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Use of Statistical Techniques to Characterize Bio-Composites Made from Sisal Fibres and Bio-Resin from Banana Peel

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Abstract
The purpose of this study was to use statistical techniques to characterise bio-composites made from sisal fibres and bio-resin from raw banana peel. The fibres were treated with sodium hydroxide, combined with a bio-resin made from banana peel, and then a bio-composite material was developed. The effect of the fibre volume fraction, glycerine and bio-resin mass on the bio-composite's tensile and compressive properties was investigated using universal rotatable design and multiple regression. The paired T-test conducted exhibited a significant improvement in the mechanical properties of the treated fibres. Sisal bio-composite showed a tensile strength of 5.2 MPa with an adjusted R^2 value of 0.91, Young's modulus of 11.99 MPa (adjusted R^2 of 0.92), percentage elongation of 1.77% (adjusted R^2 of 0.95), and compressive strength of 2.94 MPa (adjusted R^2 of 0.90). The bio-composite could be compared to a commercial composite and solid wood boards, and hence it is an alternative to non-renewable, non-biodegradable petroleum and solid wood products for partition, ceiling and notice board applications.

Key words: *sisal fibres, banana peel, bio-resin, bio-composites, universal rotatable design.*

Introduction

In a bid to fulfill the United Nations Sustainable Development Goals for 2030, global awareness of eco-friendly materials is undergoing a paradigm shift. Promoting food security and sustainable agriculture is among the key development goals. Agriculture is a global source of renewable materials for bio-composites, and sisal is among the prospective reinforcing materials, accounting for 2% of the world's plant fibre production [1-2]. Unfortunately there is a global declining trend in sisal fibre production due to competition from non-biodegradable and non-renewable polypropylene fibres, hence a need for revitalisation of the sisal industry. Bio-polymers reinforced with bio-fibres can produce novel bio-composites to substitute glass fibre reinforced composites in various applications [3].

According to [4], 6mm random sisal fibre reinforced polypropylene composites yielded a tensile strength of (29-33.84) MPa for a fibre volume fraction of (10-30)%. Similarly the tensile strength of sisal fibre reinforced low density polyethylene composites increased from (10.8-14.7) MPa for the same range of the fibre volume fraction, which was different from the tensile strength of sisal fibre reinforced cornstarch bio-composite laminate – determined as 5.4 MPa [5]. In addition, untreated sisal fibre reinforced epoxy composite exhibited a tensile strength of 45.1 MPa, as compared to the composite with treated fibres at 49.9 MPa. Random sisal fibre reinforced polypropylene composites yielded a Young's modulus of (605-940) MPa for a fibre volume fraction of 10% to 30%. Furthermore the Young's modulus of sisal fibre reinforced low density polyethylene composites increased from (324-781) MPa for the same range of the fibre volume fraction [4]. An untreated sisal fibre reinforced epoxy composite exhibited a Young's modulus of 4.87 MPa, as compared to that with treated fibres – 6.5 MPa [6]. Randomly arranged sisal fibre reinforced polypropylene composites exhibited a percentage elongation of (8-8.5) % for a fibre volume fraction of (10-30) %. However, the percentage elongation of sisal fibre reinforced low density polyethylene composites decreased from (27-7) % for the same range of the fibre volume fraction [4]. This was also in agreement with [6], where the percentage elongation decreased from 1.1% for an untreated sisal fibre reinforced epoxy composite to 1.0% a composite with treated fibres. According to [5], the compressive strength was determined as 42 MPa, while the standard required for commercial medium density fibreboards is 10 MPa [7-8]. Various studies on sisal reinforced bio-composites have been done, but none on bio-composites using a bio-resin made from banana peel and sisal fibres, hence the justification for this study. Consideration was also made with regard to the alkali treatment of sisal fibres, resulting in improved mechanical properties [9-10]. In addition, raw banana peel has been used for the development of bio-resin with potential for use in bio-composite development, which was a motivation for this study [11].

Materials and methods

Raw banana peel bio-resin as a binder

Bio-resin from raw banana peel was developed and characterised by the Uganda Industrial Research Institute with an optimum viscosity of 242.01 MPa.s and density (0.95 g/cm^3) at 25 °C.

Sisal fibres as reinforcement

Sisal fibres purchased from Kamwe Business Uganda Limited were treated with 4% W/V sodium hydroxide from Desbro Uganda Limited according to the procedure by (Varughese et al 2004), and then cooled, rinsed, dried and weighed. Treated and untreated fibres with a gauge length of 300 mm were weighed on a Mettler Toledo Balance (Model THB – 300 CAP) (Roycobel Group, Deerlijk, Belgium) to determine the weight and linear density according to ASTM D1577 – 2001. Tensile properties were determined according to ASTM D3822M – 2014 using a Universal Tensile Tester (Model TP2730) (Roycobel Group, Deerlijk, Belgium).

Table 1. Relationship between factors and levels relating to sisal bio-composites.

Factors		Levels				
		-α	Low	Medium	High	+α
Coding		-1.682	-1	0	1	1.682
Fibre volume fraction, %	X_1	20	30	40	50	60
Bio-resin mass, grams	X_2	60	76	100	124	140
Glycerin mass, grams	X_3	0	2	4.3	6	8

Table 3. Paired T-Test analysis for treated and untreated sisal fibres.

Sisal fibre responses	Mean difference	P-values
Linear density, tex – treated fibres	23.82	0.0001
Linear density, tex – untreated fibres	46	
Linear density difference	-22.18	
Elongation, % – treated fibres	1.03	0.001
Elongation, % – untreated fibres	1.85	
Elongation difference	-0.82	
Tenacity, MPa – treated fibres	217.13	0.001
Tenacity, MPa – untreated fibres	108	
Tenacity difference	109.13	
Young's Modulus, MPa – treated fibres	5.076	0.0001
Young's Modulus, MPa – untreated	4.000	
Young's Modulus difference	1.076	

Table 2. Universal rotatable experimental design setup for sisal bio-composite.

Run	X_1	X_2	X_3
1	0	0	-1.682
2	1	-1	1
3	0	0	0
4	0	-1.682	0
5	-1	-1	1
6	1.682	1.682	1.682
7	1	1	-1
8	0	1.682	0
9	0	0	0
10	1	1	1
11	-1	1	-1
12	-1.682	-1.682	-1.682
13	0	0	0
14	-1.682	0	0
15	0	0	0
16	0	0	1.682
17	-1	1	1
18	1.682	0	0
19	1	-1	-1
20	-1	-1	1

Experimental design of sisal bio-composites

The universal rotatable design using the following 3 factors: fibre volume fraction (X_1), bio-resin, (X_2) and glycerine (X_3) at 5 levels was used as shown in ***Table 1***.

Modeling of the influence of the factors above and sensitivity analysis of bio-composite properties were made using multiple regression analysis. Various second order polynomial regression equations were used to generate optimal data values and sample points with predicted response values closest to the optimal solution. These values were evaluated alongside alternative solutions closest to the optimum settings to determine if any were adequate. Standardisation of the data was effected using the backward elimination regression technique to remove insignificant terms during regression to maintain a hierarchical model at each step. Analysis of variance (P-values) and variance inflation factors were checked and verified for significance and multi-colinearity, respectively, to ensure model accuracy. Analysis of variance described the overall variance accounted for in the model used to test the null (H_0 = 0) and alternative ($H_1 \neq 0$) hypotheses. The null hypothesis showed that the expected values of regression coefficients are equal to each other and that they equal zero, indicating insignificance within the model. The alternative hypothesis proposed that the expected values of regression coefficients are not equal to each other nor equal to zero, hence significant in the model. P-values of linear, curvilinear and interaction effects were compared to the alpha value ($P < 0.05$) to determine whether they were lower; hence rejection of the null hypothesis and acceptance of the alternative hypothesis. Normal probability plots of standard residuals were employed to determine whether the data points were distributed normally.

Regression ***Equation (1)*** was used to fit the regression models, where Y = yield, b_0 = constant, b_1 to b_{20} = coefficients, and X_1 to X_5 = factors.

$$Y = b_0 + b_1x_1 + \ldots b_5x_5 + b_6x_1x_1 + \\ + \ldots b_{10}x_5x_5 + b_{11}x_1x_2 + \ldots b_{20}x_4x_5 \quad (1)$$

Table 2 is the universal rotatable design setup for the sisal bio-composite.

Production and characterisation of sisal bio-composite

Bio-composites were developed from sisal fibres as well as bio-resin from banana peel and glycerine using the hand layup method with male and female metallic moulds of dimensions of (310 x 310 x 10) mm. The sisal bio-composites were cured in an oven at 100 °C for 15 minutes. The bio-composites were cut according to ASTM D2584 – 2010, and tensile and compressive properties were determined using a universal material tester (Model WP 310) (Gunt, Hamburg, Germany) based on ASTM D638–2014 and ASTM D695 – 2010, respectively. The effect of the fibre volume fraction, bio-resin and glycerine mass on bio-composite properties was modelled using multiple regression.

Results and discussion

Pre-treatment of sisal fibres with sodium hydroxide

The linear density of untreated sisal fibres was 46 tex, as compared to 23.82 tex for treated fibres, as shown in ***Table 3***. The tenacity of treated sisal fibres was higher (217.13 MPa) than that of untreated fibres (108 MPa). A Young's modulus of 4 GPa was obtained for untreated fibres, while 5.1 GPa was for treated fibres. Untreated sisal fibres exhibited a higher percentage elongation at break of 1.85% against 1.03% for the treated fibres. Since P–values of all fibre property mean responses were less than the Alpha (α) value of 0.05, there was a significant improvement in the properties of the treated fibres. Alkali treatment of natural fibres has been used on most fibres successfully, reducing the diameter and increasing the fibre aspect ratio and tenacity [10].

Development and characterisation of sisal bio-composite

Tensile strength

Multiple regression analysis produced a tensile strength model (Y_T), with an

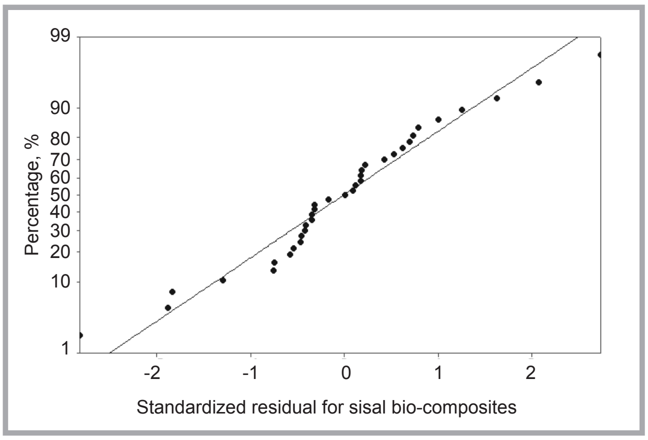

***Figure 1.** Normal probability plot for tensile strength of sisal bio-composites.*

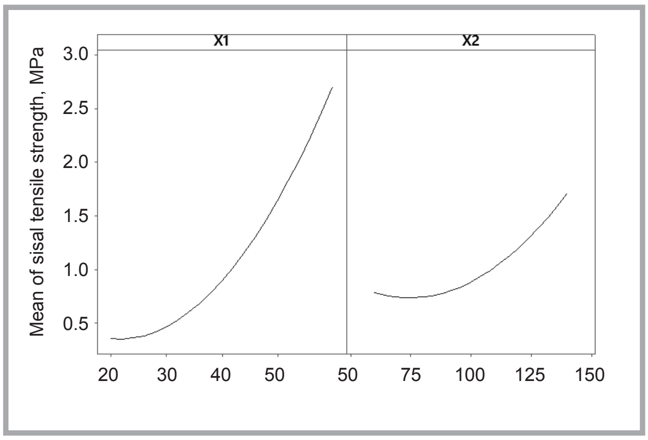

***Figure 2.** Main effects plot for tensile strength of sisal bio-composites.*

adjusted R^2 value of 0.91 and P-value of 0.0001, hence it was significant.

$$Y_T(\text{Sisal}) = 8.38 - 0.2107X_1 + 0.091X_2 + 0.001604X_1X_1 + 0.000231X_2X_2 \quad (2)$$

The sample data of 40 points was a precise estimate of the strength of the model. The normal probability plot of residuals for the tensile strength in ***Figure 1*** showed that the data points were distributed normally.

Figure 2 supported the tensile strength model by showing that squaring the fibre volume fraction (X_1^2) resulted in a significant increase in tensile strength (Y_T), as compared to squaring bio-resin mass (X_2^2). This could be due to the increase in cellulosic fibres, which have and contribute higher strength and stiffness to the bio-polymer, hence improving tensile properties of the resultant bio-composite.

Table 4 is a summary of the analysis of variance (ANOVA), percentage factor contributions and variation inflation factors (VIF) generated by the model. The model had all the P-values for individual factors as well as curvilinear and interaction effects less than the Alpha (α) value of 0.05, hence significant in the model. There was no multi-colinearity among the terms in the model, as evidenced by variance inflation factors close to 1 and below 5. Percentage contributions of various factors were obtained, with the fibre volume fraction contributing the highest percentage of 38.1% to the model and bio-resin mass – 8.1%.

Curvilinear and interaction effects revealed that interacting the fibre volume fraction with bio-resin mass ($X_1 * X_2$) contributed a higher percentage of 7.5% to the regression model compared to other effects. Curvilinear effects of squaring the fibre volume fraction (X_1) and bio-resin mass (X_2) contributed 3.5% and 1.6% to the model, respectively.

The regression model developed was used to design a prediction and optimisation report for the tensile strength of the bio-composites developed, shown in ***Table 5***. Optimal settings yielded a maximum tensile strength of 5.23 MPa for sisal-reinforced bio-composites. Furthermore at a 95% confidence interval (CI), a tensile strength range was determined between 4.59 MPa and 5.99 MPa, and a predicted interval (PI) of 4.39 MPa to 6.18 MPa. This tensile strength was close to that obtained by [5] – 5.4 MPa, but this is above the standard value required for medium density fibreboards – 1.15 MPa [8].

Percentage elongation

The normal probability plot of residuals for percentage elongation in ***Figure 3*** shows that the data points were distributed normally. Multiple regression analysis of the percentage elongation (Y_E) exhibited a model with an adjusted R^2 of 0.95 and P-values less than 0.05, hence a significant model with no multi-colinearity, shown in ***Table 6***.

$$Y_E(\text{Sisal}) = -1.063 + 0.057797X_1 + 0.00799X_2 + 0.1889X_3 - 0.00402X_1X_3 \quad (3)$$

***Table 4.** ANOVA, factor contributions and VIF for the tensile strength of sisal bio-composites.*

Source	ANOVA, P-values	Factor contributions, %	VIF
Regression	0.000	95.03	
X_1	0.000	38.11	1.00
X_2	0.000	8.11	1.01
X_1*X_1	0.000	3.48	1.05
X_2*X_2	0.020	1.59	1.05
X_1*X_2	0.000	7.49	1.00
Bio-composite type (sisal)	0.000	34.1	1.00
Error		7.12	
Lack-of-fit	0.000	6.91	
Pure error		0.21	
Total		100	

***Table 5.** Optimum settings for sisal bio-composite tensile strength. **Note:** X_1 – fibre volume fraction (%), X_2 – bio-resin weight (Grams).*

Goal: maximised tensile strength		Solution: optimum settings	
Predicted viscosity, MPa	5.23	X_1	60
95% Confidence interval, MPa	(4.59, 5.99)	X_2	140
95% Predicted interval, MPa	(4.39, 6.18)		

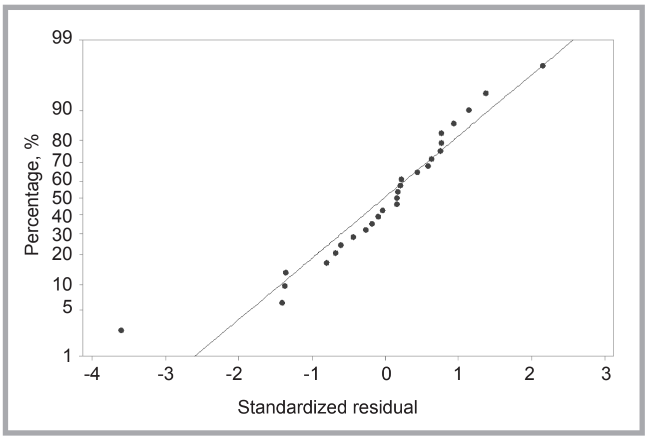

***Figure 3.** Normal probability plot for percentage elongation of sisal bio-composites.*

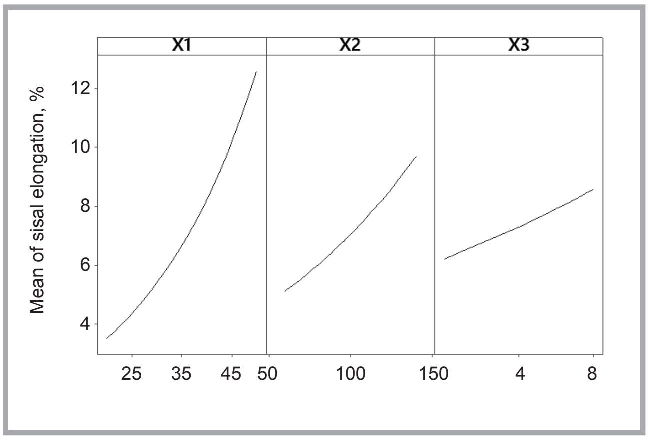

***Figure 4.** Main effects plot for percentage elongation of sisal bio-composites.*

***Table 6.** ANOVA, % contributions and VIF for elongation of sisal bio-composites.*

Source	ANOVA, P-value	Factor contributions, %	VIF
Regression	0.000	95.03	
X_1	0.000	77.08	1.23
X_2	0.000	11.09	1.21
X_3	0.002	2.77	1.08
$X_1 * X_3$	0.002	2.13	1.17
Bio-composite type (sisal)	0.007	1.06	1.00
Error		3.77	
Lack-of-fit	0.273	3.52	
Pure error		0.26	
Total		100	

***Table 7.** Optimum settings for sisal bio-composite percentage elongation.*

Goal: minimized elongation, %		Solution: optimum settings	
Predicted viscosit, %	1.77	X_1	20
95% Confidence interval, %	(1.47, 2.12)	X_2	60
95% Predicted interval, %	(1.28, 2.44)	X_3	0

***Table 8.** ANOVA, factor contributions and VIF for Young's modulus of bio-composites*

Source	ANOVA, P-value	Factor contributions, %	VIF
Regression	0.000	93.11	
X_1	0.000	45.13	1.06
X_2	0.003	4.37	1.06
$X_1 * X_1$	0.000	4.41	1.01
Bio-composite type (sisal)	0.000	39.02	1.02
Error		6.89	
Lack-of-fit	0.611	5.80	
Pure error		1.09	
Total		100	

***Table 9.** Prediction and optimisation for Young's modulus of sisal bio-composite.*

Goal: maximised Young's modulus, MPa		Solution: optimum settings	
Predicted viscosity	11.99	X_1	60
95% Confidence interval	(10.72, 13.25)	X_2	140
95% Predicted interval	(410.22.39, 13.75)		

Table 6 is a summary of ANOVA, percentage factor contributions and VIF statistics generated by the percentage elongation model. The elongation model generated showed that an increase in the fibre volume fraction (X_1), bio-resin (X_2) and glycerine mass (X_3) contributed positively to the percentage elongation yield, and was in agreement with ***Figure 4***.

The percentage contributions of various factors were obtained, with the fibre volume fraction (X_1) contributing the highest percentage of 77.8% to the model. The bio-resin mass (X_2) and glycerine mass (X_3) contributed 11.9% and 2.8% to the model, respectively. Interaction effects revealed that interacting the fibre volume fraction with bio-resin mass ($X_1 * X_3$) contributed a percentage of 2.13% to the regression model. Optimum settings of the fibre volume fraction (20%) and bio-resin mass (60 grams) yielded a minimum elongation of 1.77% for sisal-reinforced bio-composites. Furthermore at a 95% confidence interval (CI), a percentage elongation range between 1.47% and 2.12% was determined and a predicted interval (PI) of 1.28% to 2.45%. The percentage elongation was close to that for sisal fibre reinforced composites obtained by [4], ranging between 4% and 10%. The optimum settings obtained for elongation optimisation are shown in ***Table 7***.

Young's modulus

The normal probability plot of residuals for Young's modulus in ***Figure 5*** shows that the data points were distributed normally. Multiple regression analysis of the Young's modulus (Y_Y) produced models with an adjusted R^2 value of 0.92 and P-value of 0.0001, hence the model was significant.

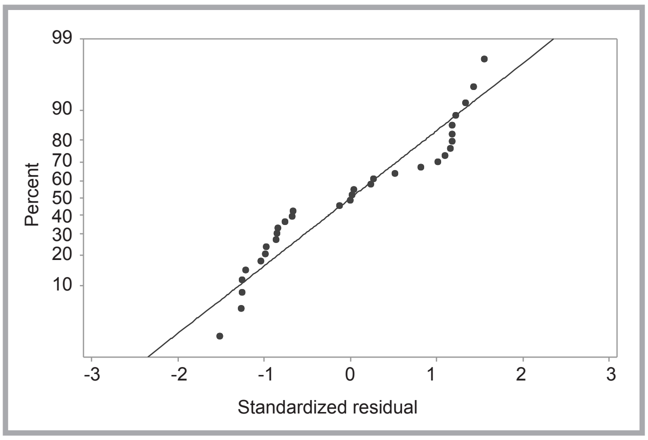

Figure 5. Normal probability plot for Young's modulus of sisal bio-composites.

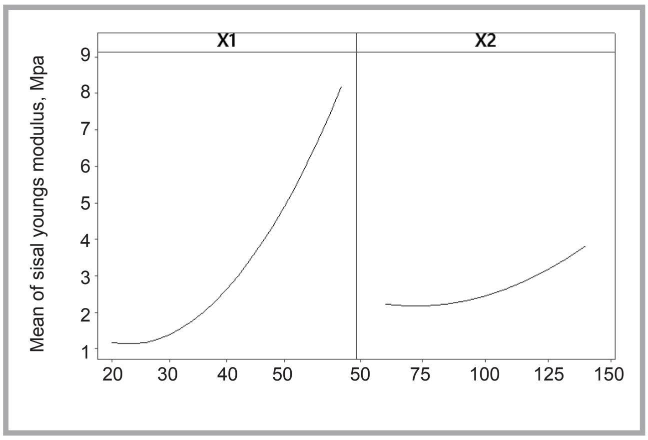

Figure 6. Main effects plot for Young's modulus of sisal bio-composites.

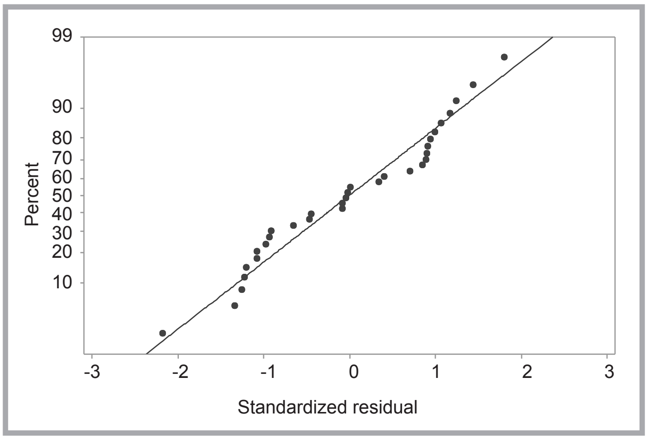

Figure 7. Normal probability plot for compressive strength of sisal bio-composites

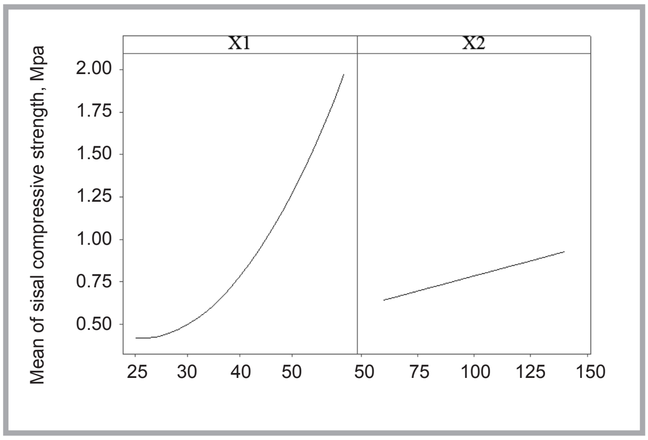

Figure 8. Main effects plot for compressive strength of sisal bio-composites

$$Y_Y(\text{Sisal}) = 3.5 - 0.2091X_1 + + 0.01801X_2 + 0.004706X_1X_1 \quad (4)$$

Table 10 is a summary of the analysis of variance, percentage factor contributions and variance inflation factor statistics generated by the models. The percentage contributions of various factors were obtained, with the fibre volume fraction contributing the highest percentage of 45.13% to the model and bio-resin mass – 4.37%. The curvilinear effect of the squaring fibre volume fraction ($X_1 * X_1$) contributed a percentage of 4.41% to the regression model. Variance inflation factors proved that there was no multi-colinearity among the linear, curvilinear and interaction effects.

The regression model was used to design a prediction and optimisation report for the Young's modulus of the bio-composites developed. Optimal settings yielded a maximum Young's modulus of 11.99 MPa for sisal-reinforced bio-composites. Furthermore at a 95% confidence interval (CI), a Young's modulus range between 10.72 MPa and 13.25 MPa was determined. Young's modulus was much lower than that for low density polyethylene sisal composites obtained by [4] – 140 MPa, which may be due to variations in the origin and gauge length of fibres used in the composite. Optimum settings of the fibre volume fraction (60%) and bio-resin mass (140 grams) for Young's modulus were obtained, given in ***Table 11***.

From the predictive regression model generated, it was evident that the fibre volume fraction (X_1) contributed the highest percentage to the model. This was further evidenced by ***Figure 6***, where a decrease in the fibre volume fraction (X_1) led to an increase in Young's modulus, whereas a double increase in (X_1) resulted in a increase in Young's modulus (Y_Y). Furthermore an increase in bio-resin mass (X_2) increased the tensile strength of the sisal bio-composite, which could be due to the presence of cellulosic fibres in the bio-composites, exhibiting a plasticising effect. The addition of fibres with higher strength and stiffness to the polymer matrix greatly improves the tensile properties of composites [12].

Compressive strength

The normal probability plot of residuals for compressive strength in ***Figure 7*** shows that the data points were distributed normally.

Multiple regression analysis of compressive strength (Y_C) exhibited models with an adjusted R^2 value of 0.90 and significant P-value of 0.0001.

$$Y_C(\text{Sisal}) = 3.041 - 0.0961X_1 + + 0.01764X_2 + 0.0009804X_1X_1 + + 0.000549X_1X_2 \quad (5)$$

Table 10 is a summary of the analysis of variance, percentage factor contributions and variation inflation factors from the model. The sisal bio-composite regression model was significant (P-values

Table 10. *ANOVA, % contributions and VIF for compressive strength of sisal bio-composites.*

Source	ANOVA, P-value	Factor contributions, %	VIF
Regression	0.000	95.03	
X_1	0.000	42.88	1.08
X_2	0.015	4.43	1.05
$X_1 * X_1$	0.008	8.45	1.44
$X_1 * X_2$	0.003	1.81	1.41
Bio-composite type (sisal)	0.000	34.10	1.01
Error		8.32	
Lack-of-fit	0.003	8.26	
Pure error		0.06	
Total		100.00	

Table 11. *Optimum settings for compressive strength of sisal bio-composites.*

Goal: maximised compressive strength, MPa		Solution: optimum settings	
Predicted compressive strength	2.94	X_1	60
95% Confidence Interval	(2.64, 3.24)	X_2	140
95% Predicted Interval	(2.38, 3.5)		

< 0.05) with no multi-colinearity, hence it was used to design optimum settings for the compressive strength of the bio-composites developed. From ***Table 11***, optimum settings of a 60% fibre volume fraction and 140 grams of bio-resin yielded a maximum compressive strength of 2.94 MPa for sisal-reinforced bio-composites. Furthermore at a 95% confidence interval (CI), a compressive strength range between 2.64 MPa and 3.24 MPa was determined. According to [5], sisal fibre reinforced bio-composite laminates yielded a compressive strength of 42 MPa, which was higher than the value obtained in this research. This could be attributed to the variations in the fibre volume fraction and fibre gauge length used in the previous study as compared to this research.

Table 10 presents the percentage contributions of various factors, with the fibre volume fraction contributing the highest percentage of 42.88% to the model and bio-resin mass – 4.43%. The curvilinear effect of squaring the fibre volume fraction ($X_1 * X_1$) contributed a percentage of 8.45% to the regression model. Variance inflation factors proved that there was no multi-colinearity among the linear, curvilinear and interaction effects. In addition, ***Figure 8*** was in agreement with the compressive strength model, and showed that squaring the fibre volume fraction (X_1^2) resulted in a significant increase in tensile strength (Y_T), whereas an increase in bio-resin mass (X_2) increased the tensile strength of the sisal bio-composite. However, the standard requirement for commercial medium density fibreboards was 10 MPa, which is close to the ranges obtained [8].

Conslusions

An investigation of the factors affecting sisal bio-composites was undertaken by studying the effect of the fibre volume fraction, mass of glycerine and mass of bio-resin. Sisal fibres were pre-treated using sodium hydroxide. According to the results obtained in this research work, the treated sisal fibres exhibited a significant improvement in mechanical properties when compared to the untreated fibres. Therefore the treated sisal fibres were used for the design of sisal bio-composites. Sisal bio-composites exhibited a model with an adjusted R^2 value of 0.92 and tensile strength of 5.23 MPa. The percentage elongation model yielded an adjusted R^2 of 0.96 and elongation of 1.53%. Multiple regression analysis of Young's modulus produced a model with an adjusted R^2 of 0.91. The compressive strength exhibited a regression model with an adjusted R^2 of 0.90 and 2.94 MPa. The tensile and compressive properties obtained were close to those of commercial bio-composites & solid wood boards.

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