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Abstract

Recently, the use and application of natural fibers reinforced polymer composites are growing reasonably due to their light weight, low cost and good mechanical performance. Natural fibers are widely reinforced with thermoplastic polymers and the result is promising except the biocomposites are limited to low load bearing interior applications. In this study, short sisal fiber reinforced poly lactic acid matrix was used for the production of fully biodegradable material samples using injection molding machine. The weight fractions of the fiber and matrix used were 15wt% and 85wt%, respectively. The samples were tested for tensile and flexural tests. The results achieved for tensile strength were 44.23 MPa, with maximum strain of 1.57% and 4.32 GPa elasticmodulus. On the other hand, the results of flexural strength were74.98 MPa, with maximum strain of 2.7% and 3.29 GPa flexural modulus. The outputs of the study revealed that the developed composites have a good mechanical performance which can be used for various engineering applications such as packing of commodities, inner car door panel, door trim panels, and car dashboards.

Keywords: Bio-composites, injection molding, mechanical properties, natural fibers, PLA, sisal fiber.

1. Introduction

Natural fiber reinforced composites are being used as sustainable alternative to synthetic fibers reinforced composites in many engineering applications such as in automotive, construction and packaging industry [1]. This has been propelled by the global concern for the environment and hence the subsequent hunt for ecofriendly materials [2]. In a situation where the load-bearing Submitted on: December 05, 2018 Accepted on: January 30, 2019

capacity and dimensional stability under elevated conditions (both temperature and moisture) are of secondary importance, bio-composites can replace engineering application of plastic products. Currently, there are abundantly used thermoplastic polymer matrices. Of these, PLA is a bio-based polymer developed from renewable sources. It is usually produced from fermentable sugar and vegetable oils [3]. PLA can be reinforced by natural fibers to produce fully biodegradable and environmental friendly bio-composites.

Several studies have been undertaken on bio-composites, and the results indicate that natural fibers reinforced polymer composites can potentially substitute synthetic fibers reinforced polymer composites. According to [4], thermoplastic polymers reinforced with natural fibers have interesting mechanical performances. In comparison to common synthetic fiber reinforcement, the use of natural fiber reinforcement fibers offers significant potential for lightweight applications. Thermoplastic bio-composites are also found in a significant increase of the tensile strength and young's modulus. That is why for over the past decade, natural fiber composites have risen in their importance for technical interior parts [5-7]. In general, many studies on mechanical performances of natural fiber reinforced thermoplastics have been conducted and remarkable results have been drawn. Table 1 indicates the mechanical performances of some common thermoplastic matrices reinforced natural fiber composites.

 Table 1: Comparison of mechanical properties of some common thermoplastic polymer matrix

 reinforced natural fiber composites

	Tensile	Flexural	Elastic	
Composite Materials	strength (MPa)	strength (MPa)	modulus (GPa)	Reference
PP/20% curaua	31.2	50.27	2.72	[8]
PE/20% sisal fiber	25.5 ± 1.5	-	1.87 ± 0.09	[9]
PVC/20% coconut	67	104	5	[10]
PP/E-glass short fiber	31	36	-	[11]

Some mechanical properties of green composites along with their fabrication techniques are also shown in table 2.

Table 2: Mechanical properties of natural fibers reinforced PLA composites

Raw m	aterials	Compos	ition	Drogogging	Tensile	Young	Flexural	
Matrix	Fiber	Matrix (%)	Fiber (%)	technique	strength (MPa)	modulus (GPa)	strength (MPa)	Ref.

PLA	Kenaf	30	70	Hot pressing	62	6.3		[12]
PLA	Bamboo	50	50	Resin film infusion	29	0.98	104	[13]
PLA	Hemp	64.5	35.5	Compression molding	58	8.064		[14]
PLA	Flax	60	40	Compression molding	44	7.3		[15]
PLA	Wood pulp	80	20	Injection molding	65.8	3.82	93.43	[16]

The angle of orientation of the fiber and the principal material orientation with respect to the applied load is one of the important factors for determining mechanical performances of the composite material [17]. The factors affecting mechanical performances of natural fiber composites are also fiber selection including: type, harvest time, extraction method, aspect ratio, treatment and fiber content, matrix selection, interfacial strength, fiber dispersion, fiber orientation, composite manufacturing process and porosity [18, 19].

PLA has properties which are competitive to many commodity polymers such as high stiffness, clarity, surface shine, and ultraviolet stability [20-22]. The compatibility between the hydrophilic sisal fiber and hydrophobic PLA matrix is poor. PLA contains both a polar 'head' (hydrophilic) and a non-polar 'tail' (hydrophobic) [9, 21].

Natural fibers have shortcomings. Though the values are dependent of the fiber types, natural fibers' challenges are : thermal sensitivity, moisture absorption, poor interfacial strength, and poor fire resistance [5-7, 17-18, 21, 23-26]. Moisture content, temperature sensitivity and other mechanical properties of natural and synthetic fibers are values which need due considerations while designing composites for a particular application. It is used to compare and contrast natural fibers as reinforcement for a polymer matrix. It also indicates the influence of properties on the fiber either positively or negatively [18, 25-26].

Therefore, the study is intended to fabricate short sisal fiber reinforced PLA composites using injection molding by appropriate process parameters setting, and characterize the mechanical properties along with investigation of the application areas.

2. Materials and Experimental Details

2.1 Materials

The reinforcement material is sisal fiber which was obtained from north Ethiopian highlands. The fiber was extracted by mechanical means using hand tools. The density of the sisal fiber which was measured using pycnometer was approximately 1.4g/cm³. The fiber was with a length and aspect ratio of 5-6mm and 5.5:0.251respectively. Depending on the gage length, the young's modulus of sisal fiber varies between 9 and 19 GPa, and the tensile strength from 347 to 577 MPa [36]. However, due to the sisal plant cultivation place, environmental condition, aging and other factors, the tensile strength and other mechanical properties of the sisal fibers can be varied. The matrix used is biodegradable poly lactic acid (PLA) (IngeoTM biopolymer 3251D). The specific type of PLA used is 'PLA 3251D'. It was obtained from KU Leuven, Brugges campus store supplied by Nature Works LLC, USA. The density, tensile strength and young's modulus of neat PLA were 1.15 to 1.31 g/cm³, 21 to 29 MPa and 1.5 to 2.9 GPa respectively [33]. Both the fiber and matrix are bio-based materials which their composites are fully biodegradable. The composites of sisal fiber filled with PLA do not produce any toxic or noxious components to the environment during and after production [20]. The reinforcement sisal fiber and the matrix PLA are shown in figure 1 and figure 2, respectively.





Figure 2: Poly lactic acid (PLA 3251D)

2.2 Fiber Diameter Measurement

An experiment of scanning short sisal fibers (shown in figure 3) was conducted to perform diameter measurements. For fibers length of 35 to 40 mm, diameters (thicknesses) measurements have been taken at three various sections of a single fiber. This process was repeated for other fibers; the total diameters measurements conducted were 293. Matlab software package was used to measure fibers diameters. Figure 3 and figure 4 indicate the scanned fiber images using scanner and measurements of fiber diameters at different sections of the fibers, respectively.



Figure 3: Scanned sisal fibers

Figure 4: Diameters measurements

Fiber diameters were measured by magnifying the images, and drawing a line from one side to the opposite side as shown in figure 5. While measuring, the lignin portion of the fiber was excluded from the measurement. This is because of the fact that the fibers used for diameter measurements were technical fibers, not elementary fibers. Technical fiber contains lignin and may not be single fiber. Elementary fiber is cleaned and single fiber. When the image of a fiber is zoomed, two or more sub fibers may become visible; thus, measurements should be for the individual fibers. This is exhibited by the first image of figures 5 in which two fibers were shown when the single image was zoomed.



Figure 5: Fiber diameter measurements

From the diameter measurements, the minimum and maximum values were identified. The average diameter and standard deviation were also determined. The actual diameter of the fiber was calculated as the summation of the average diameter and standard deviation value. Hence, the diameter of sisal fibers used was 0.251 mm. The summary of the minimum, maximum and average diameters and standard deviation is shown in table 3.

No.	Description	Value of diameter (mm)
1	Minimum	0.076
2	Maximum	0.403
3	Average	0.197
4	Standard deviation	0.054
	Diameter	0.251

Table 3: Diameter of the sisal fiber

Values of measurements were found between the maximum and minimum diameters in which frequencies of measurements repeated in certain ranges were also determined. The frequencies of diameter measurements in ranges have been represented by the chart shown in figure 6. From diameter measurements taken, more values are found in interval of 0.175 to 0.2.



Figure 6: Frequencies of diameters of sisal fiber

2.3 Preparation of the Bio-composites

Unconditioned short sisal fiber reinforced PLA (IngeoTM biopolymer 3251D) was used in production of biodegradable specimens for testing. The sisal fibers were cut with average length of 5.5mm. The prepared sisal fiber was compounded with PLA matrix and stayed inside an autoclave for 24 hours at 80° C to remove moisture. The procedure shown in figure 7 below was followed for the production of sample specimens of composites.



Figure 7: Major steps involved in fabrication of bio-composite samples

The weight fraction of the sisal fiber was 15wt% with the remaining 85wt% PLA matrix. This weight fraction is the optimum value in which ease of samples' production of sisal fiber reinforced PLA composites is possible by ARBURG injection molding. The total weight of the fibers and matrix compounded was 4.5kg. Hence, 0.85*4.5kg = 3.825kg of PLA, and 0.15*4.5kg = 0.675kg of sisal fibers were used for compounding. Silicon-free mold release agent (C-150) was used for demolding the tensile bars. No packing pressure was used in the production of tensile bars due to difficulty of bars releasing from the mold. This is because of low shrinkage property of PLA. The ARBURG injection molding machine was used to produce the test specimens from the biodegradable composites. The process parameters setting of the injection molding machine during production of specimens are summarized in table 4.

No.	Process Parameters	Parameters Setting
1	Melting temperature (⁰ C)	190
2	Mold temperature (⁰ C)	18
3	Injection speed (cm^3/s)	15
4	Holding pressure (bar)	10
5	Screw rotation speed (m/min)	15
6	Packing pressure (bar)	0
7	Cooling water flow rate (l/min)	19
8	Cooling time (s)	40
9	Injection pressure (bar)	644
10	switching pressure (bar)	644

Table 4: Setting of injection molding process parameters

The injection molding machine used for compounding and production of sisal fiber reinforced PLA bio-composites is indicated in figure 8. The effects of process parameters on the alignment and orientation of the fibers by injection molding process were discussed. In injection molding of reinforced thermoplastics, the properties of the final product highly depend on the state of fiber orientation and alignment. Since the short fiber is used for developing the composite materials, the alignment of the fiber over the matrix is random. Fiber orientation is affected by mold temperature, melting temperature, flow rate, packing pressure and others though the degree of effect varies. The melt (injection) temperature affects the viscosity of the composite in which a higher melt temperature will lower the viscosity and reduce the residual stresses in the produced specimens. But, the effect of melt temperature on fiber orientation is not at large scale [38]. On the other hand, increasing the mold temperature delays heat transfer from the melt to the mold walls (reduction in the viscosity of melt), which increases the degree of transverse alignment of fibers at the core layer

during packing stage. The effect of mold temperature on fiber orientation is significant and more than the effect of melt temperature [39]. Packing pressure also increases the degree of transverse orientation of fibers at the core layer [40]. Packing pressure leads to rotation of fibers which in turn increases transverse stress on fibers. The transverse fiber stress brings transverse orientation of fibers. Flow rate of the melt is also a factor for fiber orientation. Increasing flow rate means increasing injection speed. As the injection speed increases, it leads to reduction of fiber loading content and then the fiber orientation gets decreased. Taking all these scenarios, the optimum values of process parameters settings were considered to have a uniform fiber orientation. But it is not meant that the fiber orientation was uniform throughout the sections of specimens due to uncertainties while production, shape of specimens and materials behavior. The orientation of fibers are considered as described probabilistically by the distribution function $f(\psi)$, where ψ is the angle which each fiber makes with the normal to the crack face [41].



Figure 8: The Injection Molding Machine

The composite specimens were produced as per the processing parameters shown in table 4 by using the injection molding machine. Then after, both tensile and flexural tests were held using ASTM standard Instron 5567 with 30kN and 1kN loading cells, respectively. Samples of tensile and flexural specimens are depicted in figure 9 and figure 10 respectively.



Figure 9: Tensile test specimen

Figure 10: Flexural test specimen

2.4 Mechanical Behaviors of Developed Bio-composites

Representative specimens were prepared from the developed bio-composites to investigate the mechanical properties of both tensile and flexural test samples. The average sizes of the specimens used for tensile tests were 190 mm length, 10.37 mm width and 4.13 mm thickness; and average dimensions of specimens used for three point bending tests were 60 mm span length, 10.251 mm width and 4.109 mm thickness. The tests were done using ASTM Instron 5567 with a load cell capacity of 30kN for tensile testing and 1kN for flexural testing. Both tests were performed with crosshead speed of 1mm/min at room temperature. The six specimens of each test were held separately and the mean values of the tests have been reported.

2.5 Characterization of Composite Materials

The developed bio-composites have been characterized with respect to tensile and flexural strengths, strains and modulus. The tests were done at normal temperature in an open air. But, the specimens were put inside an autoclave of 80° C temperature for 24 hours in order to release residual stress and remove moisture before testing has been done. Both tensile and flexural tests were performed using ASTM standard Instron 5567 with 30kN and 1kN loading cells respectively. The data were analyzed in terms of tensile and flexural strengths, and young's and flexural modulus. According to ISO 527-1, modulus of elasticity in tension (young's modulus) is calculated using ratio of stress to strain with strain values ranging from 0.05 to 0.25. Using this principle, the elastic modulus was determined.

3 Results and Discussion

Experimental tests were conducted to testify the applicability of the composite for engineering generic applications.

3.1 Tensile Test

For tensile specimens, the dimensional measurements have been taken before testing. Failure positions of specimens were also identified in terms of the positions of clamps (upper and lower BJET Vol. 1, No. 1, March, 2019: 20 - 36 ©EiT-M, Mekelle University

clamps) and positions of specimens (upper and lower positions in lined with positions of mold of the injection molding machine). The dimensional measurements and failure positions of specimens are summarized in table 5.

Specimens	Width (mm)	Thickness (mm)	Gauge length (mm)	Failure position in terms of clamps	Failure position in terms of specimen
Specimen 1	10.38	4.10	64	Near to upper	Near upper part
Specimen 2	10.31	4.10	64	Near to bottom	Near lower part
Specimen 3	10.32	4.07	64	Near to middle from bottom	Near to middle from lower part
Specimen 4	10.43	4.14	64	Middle	Middle
Specimen 5	10.42	4.21	64	Near to middle from upper	Near to middle from lower part
Specimen 6	10.38	4.14	64	Near to bottom	Near to lower part

Table 5: Dimensions of specimens and their breaking positions

Following the tests, the stresses and strains have been calculated. The stress-strain curves of each test have also been drawn as shown in figure 11. Specimens have various mechanical properties even though they were made from the same weight fractions of fibers and matrix. There are possible reasons for variation of mechanical properties. Some of the causes for variation are:in fiber orientation and alignment of test specimens, internal stresses developed while producing specimens, and dimensional variations of specimens. These factors always affect the mechanical performances of composites but the influences can be varied based on their value deviation.

The strains of tensile tests were directly measured using extensometer. Extensometer measures up to a strain value of 0.3% and then is removed from testing machine for equipment safety. The maximum strain of the specimens tested in this project was below 0.3% and hence all measurements of extensometer have been directly considered as values of strains. The tensile stresses were calculated using equation 1 given below. The initial cross-sectional areas of the specimens are used in strains calculation.



Figure 11: Stress-strain graphs of tensile specimens

Preloads recorded for each specimen test have been considered in calculating stresses and modulus. The tensile data were analyzed. Accordingly, mechanical properties drawn from the analysis are summarized in table 6:

Specimens	Tensile strength (MPa)	Strain at failure (%)	E-modulus (GPa)
Specimen 1	44.930	1.550	4.40
Specimen 2	44.490	1.590	4.36
Specimen 3	45.395	1.593	4.62
Specimen 4	44.748	1.703	3.75
Specimen 5	44.135	1.450	4.51
Specimen 6	41.650	1.530	4.30
Average value	44.225	1.570	4.32

Table 6: Summarized results of tensile tests

The average tensile strength of the specimens was approximately 44.23MPa. This result was achieved by 15wt% of sisal fiber reinforced 85wt% of PLA. However, the tensile strength can be enhanced by increasing the weight fraction of the sisal fibers [37]. The positions where the specimens were failed have also been identified. Hence, the failure positions of the specimens were different. As exhibited in figure 12, the positions of failure are located near to the upper, middle, and near to the lower clamps of the specimens. Some of the causes for different failure positions were: stress concentration (residual stress) produced during cooling, dimensional (thickness and width) variations during manufacturing, fiber distribution and orientation variations. The mechanical properties of the PLA/sisal fiber composite specimens can be improved by increasing

the volume fraction of the fiber, surface modification of fiber and monomer grafting of hydrophobic matrix.



Figures 12: Tensile test specimens after testing

3.2 Flexural Test

The flexural properties were measured using a three point bending test technique as per ASTM standard. For three points bending, flexural tests were carried out on six specimens. The physical measurements of the specimens were conducted as shown in table 7. The masses of specimens were also measured after putting them inside an autoclave of 80° C temperature for 24 hours.

Specimons	Width (mm)			Thickness (mm)				Span distance	Mass	
specimens	1	2	3	Av.	1	2	3	Av.	(mm)	(g)
Specimen 1	10.2	10.3	10.2	10.2	4.1	4.0	4.2	4.1	60	4.6
Specimen 2	10.2	10.2	10.3	10.2	4.2	4.1	4.1	4.1	60	4.6
Specimen 3	10.4	10.2	10.3	10.3	4.1	4.2	4.1	4.1	60	4.6
Specimen 4	10.3	10.2	10.2	10.2	4.1	4.1	4.1	4.1	60	4.7
Specimen 5	10.2	10.2	10.3	10.2	4.1	4.1	4.1	4.1	60	4.4
Specimen 6	10.3	10.2	10.2	10.2	4.1	4.1	4.1	4.1	60	4.3

 Table 7: Dimensions of specimens for flexural tests

The flexural strain of three point bending test was calculated using equation 3.

 $\varepsilon_{\rm f} = \frac{6 {\rm Dd}}{1^2}$ Equation (3)[42]

Where, \mathcal{E}_f = flexural strain, mm/mm; D = flexural extension (deflection), mm; d = thickness of the specimen, mm; L = span length, mm.

The flexural stresses of the specimens were also calculated using equation 4.

 $\sigma_{\rm f} = \frac{3 P L}{2 b d^2}$ Equation (4)[42]

Where, σ_f = flexural stress, MPa; P = applied load, N; b = with of the specimen, mm.

The stress-strain curves of the specimens are presented figure 13. The strengths and strains at failure of the flexural test specimens were significantly different. The mechanical properties of

specimen 6 was maximum but its weight was minimum when compared with other specimens as shown on table 7. There are possible reasons for the variation of mechanical performances of specimens. The internal (residual) stress developed during specimens production may be one of the causes. The injection and packing pressures causes fibers curvature in shearing forms. This fibers curvature could lead into formation of residual stress which in turn could result in premature failure of specimens. However, it needs further investigations.



Figure 13: Stress-strain curves of specimens

The flexural strength, strain at failure and flexural modulus of the three points bending specimens were calculated and the results are summarized in table 8.

Specimens	Flexural strength (MPa)	Strain at failure (%)	Flexural modulus (GPa)
Specimen 1	75.1956	2.7925	3.04
Specimen 2	73.4614	2.7367	3.28
Specimen 3	73.5248	2.7250	3.14
Specimen 4	72.5515	2.6369	3.35
Specimen 5	75.9310	2.5652	3.45
Specimen 6	79.2060	2.7632	3.47
Average value	74.9784	2.7033	3.29

Table 8: Summarized results of mechanical properties

Figure 14 indicates the three points bending testing specimens after breakage.

As PLA is a brittle polymer material, the failure type of the specimen under bending load was breakage.



Figures 14: Bending specimens after testing

From the results of experimental tests, the developed biodegradable composites can be used for engineering applications at its state of environmental friendly. According to study [14], the mechanical characteristics of natural fiber reinforced composites for interior parts in the automobile industry are tensile strength of 2 to 60 MPa and young modulus of 1.2 to 3.0 GPa. The experimental results of the developed composites indicate that the mechanical properties are increased when compared with literature values. Based on the evaluation of tensile and flexural performances, the composites can be used for applications such as packing of commodities, inner door panel, door trim panels, car dashboards, and furniture where high load carrying capacity is a secondary requirement.

4. Conclusion

In the present experimental investigation, sisal fibre reinforced PLA composites were fabricated using injection moulding. The tensile and flexural behaviour of the developed composites was analysed. And finally the following conclusions are drawn:

- I. A short sisal fibers reinforced PLA composite was developed and the mechanical behavior was analyzed. The results reveal that mechanical performances of the developed composite are at a good status. The composite is also a fully biodegradable and environmental friendly.
- II. The investigation has also indicated that 15wt% of short sisal fibers reinforced 85wt% of PLA composite materials exhibited the higher tensile and flexural strengths. Hence, the average tensile and flexural strengths of the samples were 44.23 MPa and 74.98 MPa, respectively. The tensile and flexural moduli were also found higher and the average values were 4.32 GPa and 3.29 GPa, respectively.

- III. As presented in (I) and (II), the mechanical behavior of the developed composites was higher and fully biodegradable. So, the composites can be used for various engineering applications as they fit to the requirements.
- IV. The application areas in which the developed composites can be used are packing, interior parts of automotive and aerospace industries, furniture products, and others.

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