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Modeling and Investigation of the Influential Reinforcement Parameters on the Strength of Polypropylene Lignocellulosic Fiber Composites Using Analysis of Variances and Box-Cox Transformation Technique

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Green materials have received great interest in wide industrial applications due to their desired properties. However, the reinforcing conditions have a significant impact on how they perform in their final use. The current study intends to statistically examine the effects of three key parameters on the average tensile strength of polypropylene composites. These factors included the type of fiber, the chemical treatment, and the fiber's weight percentage. The fibers were hemp and sisal, and the weight percentages were 10, 20, and 30. While some of them received sodium hydroxide (NaOH) treatment, the rest were left untreated. The main effect and the interaction effect were both examined using the analysis of variance (ANOVA). The findings demonstrated that, on average, the weight percentage had no tangible effect on the tensile strength of polypropylene (PP) composites. Additionally, the performance of sisal and hemp composites was unaffected by treatment. The strength, however, is significantly influenced by the type of fiber. The investigation also showed that there was little difference between untreated hemp and untreated sisal in terms of tensile strength.

Keywords: *Green composites, Natural fibers, Green products, ANOVA, Box-Cox transformation, Statistical analysis, Mechanical performance.*

1. Introduction

The properties of natural fibers, such as biodegradability, availability, low cost, and lightweight along with their mechanical ones, have increased their demand for reinforcements in plastic composites[1] that are used in industrial, medical, and electrical applications[2]. Natural fibers are mainly composed of three components, which are cellulose, hemicellulose, and lignin. Additionally, they include secondary ones such as pectin and wax[3]. Cellulose content affects some mechanical characteristics like tensile strength[4-5]. The weight percentage of the major chemical composition and the tensile properties of some fibers are presented in Table 1[6]. Although natural fibers have many advantages, they mainly suffer from weak bonding with plastics, which negatively impacts their mechanical properties[7].

Chemical treatments have been used by researchers to improve the adhesion between the matrix and the fiber. One of the most often applied chemical treatments is alkaline treatment. Natural fibers that have undergone alkali pretreatment would change in terms of their fine structure, morphology, and mechanical characteristics, as alkali can convert native cellulose I to cellulose III. Such chemical treatment can remove a certain amount of lignin, wax, and oils covering the external surface of the fiber cell wall resulting in more compatibility with the composite matrix by developing more interlocking of the natural fiber within the matrix due to more surface roughness of the fiber in one hand, and improve the fiber tensile strength and Young's modulus due to increasing the crystallinity of cellulose where the addition of aqueous sodium hydroxide to natural fiber promotes the ionization of the hydroxyl group to the alkoxide.

Numerous natural fibers have been incorporated into polymer matrices as reinforcements to improve their mechanical properties. Such fibers include cypress and pine[8], lemon leaves[9], olive leaves[10], grapes[11], hemp, sisal, and jute[12], etc. Many parameters affect the tensile strength property of biopolymeric composites. But according to our screening, one of the most effective factors that significantly affect tensile strength is the type of natural fiber. For example, AL-Oqla et al.[13] studied the effects of two fibers (lemon and palm) on a polypropylene (PP) matrix. They found that when 30 weight percentage of palm fiber is replaced by 30 weight percentage of lemon fiber under the same processing conditions, the difference in tensile strength is (89%). Sarikaya et al.[14] made a comparison between birch and eucalyptus fibers in the epoxy matrix. According to their findings, eucalyptus provided tensile strength that was better by 50%. Cavalcanti et al.[15] found that replacing jute fiber with a hybrid fiber (jute and sisal) significantly improved tensile strength by 51%. AL-Oqla et al.[16] studied the effect of three wt.% (20, 30, and 40) of parsley fiber on

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Table 1. The weight percentage of the major chemical composition and the tensile properties of some fibers.

Fiber	Cellulose (wt.%)	Hemicellulose (wt.%)	Lignin (wt.%)	Tensile strength (MPa)	Young's modulus (GPa)
Hemp	70–74	17.9–22.4	3.7–5.7	690	30–60
Jute	61–71.5	13.6–20.4	12-13	393–773	13–26.5
Flax	71	18.6–20.6	2.2	345–1100	27.6
Cotton	85–90	5.7	—	400	12
Coir	32–43	0.15–0.25	40–45	220	6
Sisal	66–78	10–14	10–14	600–700	38

the PP matrix. The findings indicated that 30 wt.% of parsley could approximately raise the tensile strength by 175%. AL-Oqla[11] revealed that the addition of 30 wt.% of grape fiber could improve the strength of Low-density polyethylene (LDPE) by 133%. In addition to weight percentage and fiber type. The chemical treatment by sodium hydroxide (NaOH) also contributes to the tensile strength of natural composites. To exemplify, Ibrahim et al.[17] soaked pineapple leaf fiber in 3, 5, and 7 wt.% of NaOH. They found that 7% NaOH increased the tensile of the PP matrix by 25%. However, Islam et al.[18] found that 5 wt.% of NaOH increased the strength of 40 wt.% kenaf-recycled PP composite by 10%. Tripathy et al.[19] used 2.5 wt% of NaOH to treat date palm fiber. The difference in tensile strength of the epoxy composites was 56.79%.

The literature has demonstrated that the type of fiber, weight percentage, and chemical treatment all affect the tensile strength of natural polymeric composites. The current study intends to investigate the effects of these parameters on the tensile strength of the PP matrix in a statistical framework based on data gathered from previous studies. Three weight percentages (10, 20, and 30) of either hemp or sisal fiber were examined for their effects. Due to their high cellulose content, hemp and sisal fibers were chosen. While some of the fiber was saturated with NaOH, some of it wasn't. Both the main factors and their interactions will also be studied to develop a better understanding of green fiber/PP composite. The analysis of variances was used to make these predictions about the impacts of each parameter and their interactions, and the Box-Cox transformation approach was applied to improve the model's reliability.

2. Methodology

2.1. Materials

To examine the effects of hemp fiber and sisal fiber on the tensile strength in statistical analysis as one of the key influencing factors, the tensile strength values of hemp/PP and sisal/PP composites were chosen from the literature for the current study. From previous studies, several PP matrices, including homopolymer, copolymer, isostatic, and even recycled PP, were used. In this study, all matrices are referred to as PPs. The fibers that were employed have a length of less than 16 mm. Injection molding or compression molding was used to manufacture all composites, and a screw extruder or two hot rollers were used to mix the materials without the need of any coupling agents. Nevertheless, some fiber was subjected to sodium hydroxide (NaOH) treatment while the remainder was left as raw fiber.

2.2. Data collection

Data on the effect of fiber reinforcement weight percentage and the existence of chemical treatment (NaOH) on tensile strength have been collected from numerous studies worldwide. This implies that the experiments were carried out under different conditions. However, average tensile strength values were recorded. The combinations of the reinforcement conditions of the considered experimental works are tabulated in Table 2. These were arranged as the tensile strength of three wt. % (10, 20, and 30) of two fibers (hemp and sisal) with PP matrix. Some of these fibers were chemically treated with NaOH, while others were not. Several concentrations of NaOH have been reported to be used to determine its effect on tensile strength. However, the present research has taken into account the concentration that gave the highest tensile strength.

To exemplify, if more than one researcher studied the effect of the different concentrations of NaOH on the tensile strength, then the highest value of the tensile strength would be considered. For example, Ibrahim et al.[26] and Joseph et al.[30] assessed the effect of reinforcing PP by 20 wt.% of sisal, but every study used different concentrations of NaOH (5 wt.% and 10 wt.%, respectively). Then, 20 wt.% sisal (treated in 5 wt.% NaOH) resulted in tensile strength of 37.5 MPa, while 20 wt.% sisal (treated in 10 wt.% NaOH) resulted in tensile strength of 40.35 MPa. Consequently, a tensile strength of 40.35 MPa was considered in this study

2.3. Full factorial design

A full factorial design is primarily used to study the effects of more than one factor on a response variable at the same time[31]. The current factorial design was used to study the effect of different levels of three factors on the tensile strength (response variable) of PP composites. These factors are the weight percentage (10, 20, and 30), the existence of NaOH (yes and no, regardless of the concentration), and the type of fiber (hemp and sisal). The factorial design is presented in Table 3. The effects of these factors on tensile strength were investigated using variance analysis (ANOVA). In this study, the Minitab software was used to analyze the data with a 95% confidence level. The adjusted square correlation coefficient (R^2adj) was used to assess the adequacy of the model.

Table 2. The effect of weight percentage of untreated hemp fiber on tensile strength of PP composite.

Hemp wt.%	NaOH	TS1 (MPa)	TS2 (MPa)	TS3 (MPa)	TS4 (MPa)	TS5 (MPa)	TS6 (MPa)	Average (MPa)
10	0	24.42[20]	35.5[21]	29[22]	-	-	-	29.6
20	0	35[21]	21[22]	-	-	-	-	28.0
30	0	18[23]	21.99[20]	30[24]	37.5[12]	35[21]	35[22]	29.6
10	1.6 mol/L	26.25[25]	-	-	-	-	-	26.25
20	1.6 mol/L	26.5[25]	-	-	-	-	-	26.5
30	5 wt.%	25.7[20]	-	-	-	-	-	25.7
Sisal wt.%								
10	0	38[26]	22.5[27]	24.17[28]	-	-	-	28.2
20	0	37[26]	24.5[27]	-	-	-	-	30.8
30	0	37[12]	37.5[26]	29[29]	25.25[27]	29.25[28]	-	31.6
10	5	38.5[26]	-	-	-	-	-	38.5
20	10	40.35[30]	-	-	-	-	-	40.35
30	10	44.35[30]	-	-	-	-	-	44.35

Some tensile strength values in this table were estimated from the figures in their articles.

Table 3. Full factorial design.

Experimental Condition	wt.% (Factor 1)	NaOH (Factor 2)	Fiber type (Factor 3)	Tensile Strength (MPa) (Response variable)
1	10	Yes	Hemp	26.3
2	20	Yes	Hemp	26.5
3	30	Yes	Hemp	25.7
4	10	No	Hemp	29.6
5	20	No	Hemp	28.0
6	30	No	Hemp	29.6
7	10	Yes	Sisal	38.5
8	20	Yes	Sisal	40.4
9	30	Yes	Sisal	44.4
10	10	No	Sisal	28.2
11	20	No	Sisal	30.8
12	30	No	Sisal	31.6

2.4. Data transformation

The ANOVA tool is based on two important assumptions. The first is that the data has to be distributed normally, whereas the second is the homogeneity of the variance. The validation of the first assumption can be investigated by the probability plot[32]. This test determines if the data is normally distributed or not. The null hypothesis (H_0) in this test indicates that the data are normally distributed, whereas the alternative hypothesis (H_1) involves a non-normal distribution[33]. For ease of interpretation, if the P-value is greater than or equal to 0.05, it may be stated that the data are normally distributed. In this work, the Box-Cox technique is used to transform the response variable in order to obtain normally distributed residuals. Many researchers used this technique in order to obtain a more precise model[34]

3. Results and Discussion

3.1. Checking the validation of ANOVA

As mentioned above, a visual inspection and statistical test may be used to examine the normality of residues. A visual inspection may be performed using the normal probability plot or histogram. The latter one is preferable for large sample sizes, normal probability plots are used for both small and large sample sizes[35]. Therefore, probability plots are used in this study due to the small sample size.

Figure 1 shows a normal probability plot of the tensile strength residuals of the PP composites. From the residual plot, it can be visually seen that the points are not along a straight line. This means that the residuals for the tensile strength do not fit a normal distribution. However, visual inspection is not quite enough. Therefore, an Anderson-Darling test was performed to increase certainty. The P-value for the Anderson-Darling test was calculated and was 0.031. This value is less than 0.05. Therefore, we reject the null hypothesis was rejected and it can be concluded that the residuals deviate from the normal distribution. In this case, the ANOVA tool cannot be used and the response variable must be transformed.

In this work, Box-Cox transformation was used to correct the data. The optimal value for exponent lambda (λ) was calculated by Minitab software. The rounded (λ) was (-3) which means that the response variable should be transformed according to Equation 1:

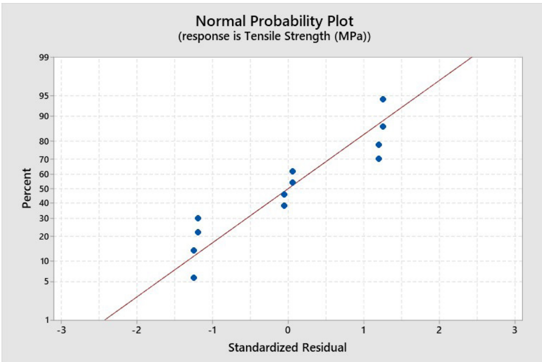

Figure 1. Normal probability plot of the residuals for the tensile strength of PP composites.

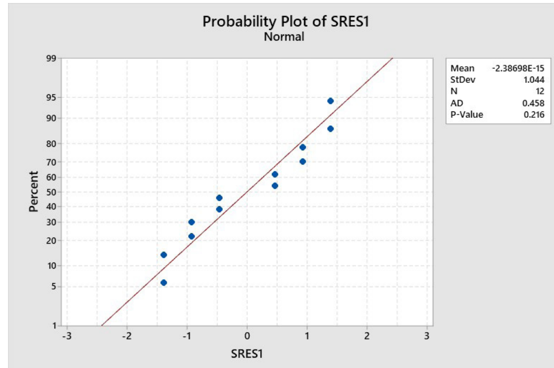

Figure 2. Normal probability plot of the residuals for the transformed tensile strength.

$$(TS)_t = 1/\left(TS^3\right) \quad (1)$$

Where TS is the original tensile strength, and $(TS)_t$ is the transformed tensile strength. Table 4 displays the data after transformation. The P-value for the Anderson-Darling test was recalculated following the transformation of the tensile strength, and it was found to be 0.216.

This indicates that the residuals of transformed strength follow the normal distribution. Further, the normal probability plot shows that the points are closer to the straight line, as shown in Figure 2. A similar procedure was conducted for impact strength by[34].

Homogeneity of variance is another assumption of interest to researchers when performing ANOVA analyses. Homogeneity of variance means that the variances between groups are equal. Plotting residuals versus fits is one common method used to ensure constant variance[36]. This plot relies on visual inspection. The constant variance assumption is valid if the residuals show a random pattern around the horizontal line. Otherwise the assumption is violated. Figure 3 shows the residual vs. fitted value plot[37]. From this plot, it can be seen that the residuals are randomly scattered around the zero line. This indicates that the assumption of homogeneity of variance is valid. Consequently, the ANOVA tool can be used with the current data.

3.2. Analysis of variance (ANOVA) for tensile strength

To assess the effects of main and interaction factors tensile strength, the sum of square (SS), mean square (MS), Fisher\`s variance ratio (F-value), and probability value (P-value) were calculated by Minitab software and presented in Table 5. Factors with P-values less than (α = 0.05) are considered significant. Therefore, among the main factors, only fiber type (whose P-value is 0.012) can be considered a significant factor for tensile strength. This means using both hemp and sisal has a significant effect on the tensile of the PP matrix. Regarding the fiber weight percentage, it can be concluded that the wt.% factor does not affect the tensile strength. This means that the engineers can use the highest fiber weight percentage. Furthermore, treating the fiber in NaOH does not significantly affect its tensile strength.

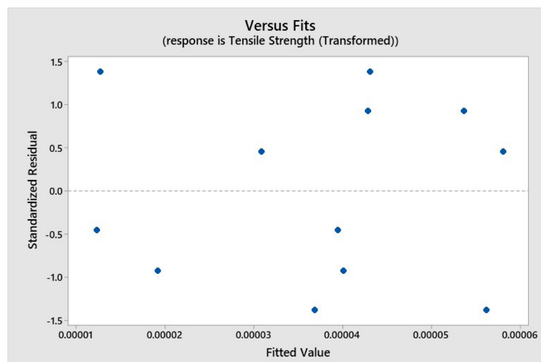

Figure 3. The plot of the residual versus fit for the tensile strength.

Table 4. Original and transformed tensile strength values.

Run Order	Tensile Strength (Original)	Tensile Strength (Transformed)
1	26.3	0.0000553
2	26.5	0.0000537
3	25.7	0.0000589
4	29.6	0.0000384
5	28.0	0.0000456
6	29.6	0.0000386
7	38.5	0.0000175
8	40.4	0.0000152
9	44.4	0.0000115
10	28.2	0.0000445
11	30.8	0.0000344
12	31.6	0.0000317

As for the two-way interactions, the wt%/treatment and wt%/fiber type interactions are not significant. In contrast, an effective interaction between treatment and fiber type is observed as seen in Table 5. To assess the validity of the developed model, the coefficient of determination (R^2) was calculated. In general, the higher the R^2, the better the fit. However, the adjusted R^2 takes into account the number of variables and therefore gives more accurate conclusions

Table 5. Analysis of Variance (ANOVA) for transformed tensile strength.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
wt.%	2	0.00000000273	0.000000000303	0.74	0.574
Treatment	1	0.00000000160	0.000000000400	1.94	0.299
Fiber Type	1	0.00000000003	0.000000000014	80.81	**0.012**
wt.%*Treatment	2	0.00000000004	0.000000000037	0.49	0.669
wt.%*Fiber Type	2	0.00000000154	0.000000001536	1.89	0.346
Treatment*Fiber Type	1	0.00000000113	0.000000000226	54.72	**0.018**
Error	2	0.00000000002	0.000000000009		
Total	11	0.00000000007			

S = 0.0000044, R-Sq = 98.63%, R-Sq (adj) = 92.45%.

about the accuracy of the model[38]. In this study, R^2(adj) is 92.44, implying that the current model fits the results strongly.

3.3. Factorial plots for transformed tensile strength

The main effect plot shows how changing the values of various factors affects the response variable[39]. A higher influence of a factor on a response variable is indicated by a steeper slope. The main effect plot in Figure 4 shows that the fiber type factor has the greatest influence on tensile strength, where the transformed tensile strength decreased by 46.7% when the fiber changed from hemp to sisal. About the treatment factor, it can be seen that its slope is milder than that of the fiber weight percentage. The converted tensile was changed by untreated fiber by 9.9%. A straighter line with a slight variation in tensile strength (4.3% and 5.5%, respectively) is produced by increasing the weight percentage of the fiber from 10 to 20 and from 20 to 30.

The relationship between a factor and a response variable depending on another factor is depicted using an interaction plot[40]. Figure 5 makes it clear that there is no interaction between the weight percentage and treatment. In other words, the relationship between the transformed tensile strength and the weight percentage does not depend on the levels of treatment factor. For further clarification, the tensile strength difference between treated and untreated materials is 12% at 10 weight percentage and 13.8% at 20 weight percentage.

Furthermore, the effect of wt.% on the transformed strength does not depend on the fiber type. To exemplify, the difference in transformed tensile strength at 10, and 30 wt.% between hemp and sisal is 33.8, 50, and 55.7%, respectively. Regarding the interaction between treatment and fiber type, it can be noted that the relationship between the fiber type and the transformed tensile strength strongly depends on the chemical treatment. To illustrate, the difference in transformed strength between hemp and sisal at the Yes level is 73.6% while the difference at the No level is 9.8%. This means that the difference between the high and low levels is 63.8% which is considered a significant difference, as indicated in Table 5. Moreover, it can be seen that the type of fiber has little effect at No level (without treatment). This indicates that the effect of both fiber types on the response is equal. As a result, if they are not treated, both of them provide tensile strength that is fairly comparable. In contrast to sisal, processed hemp fiber has a significantly greater transformed tensile strength. If the treated sisal is substituted with treated hemp, there will be a noticeable difference when the interaction plot is drawn using the original tensile strength values (without transformation), as shown in Figure 6.

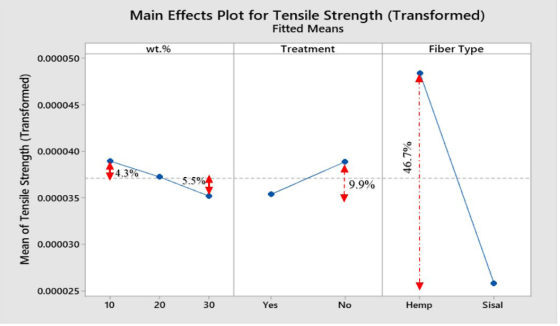

Figure 4. The main effect plot for the mean transformed tensile strength.

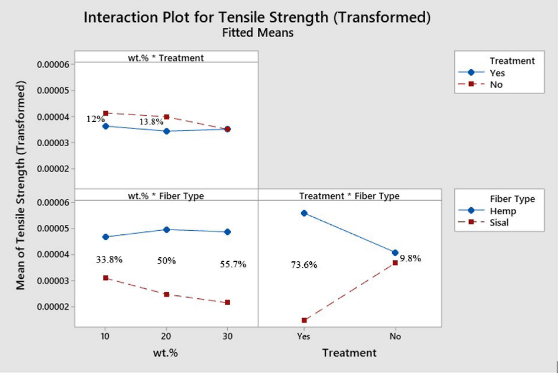

Figure 5. The second-order interaction effect plot for the mean transformed tensile strength.

A horizontal Pareto chart, such as the one in Figure 7, is used to show the statistical significance of the main and interaction effects on the response variable. The bars are arranged in this chart from largest to smallest based on their significance. Every bar that crosses the vertical line (in our example, the t-value of 4.3) is regarded as a significant factor[40]. Figure 7 indicates that the fiber type has the highest significant effect on the strength, and it is followed by the

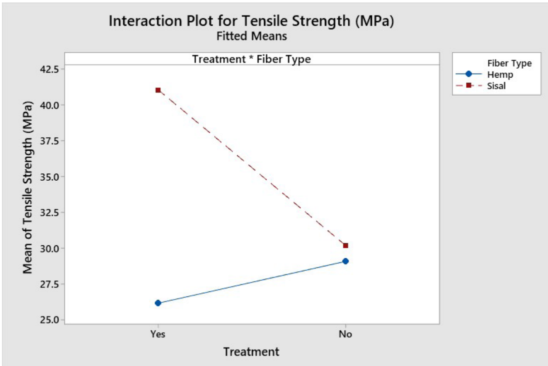

Figure 6. The second-order interaction effect plot of fiber type and treatment for the mean tensile strength.

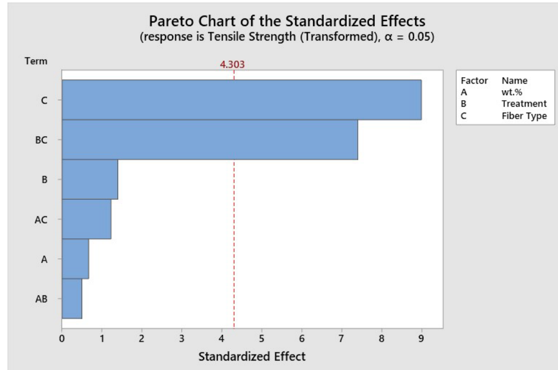

Figure 7. Pareto chart for the tensile strength of the bio-PP composite.

interaction between the treatment and fiber type. However, the interaction between the weight percentage and the treatment has the least significant influence on the tensile strength.

3.4. Mathematical model development

The regression equation represents the statistical relationship between a response variable and different independent variables. The general formula of the regression equation is shown in Equation 2[39]

$$Y = \beta 0 + \beta 1 (A) + \beta 2 (B) + \beta 3 (C) + \beta 4 (AB) + \beta 5 (AC) + \beta 6 (BC) + \beta 7 (ABC) \quad (2)$$

Where
Y is the response variable (tensile strength)
A, B, and C are the independent variables (wt.%, treatment, and fiber type)
β0 represents the average tensile strength value.
β1, β2 … β7 are the regression coefficients of the main and interaction effects
In our case, the regression model can be expressed by Equation 3.

$$TS\ (transformed) = 0.000037 + 0.000011\, Fiber\ Type / Hemp - 0.000011\, Fiber\ Type / Sisal + 0.000009\, Treatment / Yes * Fiber\ Type / Hemp - 0.000009\, Treatment / Yes * Fiber\ Type / Sisal - 0.000009\, Treatment / No * Fiber\ Type / Hemp + 0.000009\, Treatment\ (No) * Fiber\ Type / Sisal \quad (3)$$

All non-significant terms were eliminated. The tensile strength of the composites can be predicted by substituting categorical predictor coding (0, 1) for the type of fiber and the interaction between the treatment and the fiber type. Figures 8-11 show the substitution results for hemp and sisal, both treated and untreated.

The findings of the comparison between the regression model and the actual tensile strength values are shown in Figures 8-11. Figure 8 shows that the projected transformed

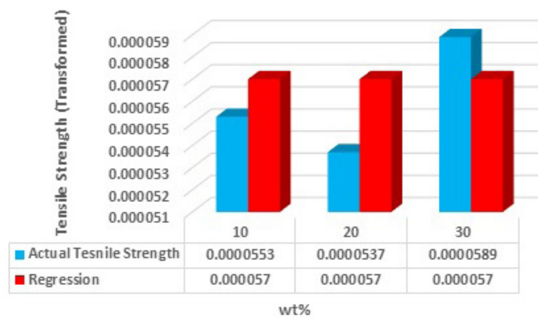

Figure 8. A comparison between the regression equation and the actual value of the tensile strength of treated hemp-PP composite.

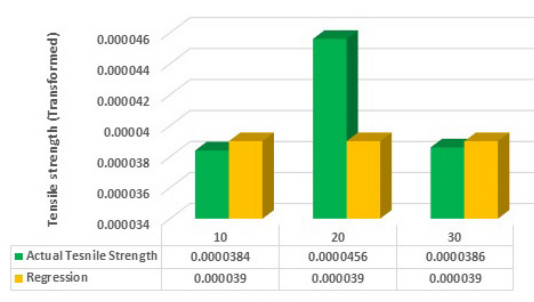

Figure 9. A comparison between the regression equation and the actual value of the tensile strength of untreated hemp-PP composite.

tensile strength of treated hemp-PP remains constant as the weight percentage rises. The same trend may be seen in a composite made of untreated hemp and PP (Figure 9), treated sisal and PP (Figure 10), and untreated sisal and PP (Figure 11). This is becaus the weight percentage was not considered in the regression equation because it was not significant in the ANOVA model.

The smallest difference in the tensile strength appears at 10 wt.% of untreated hemp/PP whereas the biggest difference is 5.96% at 20 wt.% (Figure 8). From Figure 9, it can be noticed that the difference in the tensile strength hit the lowest value, 1%, among all the composites at 30 wt.% untreated

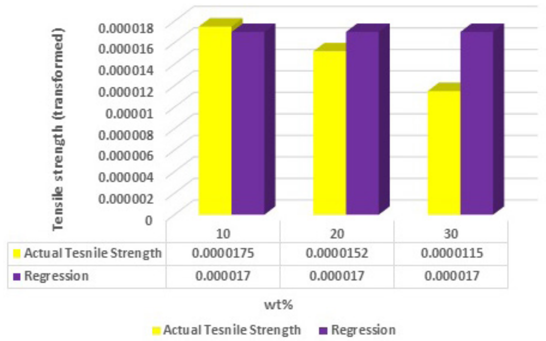

Figure 10. A comparison between the regression equation and the actual value of the tensile strength of treated sisal-PP composite.

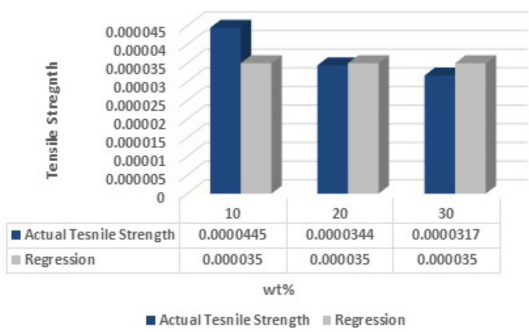

Figure 11. A comparison between the regression equation and the actual value of the tensile strength of untreated sisal-PP composite.

hemp/PP composite. However, the tensile strength difference reached the highest value, 38.6%, among all composites at 30 wt.% untreated sisal/PP (Figure 11).

4. Conclusions

The effect of fiber type, weight percentage, and treatment with sodium hydroxide on the tensile strength of the bio-PP composite was investigated using the design of experiments. The findings have revealed that using 10, 20, or 30 wt.% of either hemp or sisal has not had a significant effect on the tensile strength. Therefore, it is suggested to use the highest amount of fiber to reduce both the density of the composite and the detrimental influences of PP on our environment. Also, it is worthwhile to mention that the treatment factor has a marginal effect on tensile strength.

However, when the interaction between the fiber type and the treatment factors was examined, it was discovered that this interaction has a significant effect. This means that the effect of treatment on the tensile strength of PP depends on the fiber type. In other words, both hemp and sisal fibers have a very close influence on tensile strength when the fiber is not treated. However, there is a big difference in the tensile strength when the fiber is treated. Consequently, it is proposed to use sisal fiber (if it is untreated) rather than hemp since many studies have recently proved that hemp has harsh influences on human health. Eventually, the fiber type factor has a dramatic impact on the tensile strength when it is studied at the same time and when it interacts with the treatment factor.

5. Acknowledgments

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