Mathematical Modelling of Engineering Problems
Vol. 10, No. 1, February, 2023, pp. 242-249
Journal homepage: http://iieta.org/journals/mmep

Investigating and Predicting the Effects of Fiber Chemical Composition and Treatment on the Mechanical Properties of Natural Fiber Composites by Response Surface Method

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https://doi.org/10.18280/mmep.100128

Received: 14 December 2022
Accepted: 1 February 2023

Keywords:
green composites, biomaterials, natural fiber, mechanical performance, Cellulose

ABSTRACT

This paper aims to integrate the response surface methodologies to investigate the effect of the cellulose and hemicellulose contents on the mechanical properties of the polypropylene-based composites with green fiber reinforcement conditions. In this study, the tested data are collected from various literature resources demonstrating the green fiber type, the chemical treatment condition, and the resultant mechanical properties, i.e., the tensile modulus and tensile strength. Accordingly, the response surface analysis is utilized to obtain a high-accuracy first order regression model to formulate the influence of the cellulose, hemicellulose, and the treatment condition on the tensile characteristics of the green composite. The results showed that the bio-composites samples with higher cellulose and lower hemicellulose percentages have significantly better tensile modulus properties. However, such samples would have lower tensile strength qualities. Additionally, the presence of chemical treatment can significantly improve the tensile properties of the polypropylene-based composites.

1. INTRODUCTION

Advanced bio-composite materials have brought fresh perspectives to the development of modern societies. Their light weight, low cost, great specific strength, degradability features and the flexibility in design have all had a considerable influence on the business of green products. As a result, bio-composite materials have been implemented in several industrial applications including automotive, aerospace, electronics, medical and packaging applications [1-4]. Natural fiber composites are potential bio-based materials that contain lignocellulosic fibers as reinforcement. Natural fibers are complex and unpredictable, which make the properties of bio-composites are very sensitive to the manufacturing processes [5-7]. Natural fiber extraction is also important as it can affect its properties [8, 9]. Plant based fibers extraction from crops is made by the retting process where the non-fibrous tissues are removed through decomposition and degradation of hemicellulose and pectin resulting in individual fibers [10, 11]. One way for retting is using enzymes, which results in a better controllability, faster extraction, and low environmental impact [11, 12]. However, this method is expensive and non- fibrous materials must be removed after retting process. Therefore fibers are extracted by breaking, milling, scutching then cleaned, refined and processed to be useable for certain application.

Biocomposite materials are aimed to be utilized in various industrials applications where mechanical characteristics including tensile strength, tensile modulus, impact strength and elongation at break are vital in achieving sustainable, functional, and dimensional stability successful green products [13-19]. Such mechanical features of the materials are responsible for composite behavior regarding deformations and load resistance, and thus their geometrical stability. Materials stiffness or Young’s modulus, as well as impact strengths and materials ductility, are very important to be investigated for the biomaterials before implemented in various products as they will determine the functional requirements for a certain application[4, 20-23]. Thus, all these mechanical features were appropriately and carefully examined bio-composite materials under the effect of various reinforcing conditions including chemical treatment of the fibers [24-27].

Moreover, the compatibility between fibers and matrix is generally weak due to their hydrophilic- hydrophobic nature. This in order would make the interfacial bonding developed inside the composite not sufficiently strong if not compensated by proper fiber loading and physical and /or chemical treatments for the lignocellulosic fibers [28-30]. This would generally develop their interfacial bonding with the matrix. Furthermore, tensile properties of the natural fiber composites have been found to be dependent on the chemical treatment of natural fibers [30, 31]. Besides, excess fibers were not found efficient in reducing the brittleness of the composites due to the improper interlocking with the matrix. This means that some fibers may slide over each other or pulled out from their positions during elongations and make the polymer elongated more freely without restrictions of fibers. The mechanical properties of bio-polyethylene reinforced with coffee silverskin were investigated. Two treatment methods were used alkali bleaching and esterification with palmitoyl chloride, it was shown that there was an improvement in the elastic modulus and a reduction in strain at maximum stress with the increase in coffee sliverskin fraction for the untreated and treated composites [32]. It was also reported that there was an increase in tensile and flexural strength in alkali treated natural fiber-reinforced polymer composite [33]. In addition, it was shown that there was an increase in impact strength with jute, hemp and flax-based polymeric composites when subjected to chemical treatments or with hybrid with other reinforcements [34-36].

Accordingly, the mechanical properties of the natural fiber composites are of paramount importance to develop more functional green products. Thus, this work aims to investigate and predict the effect of the natural fiber chemical composition, particularly, the cellulose and hemicellulose content as well as the chemical treatment of the fibers and their interactions on their composite mechanical properties by using response surface method to enhance more reliable composites capable of producing more sustainable and functional products.

2. METHODOLOGY

Design of experiments and Response surface method (RSM) were implemented for the natural fiber composites to investigate the effects of the fiber's chemical composition and chemical treatment on their mechanical performance. This was important as natural fibers show scatter in their mechanical properties due to their inherent chemical composition mainly the cellulose and hemicellulose contents. Thus, the natural fiber composite usually demonstrate variations in their mechanical performance due to this issue as well as the different conditions of possessing. Therefore, natural fiber composites can be examined according to different properties before being considered to be used in the design process for any particular application. This can be performed considering various natural fiber's types and conditions.

Response surface method (RSM) was implemented in the present paper to examine the effect of cellulose and hemicellulose content as well as the treatment conditions on the tensile strength and tensile modulus of the polypropylene-based green fiber composites. In fact, the RSM is commonly used to identify the main and the interaction effects of the independent variable on a certain response using a collection of statistical and mathematical procedures [37]. RSM is widely used in the analysis of several engineering processes [38-40]. In the present paper, cellulose and hemicellulose levels and the treatment conditions are the independent variable and the tensile properties, i.e., tensile strength (TS) and tensile modulus (TM) are the responses. Thus, the RSM is employed here to fit the responses using a first order response surface as:

$$\mathrm{Y} = \beta_o + \sum_{i=1}^{k} \beta_i Z_i + \sum_{i \le j} \sum \beta_{ij} Z_i Z_j + e \qquad (1)$$

where, Y is the response; Z_i and Z_j are the independent variables; β_o is the constant coefficient; β_i, and β_{ij} are the coefficients of the main and the interaction effects, respectively; and e is the random error. In this equation, Y can be either used for (TS) or (TM) responses.

The green fiber data presented in this paper were collected from different reliable literature source [41-49] to demonstrate various fiber types and reinforcement conditions. Table 1 shows the complete data set and Table 2 shows the cellulose and hemicellulose in each fiber type. This also was obtained from literature sources [5, 9, 50-53].

In order to obtain the cellulose and hemicellulose in combination of Table 1, some data processing was required. First, is to obtain the average content of cellulose and hemicellulose of each fiber type of Table 2. For example, for the Jute fiber the cellulose is found to be 66.25% and the hemicellulose content is calculated as 17%. The second step is to find the percentage of the cellulose and hemicellulose in each alternative by multiplying the average cellulose or hemicellulose percentage determined the step 1 above by the percentage weight of each corresponding alternative. For example, item number 10 where the Jute percentage weight is 20%, the cellulose and hemicellulose contents of this item are computed as 13.25% and 3.40% respectively. Furthermore, the values of the tensile modulus and strength were considered to be the average value as well. The complete processed data set is summarized is Table 3. This data set was analyzed using the response surface methodology, as will be shown in the following subsection.

Table 1. Complete data set for tensile modulus and strength values

Item #	Alternative	Tensile Strength (*MPa*)	Tensile Modulus (*MPa*)
1	Coir 15wt% /PP Treated	26.1 28 29	2210 2400
2	Coir 20wt% /PP Treated	26 29.5	3000
3	Date palm 30wt% /PP	24 18 21 17	700 1550 1610 650
4	Date palm 30wt% /PP Treated	29 25 28	800 1350 800
5	Flax 30wt% /PP	40.1 26	5178 1700
6	Flax 30wt% /PP Treated	44.1 43	5701 2000
7	Flax 50wt% /PP	24	2500
8	Flax 50wt% /PP Treated	50 53	2700
9	Jute 20wt% /PP	25.8	1680
10	Jute 20wt% /PP Treated	29.8	2490 1880
11	Jute 30wt% /PP Treated	28.7	2250
12	Kenaf 30wt% /PP Treated	46 23.18 57 50	2600 6000 1774
13	Kenaf 40wt% /PP	35 10.33	8000 1282
14	Kenaf 40wt% /PP Treated	44 19.84 55	6000 1778
15	Sisal 15wt% /PP Treated	28	2640

Table 2. Cellulose and hemicellulose content of various fiber types

Green Fiber	Cellulose %	Hemicellulose %
Flax	71	18.6-20.6
Hemp	70-74	17.9-22.4
Date palm	46	18
Jute	61-71.5	13.6-20.4
Kenaf	45-57	21.5
Coir	32-45	0.15-0.25
Sisal	66-78	10 – 14.2

Table 3. The complete data set after processing

Cellulose %	Hemicellulose %	Treatment	TS (*MPa*)	TM (*MPa*)
15.8	6.45	YES	42.1	3458
20.4	8.6	NO	22.7	5641
20.4	8.6	YES	32	2889
5.775	0.03	YES	27.5	2305
7.7	0.04	YES	26	3000
13.8	5.4	NO	20	2255
13.8	5.4	YES	27.3	983
10.8	1.815	YES	28	2640
21.3	5.88	NO	33.1	3439
21.3	5.88	YES	43.6	3851
35.5	9.8	NO	24	2500
35.5	9.8	YES	51.5	2700
13.25	3.4	NO	25.8	1680
13.25	3.4	YES	29.8	2185
19.875	5.1	YES	28.7	2250

3. RESULTS AND DISCUSSIONS

Before conducting the RSM analysis, it is often desirable to visualize the data to check for any possible trends and to ensure that the data are normally distributed. Figure 1 shows a scatter plot for the tensile strength versus the amount of cellulose and hemicellulose, regardless of the treatment condition, and Figure 2 shows the same for the tensile modulus response.

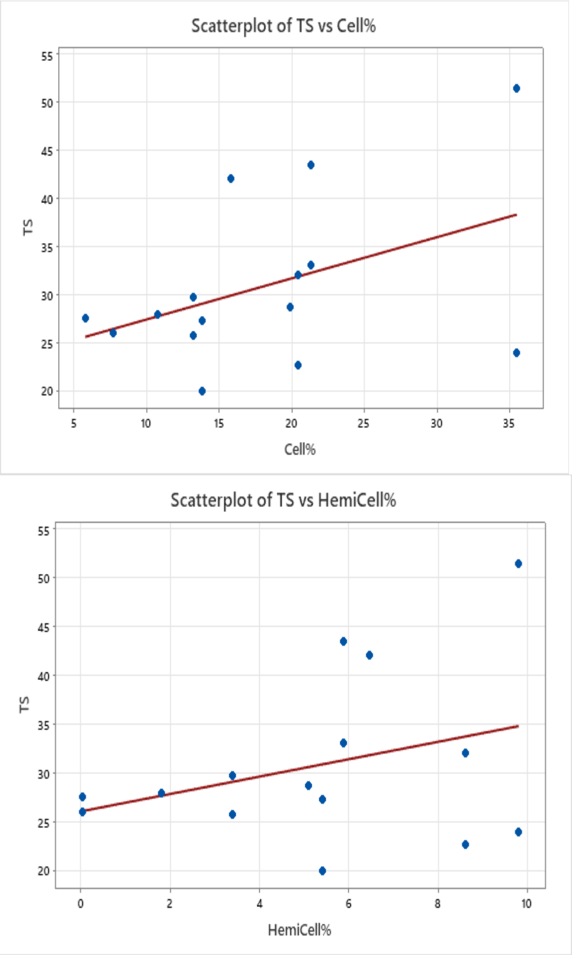

Figure 1. Scatter plot of the experimentally obtained tensile strength versus (up) cellulose and (down) hemicellulose contents

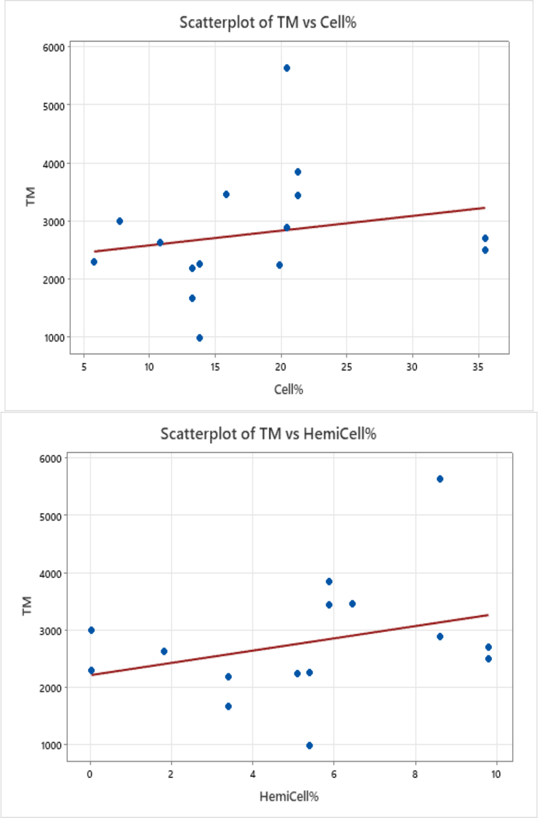

Figure 2. Scatter plot of the experimentally obtained tensile modulus versus (up) cellulose and (down) hemicellulose contents

Both scatter plots show that both mechanical properties data follow very scattered trends and there are no obvious effects that can be conveniently observed. Thus, it is expected to be difficult to obtain a high accuracy regression formula to describe both responses. However, some curve-fitted equations with reasonable accuracy could be achieved. For the normality checks, Figure 3 depicts the normal probability plots for the tensile strength and tensile modulus data with a 95% confidence interval (95 % CI). It is seen that both mechanical properties are following a normal distribution and all points are within the 95% CI except for one data point in the tensile modulus plot. Nonetheless, such one outlier is not expected to significantly affect the analysis especially it is not too far from the 95% CI limits.

As a result, the use of RSM is reasonable and it could lead to accurate analysis and predictions of the main and interaction effects of the cellulose and hemicellulose amount as well as the treatment conditions on both tensile properties of the natural fiber composites.

RSM is used in the current paper to explore the effect of the cellulose and hemicellulose contents and the treatment conditions on the tensile strength and modulus of the natural fiber composites through fitting a first order response surface. The resultant regression equations for the *TS* and *TM* are formulated as:

$$\begin{array}{ll} \text{Treatment: Yes} & TS=26.8+0.56A-2B-0.0012A*B \\ \text{Treatment: No} & TS=19.54+0.895A-0.1B-0.0012A*B \end{array} \qquad (2)$$

$$\begin{array}{ll} \text{Treatment: Yes} & TM=-63-114A+1053B-9A*B \\ \text{Treatment: No} & TM=1442+131A-6B-9.2A*B \end{array} \qquad (3)$$

where, A and B are the cellulose and hemicellulose percentages, respectively. The goodness of fit for the above equations is tested by the value of the coefficient of determinations (R^2) and it is found to be 70% for the *TS* equation and 50% for the *TM* formula. The not too high value of R^2 can be justified due to the limited number of data points, which is the obstacle that such current work try to solve, i.e., predicting the mechanical property trends of the natural fiber composites despite of the shortage in the available data, as well as the huge scatter of the *TS* and *TM* values, as discussed earlier. However, both equations are considered to be satisfactory in the present paper and they would be used for further analysis.

Considering the tensile strength response, Figure 4 shows the main effects of the cellulose and hemicellulose percentages and the treatment status. It is shown that the presence of the cellulose in the specimen has a positive impact on the TS value. In other words, as the cellulose % increases, the TS also increases. In contrast, the hemicellulose amount has a negative influence on the TM values. Additionally, the treated samples have higher tensile strength than the untreated samples. Thus, the treatment can positively improve the tensile strength of the green fiber composites.

Figure 5 shows the interaction effects of the previously described independent variables on the tensile strength properties. In Figure 5(a) the interaction effect between the cellulose and hemicellulose percentages is shown. This subfigure suggested that the cellulose presence can increase the *TS* value while the content of the hemicellulose would reduce the *TS*. In Figure 5(b), the interaction of treatment condition and the cellulose amount is presented. This shows that the cellulose content can positively improve the mechanical strength properties in both treated and untreated samples. However, the cellulose amount has a greater impact on the *TS* in the treated samples as it has a sharper positive slop (red dashed lines) more than the samples without treatment (solid blue lines). Therefore, the treatment is very important when higher tensile strength properties are targeted. The influence of the hemicellulose % and treatment condition interaction is available in Figure 5(c). This effect shows that, in the untreated specimens, deterioration in the TS is expected for higher hemicellulose content (blue solid line). This deterioration can be prevented if the samples are treated (red dashed line). Therefore, the treatment condition may not improve the TS value with hemicellulose presence, but it could prevent the deprivation in the TS due to the hemicellulose higher amount.

Such observations are further confirmed with the contour plots shown in Figure 6 where the effects of the amount of cellulose and hemicellulose at both treatment situations are examined. The contours show that for higher cellulose and lower hemicellulose contents the tensile strength is expected to increase at both treatment conditions. As a result of the previous discussions, it is often desirable to treat the samples properly for higher strength requirements.

For the analysis of the tensile modulus, Figure 7 depicts the main effects of the cellulose and hemicellulose contents and treatment considerations. It can be demonstrated that the cellulose % have a negative influence on the elastic modulus while the hemicellulose content would increase the modulus value. Additionally, the presence of treatment is positively affecting the tensile modulus properties.

For a further consideration of such observations, Figure 8 is plotted to investigate the interaction effects of the variables on the tensile modulus. Considering Figure 8(a), which depicts the interaction effect of the percentages of the cellulose and hemicellulose on TM, it is shown that as the hemicellulose amount increases in the sample, the tensile modulus value also increases. However, when the cellulose content is higher, the TM value is expected to decrease. This negative influence of the cellulose content becomes more significant in samples with higher hemicellulose amount. The effect of the treatment state and the cellulose % is in Figure 8(b).

The results here provide that the higher cellulose amount could reduce the modulus value in the untreated specimens. Nonetheless, that would improve the modulus value in treated samples. Opposite behavior is observed in the interaction between the hemicellulose % and the treatment type, as presented in Figure 8(c). In the untreated situation, the higher content of hemicellulose can significantly increase the tensile modulus properties. However, this would reduce the modulus value in the treated samples, but the deterioration is minor when compared to the untreated conditions. Such behaviors are confirmed by the contour plots of the TM values as shown in Figure 9. In the treated samples, higher cellulose and lower hemicellulose levels can lead to higher tensile modulus values. On the other hand, the lower content of cellulose and higher amounts of the hemicellulose can lead to better tensile modulus properties in the untreated samples.

For the error (residuals) visualization, the standardized residuals are plotted in Figure 10 and Figure11 for the tensile strength and tensile modulus responses, respectively. The residual plots show that the errors between the experimental data and fitted results are normally distributed and have a constant variance (residuals vs fitted values). In other words, the residuals are not related to the independent variable and are randomly distributed over the sample's population. This further confirms the validity of the RSM-based fits for both TS and TM responses.

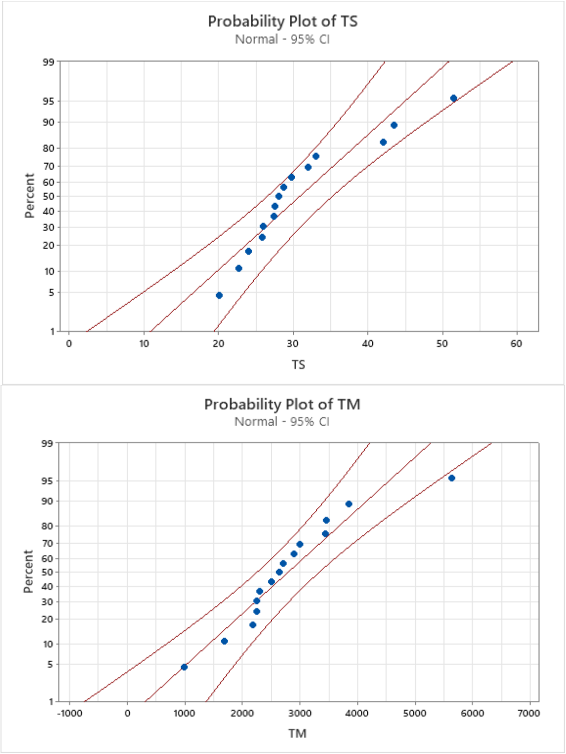

Figure 3. Probability plots for the (left) tensile strength and (right) tensile modulus experimental data

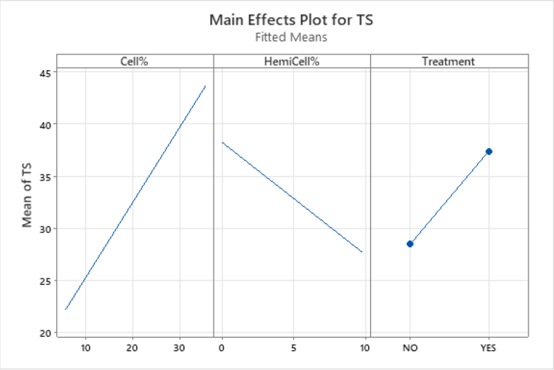

Figure 4. Main effects for the tensile strength

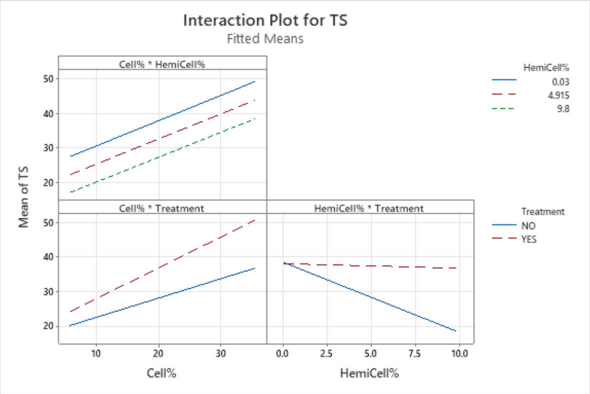

Figure 5. Interaction effects for the tensile strength

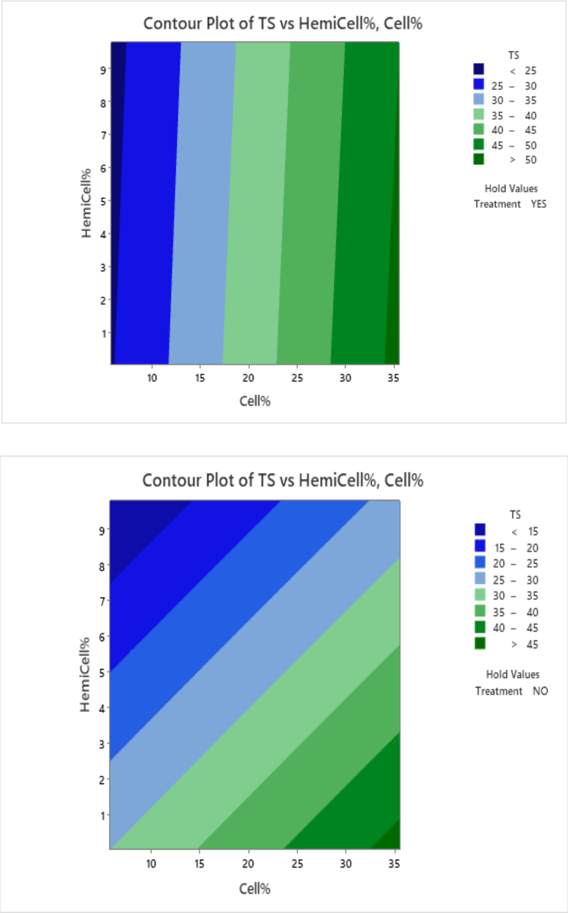

Figure 6. Contour plots for the effect of the cellulose and hemicellulose contents on tensile strength at (up) treated and (down) untreated conditions

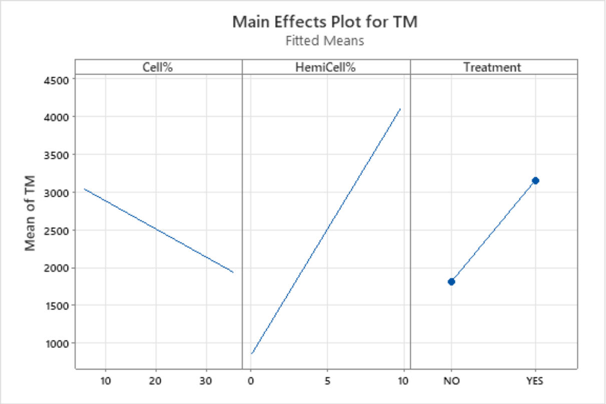

Figure 7. Main effects for the tensile modulus.

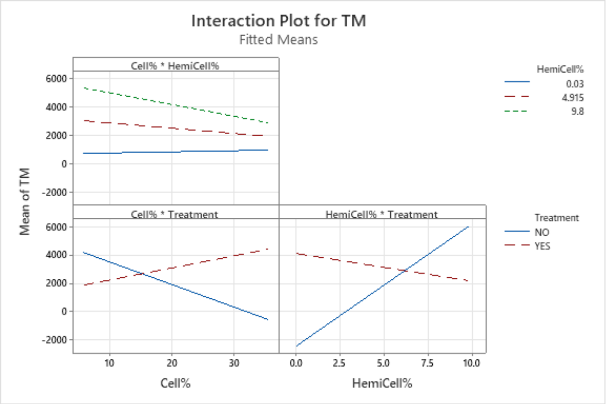

Figure 8. Interaction effects for the tensile modulus

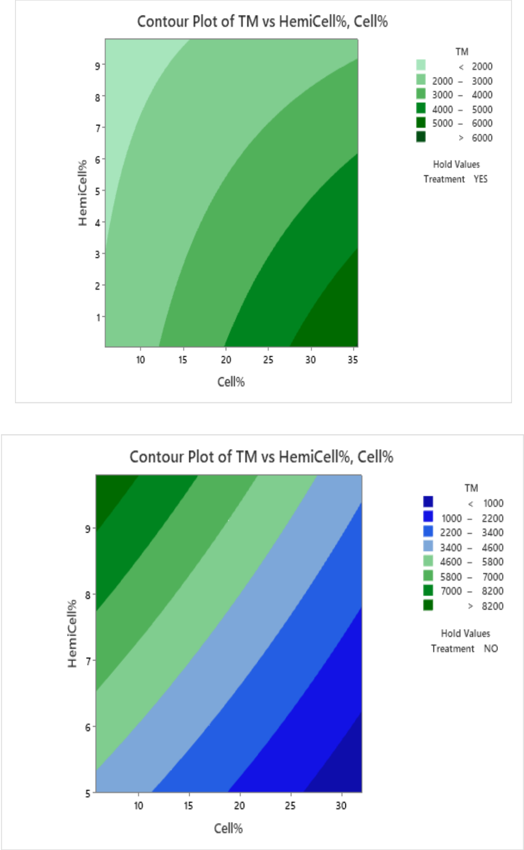

Figure 9. Contour plots for the effect of the cellulose and hemicellulose contents on tensile modulus at (up) treated and (down) untreated conditions

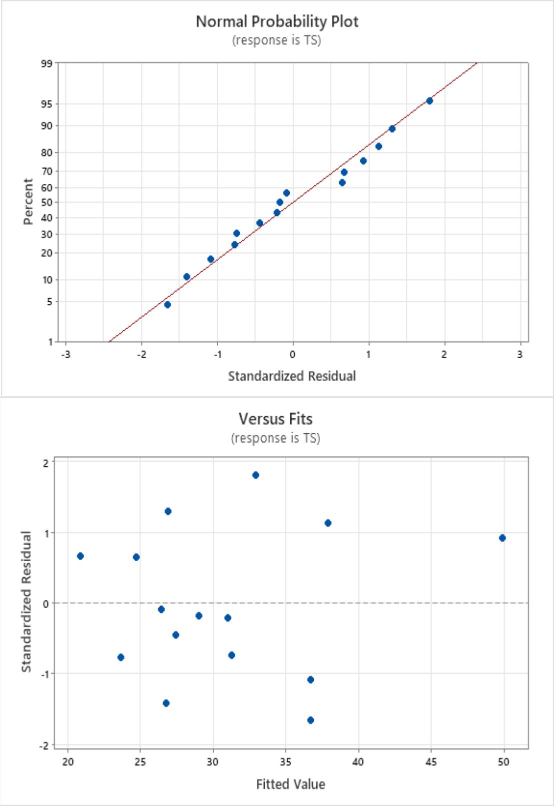

Figure 10. Residuals plots for the TS response

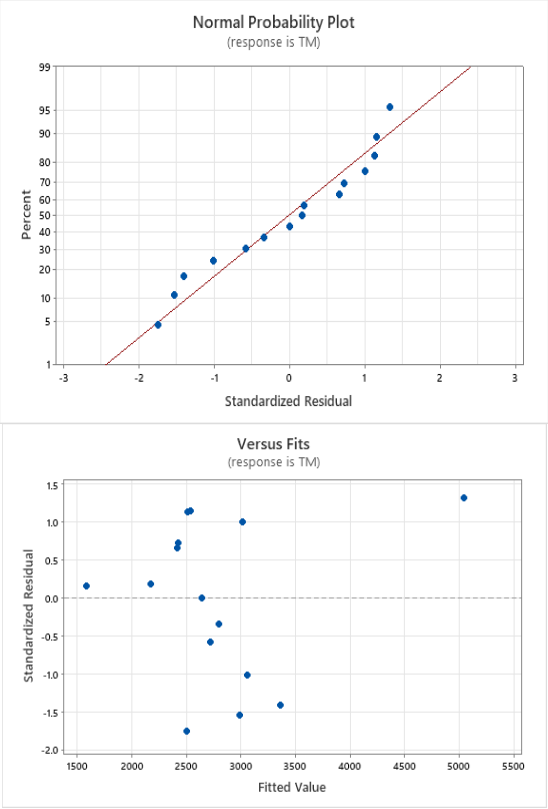

Figure 11. Residuals plots for the TM response

4. CONCLUSIONS

This paper investigated the effect of the cellulose and hemicellulose contents as well as the chemical treatment conditions on the mechanical properties of the fiber-reinforced polypropylene-based composites. The green fiber type, the chemical treatment condition, and the resultant mechanical properties data were collected from literature Consequently, the response surface analysis was applied to obtain a regression model to express the influence of the cellulose, hemicellulose, and the treatment condition on the tensile characteristics of the polypropylene-based green composite. The results showed that the higher cellulose and lower hemicellulose percentages would significantly result in higher tensile modulus values. Nevertheless, this would lead to lower tensile strength values. Moreover, the presence of chemical treatment considerably improves the tensile properties of the polypropylene-based composites.

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