressure and temperature. Both the injection pressure and temperature were found to be insignificant because of the higher statistical p-values. The fibre dispersion in the polymer was quite uniform, which improved the tensile properties of the JF/PLA composite at the higher injection speed. A further increment in the injection temperature degraded the tensile properties of the JF/PLA composite due to thermal degradation of the fibre. The optimum level for flexural strength was ascertained as the injection pressure of 90 bars, injection speed of 60 mm/s, and temperature of 165°C. Meanwhile, all the process parameters were found to be significant for the flexural strength of the JF/PLA composite. The p-values of the injection pressure, injection speed, and temperature were 0.017, 0.023, and 0.042. The p-values are less than 0.05 for all the process parameters. The injection pressure contributed more than 46%, followed by the injection speed and temperature as 34.06 and 18.45%. Figure 5 presents the contribution of the processing parameters of the JF/PLA composite. All the process parameters played a vital role to achieve superior flexural properties. The fibre-matrix interfacial bonding was good at the optimum level. Table 6 shows the model summary of the tensile and flexural strength of the JF/PLA composite. The R-sq. values for the tensile and flexural strength were quite high as 99.21 and 99.20%. This indicates that the data fit well the statistical model.

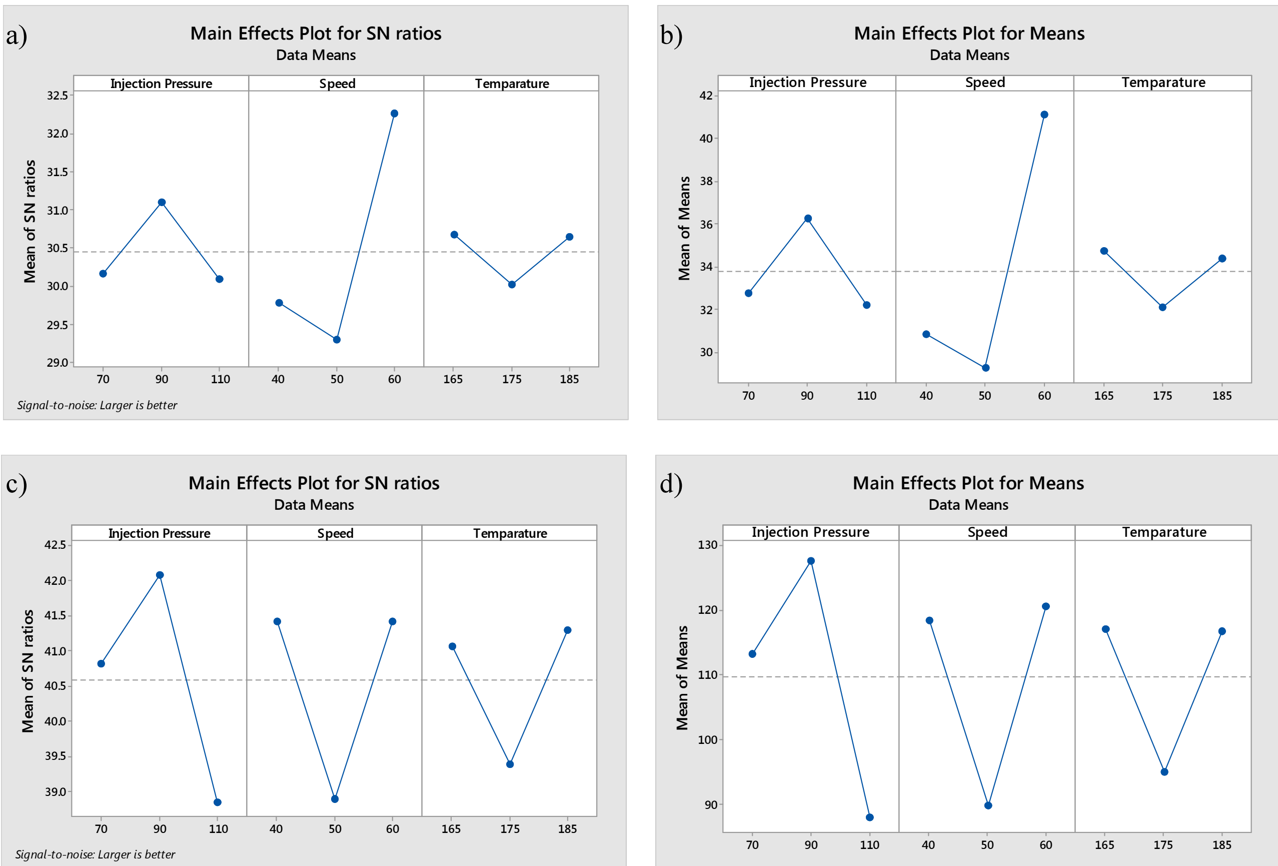

Fig. 4. Main effect plots of JF/PLA composite tensile strength (a-b) and flexural strength (c-d)

TABLE 4. **ANOVA of tensile strength for JF/PLA composite**

Source	D.F.	Adj. S.S.	Adj. M.S.	F-Value	p-Value	% Contribution
Pressure	2	28.953	14.477	12.59	0.074	9.92
Speed	2	248.276	124.138	107.93	0.009	85.11
Temperature	2	12.149	6.074	5.28	0.159	4.16
Error	2	2.300	1.150			

TABLE 5. **ANOVA of flexural strength for JF/PLA composite**

Source	D.F.	Adj. S.S.	Adj. M.S.	F-Value	p-Value	% Contribution
Pressure	2	2406.90	1203.45	58.27	0.017	46.68
Speed	2	1756.33	878.17	42.52	0.023	34.06
Temperature	2	951.27	475.64	23.03	0.042	18.45
Error	2	41.30	20.65			

TABLE 6. **Model summary of JF/PLA composite**

Properties	S	R-sq.	R-sq. (adj.)
TS	1.07246	99.21%	96.85%
TM	4.54439	99.20%	96.80%

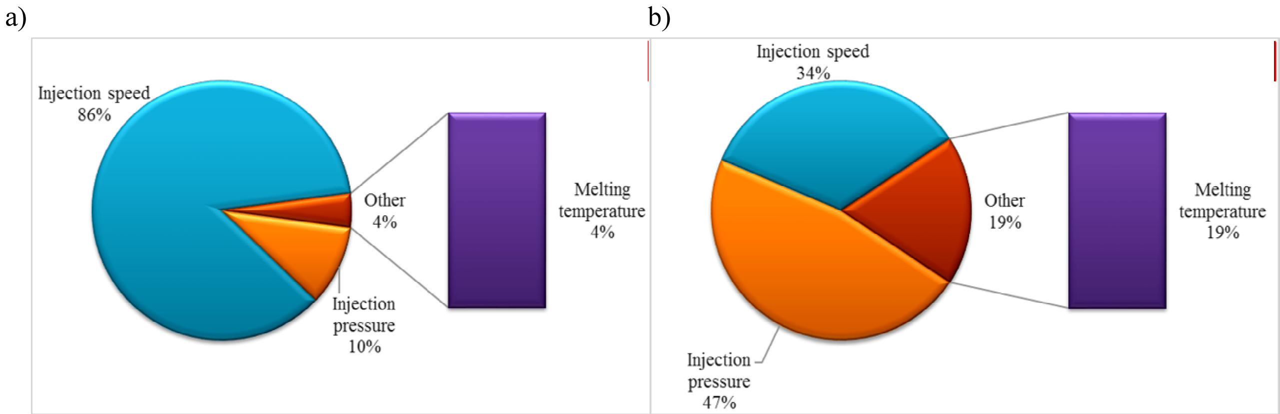

Fig. 5. Contribution of injection parameters on tensile (a) and flexural strength (b) of JF/PLA composite

Mechanical properties of SF/PLA composite

The maximum tensile strength and tensile modulus of the SF/PLA composite were obtained as 45.56 MPa and 4.16 GPa, whereas the maximum flexural strength and flexural modulus of the SF/PLA composite were 190.31 MPa and 8.78 GPa. It can be inferred that the tensile properties of the SF/PLA composite are the maximum at the injection temperature of 165°C. The properties deteriorated as the temperature is further increased. It implies that injection temperature is the most influencing factor among the chosen parameters. The tensile properties also deteriorated as the injection speed is increased to 60 mm/s and injection pressure to 110 bar. Similarly, it can be inferred that at the injection temperature of 165°C, the flexural properties of SF/PLA are the maximum. The properties declined as the temperature is further increased. This indicates that injection temperature is the most influencing factor affecting the flexural properties.

The flexural properties are maximum at the injection speed of 60 mm/s, injection pressure of 90 bars, and injection temperature of 165°C. Injection temperature is an important factor that decides the bonding between the fibre and matrix. Also, the melting of the matrix and the flow of the fibre-matrix mixture into the mould depends on the injection temperature. The melting and flow of the material increases with the temperature, which deteriorate the fibre-matrix interfacial bonding. Therefore, in the case of the SF/PLA composite, at the low injection temperature of 165°C the specimens showed good properties. The main plot curves of the tensile and flexural strength of the SF/PLA composite are shown in Figure 6. The optimum level for tensile strength from the main plots was the combination of injection pressure of 70 bar, injection speed of 40 mm/sec, and temperature of 165°C. For flexural strength, the optimum level was found to be the injection pressure of 90 bar, injection speed of 60 mm/sec, and temperature of 165°C.

The ANOVA results for the tensile and flexural strength of the SF/PLA composite are presented in Tables 7 and 8. The p-values of the injection pressure, injection speed, and melting temperature for the tensile strength of the SF/PLA composite were 0.044, 0.059, and 0.017. This shows that the injection pressure and melting temperature are the most influencing parameters to attain the maximum tensile strength.

The contribution of the melting temperature (60.45%) was the greatest, followed by the injection pressure (22.30%) and injection speed (16.21%). The fibre was well dispersed in the polymer because of the shearing effect of sisal fibre at the lower injection pressure of 70 bar, which resulted in achieving better tensile strength. The higher melting temperature caused thermal degradation of the sisal fibre. The melting temperature is the most influential parameter affecting the flexural strength as its p-value is less than 0.05. The melting temperature had a percentage contribution of 84.53%.

The contribution of the process parameters is graphically represented in Figure 7. The R-sq. values for the tensile and flexural strength of the SF/PLA composite are shown in Table 9.

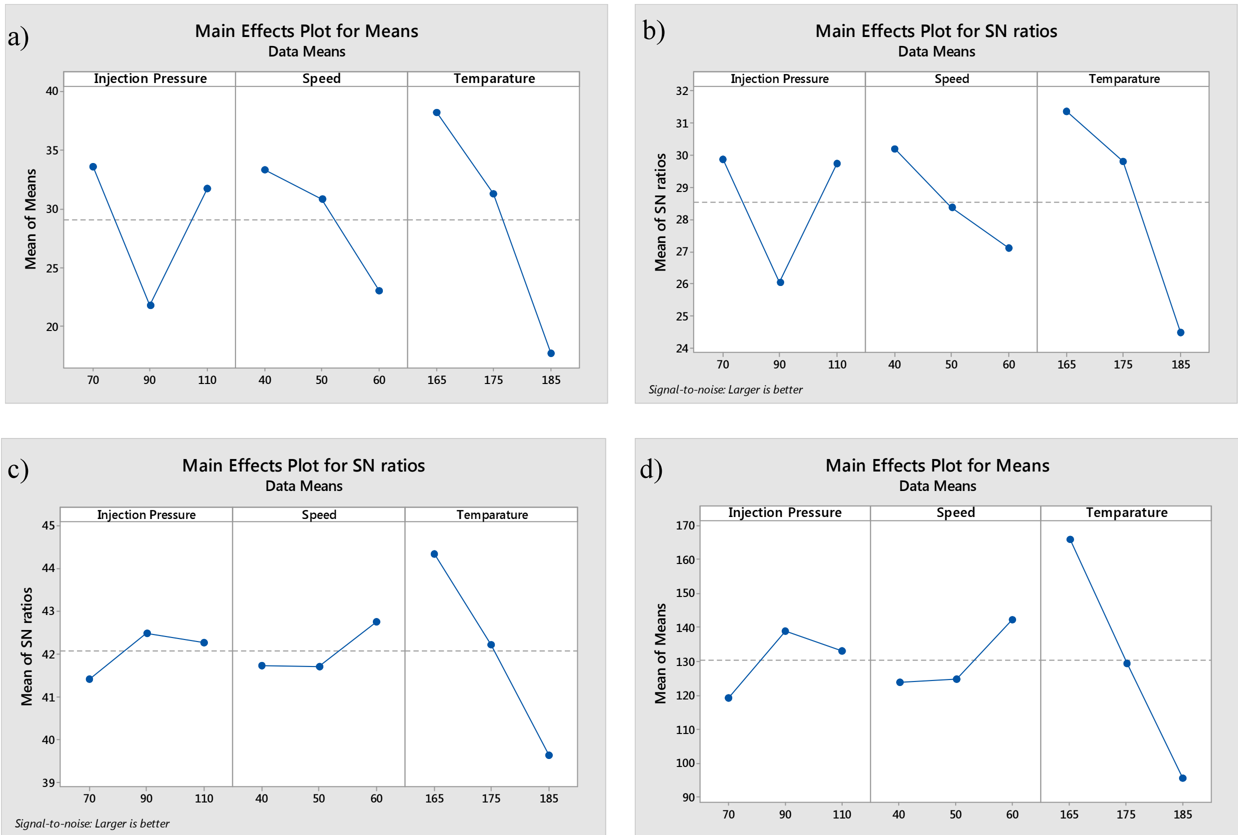

Fig. 6. Main effect plots of SF/PLA composite tensile strength (a-b) and flexural strength (c-d)

TABLE 7. **ANOVA of tensile strength for SF/PLA composite**

Source	D.F.	Adj. S.S.	Adj. M.S.	F-Value	p-Value	% Contribution
Pressure	2	240.31	120.154	21.86	0.044	22.30
Speed	2	174.65	87.326	15.89	0.059	16.21
Temperature	2	651.28	325.642	59.24	0.017	60.45
Error	2	10.99	5.497			

TABLE 8. **ANOVA of flexural strength for SF/PLA composite**

Source	D.F.	Adj. S.S.	Adj. M.S.	F-Value	p-Value	% Contribution
Pressure	2	597.58	298.79	6.00	0.143	6.85
Speed	2	652.02	326.01	6.55	0.133	7.47
Temperature	2	7372.23	3686.12	74.02	0.013	84.53
Error	2	99.60	49.80			

TABLE 9. **Model summary of SF/PLA composite**

Properties	S	R-sq.	R-sq. (adj.)
TS	2.345	99.00%	95.9%
TM	7.057	98.90%	95.4%

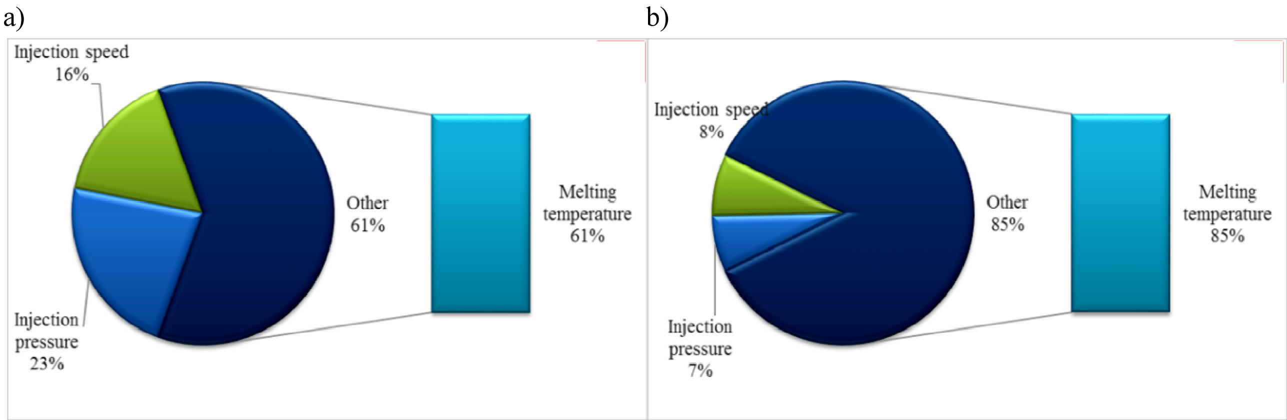

Fig. 7. Contribution of injection parameters on tensile (a) and flexural strength (b) of SF/PLA composite

CONCLUSIONS

The investigations of the mechanical properties (tensile and flexural) of different short natural fibre, i.e. jute and sisal-reinforced composites fabricated by injection moulding allow the following conclusions to be drawn:

1. The investigations revealed that at a 10 wt.% fibre content, the SF/PLA composite exhibited superior mechanical properties over the JF/PLA composites when the fibres are treated with a 2% NaOH solution to develop the composites.
2. The maximum tensile strength (TS), tensile modulus (TM), flexural strength (FS), and flexural modulus (FM) of the JF/PLA composite were 44.01 MPa, 1.89 GPa, 144.17 MPa, and 8.05 GPa, repectively.
3. The optimum levels from the main plots of both the tensile and flexural strength of the JF/PLA composite were ascertained as the injection pressure of 90 bars, injection speed of 60 mm/sec, and injection temperature of 165°C.
4. The maximum tensile strength (TS), tensile modulus (TM), flexural strength (FS), and flexural modulus (FM) of the SF/PLA composite were found to be 45.56 MPa, 4.16 GPa, 190.31 MPa, and 8.78 GPa, respectively.
5. In the case of the SF/PLA-based composite, the optimum level of tensile strength was recorded as the injection pressure of 70 bars, injection speed of 40 mm/s, and temperature of 165°C. For the flexural strength, the optimum level was the injection pressure of 90 bars, injection speed of 60 mm/s, and temperature of 165°C.

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