Development of a Mathematical Model Using DOE to Model the Young's Modulus of Biocompatible Palm Fiber / LDPE Composite

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Abstract

There has been considerable research dealing with the use of ecofriendly materials for the development of electronic devices. Within this context, Palm fibers were used to develop a biocompatible composite with a robust mechanical performance. To this end, this study focused on the preparation method of the Palm fiber / Low-Density Polyethylene (LDPE) biocomposite for a potential use as a substrate of acceleration sensor. Following the Design Of Experiments (DOE) methodology 10% and 20% of weight ratio of Palm fiber additive were added to the LDPE matrix to study the mechanical behavior of the proposed composite. A mathematical model describing the Young’s Modulus of the composite was proposed and checked through rigorous experimentation and statistical analysis proposed by the DOE. The resulting biocomposite exhibits improved mechanical properties in terms of Young’s modulus and ultimate strength.

Keywords Biocomposite, Palm fiber, DOE, Mechanical characterization, Substrate

Introduction

As the conventional plastics used in electronic devices have resulted in serious environmental problems, the demand for biocompatible materials has received a huge amount of interest among researchers in the field.

Following this research trend, this book chapter introduced a biocompatible eco-friendly composite material, developed using natural fibers and biopolymer to create substrates for electronic devices. However, the empirical research on the issue is rather scarce. This led us to look for alternative mathematical models that would provide a numerical model for simulation.

In this context, several previous studies have explored the use of various fibers from natural resources and wastes in different industrial fields. Faruk et al. in [1] and Kerni et al. in [2] reviewed numerous natural fibers and reinforced natural fibers composites. Paul, V. et al. studied biocomposites based on fruit and vegetable wastes and their applications in [3]. In [4], Dilawar et Eskicioglu assessed the biodegrability on laboratory and field scales of biocomposite cellphone cases and studied their end of life management. Others conducted research on cement-based building materials. Pacheco-Torgal and Jalali, for instance, investigated the use of natural fibers as reinforcement in cementitious matrix in [5].

Some studies focused specifically on the use of Palm fiber composites in various engineering applications. Date palm fiber was characterized by Elseify et Midani in [6] investigating its morphological, physical, mechanical and thermal properties. In [7] Al-Oqla et sapuan and in [8] Muthalagu et al. explored the applicability of date palm fibers as a reinforcement for polymer composites in industrial automotive applications. In [9], Laasri and Naim evaluated the environmental impact of Palm fiber-based geotextiles. Date palm fibers and their composites processing and properties were covered mainly for packaging purposes in by AL-Oqla et al. in [10].

Researchers also explored the effects of palm fibers modifications and treatments on composites. Sbiai et al. in [11] studied the effect of the palm fiber size on the mechanical and physiochemical properties of epoxy composites. Abdal-hay et al. in [12] studied the impact of alkali treatment as well as the fiber diameter size on the tensile properties of date palm fiber in epoxy composites while Oushabi et al. [13] focused on the effect of this treatment on the mechanical, morphological and thermal properties but took into account the interface of palm fiber Polyurethane composite. Taha et al. further optimized this treatment process for polymeric composites in [14]. Other researchers dealt with different types of treatments on this fiber. Chaari et al. in [15], for example, explained this treatment and its effect on poly(butylene succinate)/palm fibers properties. As for Delzendehrooy et al. [16], they confirmed the improvement of the mechanical strength of adhesively bonded single lap joints with date palm fibers. Their main focus was to showcase the effect of the fibers type, size, treatment method and density effect on the composite strength. Lahouioui et al. further investigated the fiber surface treatment effect on mechanical, thermal and acoustical properties in cementitious composites [17].

Within the same context, several scientists tried to identify the suggested empirical methods for systematic experimentation. Indeed, Erikson et al. [18] illustrated the Design of experiment (DOE) methodologies and explained its approaches for planning, conducting, analyzing and interpreting experiments.

The objectives of this study are to exhibit the mechanical behavior of the conceived material to optimize its proportions to achieve the best performance possible. To this end, and relying on the DOE methodology, a mathematical model of the young's modulus that describes the rigidity in function of mixture proportions was proposed and statistically confirmed. Moreover, the overall mechanical behavior was discussed, the composite preparation method was detailed and the mechanical tests were conducted according to the ASTM standards.

The remainder of this chapter was organized as follows: Section 2 described in details the materials and methods used including the preparation steps, techniques, methodology and experimental setup. Section 3 was devoted to the display of the achieved results relying on the DOE and the statistical verification of the obtained mathematical model. A comprehensive analysis of the composite mechanical behavior and the effect of varying the Palm fibers ratio was also proposed. Section 4 summarized the key findings and the potential applications.

Materials and methods

This section detailed the preparation method of the candidate matrices, the additive and the proposed bio-composite used as the substrate material while relying on the DOE methodology.

Matrix Choice

The matrix choice in this study was made according to the early design propositions of the accelerometer. A quick glance at the literature allows us to note multiple materials classified from the less rigid (around 10MPa) to the more rigid (up to 1GPa) according to Young's modulus complying with the conventional industrial plastic materials databases to test and choose the most suitable to our end purpose. The proposed materials are:

PDMS TR55: Polydimethylsiloxane, LDPE: Low-Density Polyethylene, ABS: Acrylonitrile Butadiene Styrene, Recycled ABS, PP: Polypropylene

The granules of the RAW materials are firstly heated at 80°C for 8 hours to get rid of any humidity that could cause problems and imperfections in the specimens during injection and molding. The materials are then injected under pressure through a nozzle into the mold cavity so as to get dumbbell-shaped test specimens according to the ISO 527-2-1B standard intended for mechanical characterization in traction.

The injection parameters for all prepared materials are displayed in table 1.

Table 1. Injection molding parameters

Parameters	TR55	PP	ABS	Recycled ABS	LDPE
Screw temperature profile (°C)	150-160-170	210-220-230	210-220-230	210-220-230	170-185-190

Dosage back pressure (bar)	1	1	1	1	1
Screw rotation speed (%)	50	50	50	50	50
Mold temperature (°C)	80	35	35	60	25
Injection and maintenance pressure (MPa)	90	90	90	90	90
Cooling time (s)	30	30	30	30	30

Mechanical tests on these materials are conducted to check the elastic modulus values.

Then, several tensile tests are performed in order to study the mechanical properties of the composites produced before and after the addition of fibers and fillers. The plot of the stress curve as a function of the strain shows the behavior of each material. The Young's modulus E (MPa), which reflects the rigidity of the material, is also calculated.

The tensile tests are carried out according to the ISO 527-2-1B standard using a WDW-5 universal tester with a capacity of 5kN. The speed of the tests is kept constant at 20 mm/min.

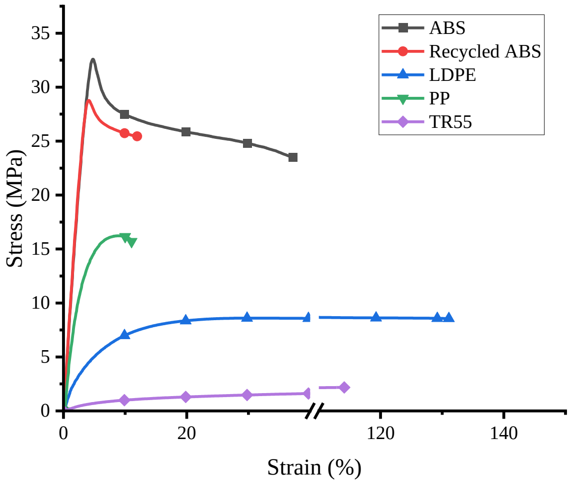

Fig 1. Stress strain curve of candidate matrices

Each polymer shows a specific different behavior (Fig. 1). The TR55 has the lowest Young's modulus and shows a rubbery elastic behavior. The LDPE has a higher Young's Modulus than TR55 and the highest strain at failure of all the materials. It has a plastic behavior due to its substantial plastic domain where it shows a ductile property. The PP curve rises until reaching a yield point toward the plastic domain. PP is less ductile than the LDPE and its plastic domain is minor in comparison with the other two other materials. ABS and recycled ABS curves are almost identical. It is noticeable that they have the smallest elastic domain of all of the proposed materials but still show a ductile behavior and a substantial plastic domain. ABS showed the highest tensile Modulus (Fig. 2).

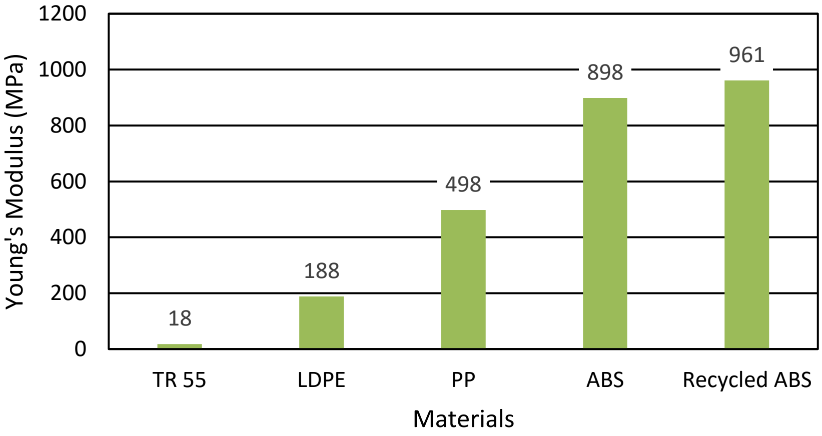

Fig 2. Young's Modulus values of candidate matrices

The proposed hypothesis in this study is that adding palm fiber to the matrix not only contributes as a biodegradable additive but also increases the overall mechanical performance of the obtained bio-composite.

The LDPE was retained as a matrix for the composite to design owing to diverse reasons. To begin with, the study aimed to design a bio-composite that requires flexibility. Besides, the end product should be cost-effective and easy to processing. Moreover, it should be of a good electrical insulation, versatile and able to blend with additives. It is for these reasons that we opted for the LPDE as a matrix. In what follows, the preparation of the palm fiber/LDPE biocomposite was detailed.

Palm fiber / LDPE bio-composite preparation

Design Of Experiment (DOE)

This work relied on the mixture design of experiment. It investigated varying the proportions of two ingredients in different mixtures.

It is worth emphasizing that this study sought to optimize and study the Young's Modulus and thus the responses to model. Therefore, it is the most required indicative criterion in composites in the FEA analysis of a conceived product.

In a second step, the main issue is mathematically formulated as fractions of a mixture where non-negative proportions are added up to yield a unit (or one) (eq. 1). These proportions represent the ingredients expressed in mole, weight or volume fraction.

$$\sum_{i=1}^{q} X_i = X1 + X2 + ... + Xq = 1 \tag{1}$$

where

$$0 \leq Xi \leq 1, i = 1, 2, ..., q \tag{2}$$

where q represents the number of components (or ingredients) and Xi is the fractional proportion of the mixtures' i^{th} ingredient (eq. 2)

Thus, the mathematical model can now be proposed. It translates the dependence or the response that we chose to be the Young's Modulus E and the factors that are the Palm fiber additive and the LDPE matrix. The adequate model in this study is a second degree polynomic (eq. 3).

$$E = b1X1 + b2X2 + b12X1X2 \tag{3}$$

where E is the measured response, X1 is the LDPE proportion, X2 is the palm fiber proportion and bi stands for the coefficients to model.

The third step is to propose the experimental domain which is in this work limited by the proportion of each component. A mixture of equal proportions of the two components is located in the middle of the segment [AB] while the two ends correspond to the two pure substances. In general terms, when M denotes the point associated with the mixture where the component 1 is in proportion X1, it can be written as follows (eq. 4)

$$X1\overrightarrow{MA} + (1 - X1)\overrightarrow{MB} = \vec{0} \tag{4}$$

In other words, the mixture is therefore geometrically identified at the barycenter of the points A and B, assigned the weights X1 and X2 = 1 - X1.

The limits for the palm fiber proportion are set from 0% to 20%.

A graphical representation of the domain is shown in Fig. 3.

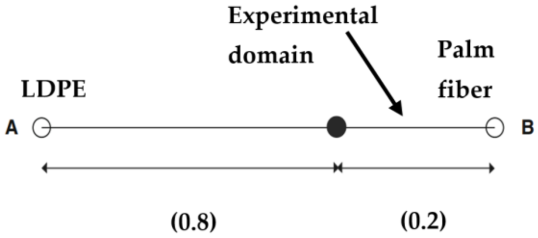

Fig 3. Experimental domain representation of mixture design

The fourth step is to set the experience matrix to follow. To determine this matrix, Minitab software proposes a number of experimental points chosen as being particular points in the experimental field, optimized in our case to be on the edges and in the middle of the domain. These points constitute the set of experiments to be carried out and are called "candidate points".

In this research work, 3 experimental points were proposed, each of which is repeated following the ISO 527-2-1B standard.

The experience matrix is given in Table 2.

Table 2. Experience matrix

Experience	Bloc	LDPE	Palm fiber
1	1	0.8	0.2
2	1	1	0
3	1	0.9	0.1
4	2	0.8	0.2
5	2	1	0
6	2	0.9	0.1
7	3	0.8	0.2
8	3	1	0
9	3	0.9	0.1
10	4	0.8	0.2
11	4	1	0
12	4	0.9	0.1
13	5	0.8	0.2
14	5	1	0
15	5	0.9	0.1

Palm fiber / LDPE preparation method

The already ground palm fibers were sieved to ensure a specific fiber size (between 62µm and 160µm). This is obtained by placing the fibers in the upper part of sieves and the particle classifications are obtained by vibrating the sieves column with a vibrating sieve shaker. The method is conducted following the ASAE S319.3 standard, which describes the sieving method.

The fibers are then dried in the oven before being mixed with the LDPE polymer.

Extrusion produces a homogenized mixture of cellulosic fibers, fillers and a thermoplastic polymer at high temperature. In our case, we aimed at developing mixtures with good dispersion of fibers and filler in the molten polymer matrix.

A co-rotating twin-screw conical extruder type SJSZ35 / 80 was used in order to get extrudates of the proposed mixture proportions. The choice of the temperature profile is based on the technical data sheet of the plastic materials in order to ensure fluidity, on the one hand, and on the thermal degradation of the hemicellulose and cellulose components of the fiber, launched consecutively at 280°C and at 360°C according to thermogravimetric analysis from the literature [19], [20], on the other hand.

The temperature profile of the 6-cylinder heating zones from supply to outlet has been set as follows: 160°C, 160°C, 160°C, 170°C, 180°C et 180°C. This profile has been changed and optimized in order to have a well melted, homogeneous and stable extrudate at the exit of the die. When leaving the die, the extrudate is cooled in a water bath and then dried in an air dryer before being granulated with a rotary cutter mill. After granulation, the obtained composite is dried again (Fig. 4).

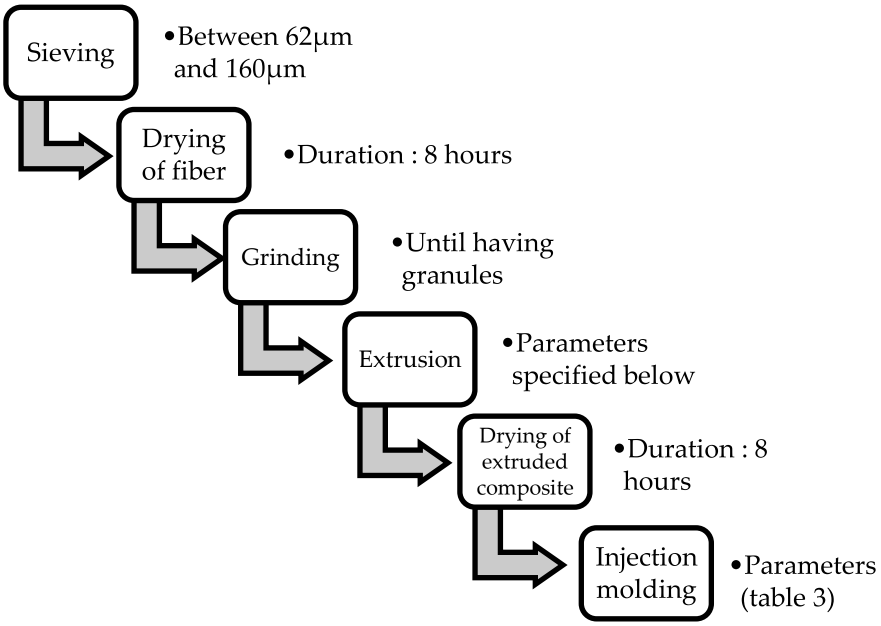

Fig 4. Palm fiber / LDPE composite preparation process

Finally, the specimens for the mechanical test are manufactured by injection molding following the ISO 527-2-1B standard. The process parameters are displayed in Table 3.

Table 3. Injection molding parameters for Palm fiber / LDPE composite

Parameter	Value / profile
Screw temperature (°C)	170-185-190
Dosage back pressure (bar)	1
Screw rotation speed (%)	50
Mold temperature	25
Injection and maintenance pressure (MPa)	90
Cooling time (s)	30

Results and discussion

DOE results

The obtained results achieved through the experiments in accordance with the postulated experience matrix are shown in Table 4.

Table 4. Young's Modulus values following the experiment matrix

Experience	LDPE	Palm fiber	Young's Modulus E
1	0.8	0.2	545
2	1	0	163
3	0.9	0.1	44
4	0.8	0.2	530
5	1	0	186
6	0.9	0.1	505
7	0.8	0.2	529
8	1	0	179
9	0.9	0.1	494
10	0.8	0.2	513
11	1	0	188
12	0.9	0.1	484
13	0.8	0.2	526
14	1	0	194
15	0.9	0.1	449

Based on these results, a mathematical model describing the rigidity of the studied Palm fiber/LDPE biocomposite can be expressed as follows, (eq. 5)

$$y = 186.59x1 - 7540x2 + 11799x1x2 \tag{5}$$

The statistical verification achieved through the use of the least square method allows the evaluation of the quality of the estimates of coefficient estimates.

This method reveals the standard error, the standard deviation and the Variance inflation factor (VIF). The obtained results are displayed in Table 5.

Table 5. DOE evaluation by least squares method

Coefficient	Value	Standard error	Standard deviation	VIF
B1	186.59	8.63	11.6	2.45
B2	-7540	850	24.1474	486.05
B12	11799	1056	5.5701	505.50

The evaluation of the quality of the model was achieved using both correlation coefficients and ANOVA (Analysis Of Variance).

The correlation coefficient is the specific measure that quantifies the strength of the linear relationship between two variables in a correlation analysis. The coefficient is noted R in a correlation report.

The results of these coefficients are shown in Table 6. Indeed, the correlation coefficients have values that are close to 1. Therefore, the proposed model makes it possible to correctly describe the variation of the Young's modulus; hence, the quality of the adjustment inside the tested domain is good. Furthermore, the provisional correlation coefficient R^2 is higher than 0.95, which indicates that the model is valid even outside the tested domain.

Table 6. Correlation coefficients values

R^2 (%)	R^2 adjusted (%)	R^2 provisional (%)
99.13	98.47	96.95

The results given by the correlation coefficients are confirmed through ANOVA. This method consists in converting the sum of squares to mean squares by dividing the sums on the degrees of freedom (Table 7). This allows for the comparison of the obtained ratios to Fisher's critical values and the determination of whether there is a significant difference (eq. 6).

Table 7. DOE evaluation by ANOVA method

R^2	DOF	SS
Regression	2	339653
Residual error	12	2976
Total	14	343264

$$F_{\exp 1} = \frac{\dfrac{SS_{reg}}{dof_{reg}}}{\dfrac{SS_{reg}}{dof_{res}}} \tag{6}$$

$$F_{\exp 1} = 493.194 \tag{7}$$

This $F_{\exp 1}$ value (eq.7) is greater than Fisher's critical value ($F_{0.001}$ (2/12=0.166) = 12.97), which means that the quality of the adjustment is ensured.

The last step of the DOE is to validate the model. This was carried out by calculating the Lack Of Fit (LOF) and comparing it to Fisher's critical value.

To this end, the following numerals are calculated:

- The sum of the squares of the error: sum of the squares of the differences between the experimental values and the mean of the values for the repeated experiments only (eq. 8).

$$SS_{pure\_error} = \sum \left(y_{i0} - \overline{y_0} \right)^2 \tag{8}$$

- The sum of squares related to the LOF: It has to be equal to the difference between the sum of the squares of the residuals and the sum of the squares of the error (eq. 9).

$$SS_{LOF} = SS_{residual\_error} - SS_{pure\_error} \tag{9}$$

- Degrees of freedom of error: number of repetitions of an experiment minus 1.
- Degrees of freedom for lack of fit: the number of total experiments minus the number of repetitions of an experiment minus the number of the model coefficients plus one.

Table 7 summarizes the obtained results.

Table 7. LOF test values

	SS	DOF	Medium square
Regression	339653	2	169826.5
Residual error	4132.18453	12	344.34
LOF	11799	1056	5.5701

Pure error	4052	4	0.09

$$F_{\exp 2} = 3.777 \tag{10}$$

The value of F_{exp2} (eq.10) is lower than Fisher's critical value (F0.05 (8/4=2) = 6.04), which shows that the lack of adjustment is of the same order of magnitude as the pure error which proves that the proposed model is valid.

Mechanical test

In this section, the results of the mechanical tests were analyzed and the impact of introducing the palm fibers on the mechanical behavior of the composite was discussed.

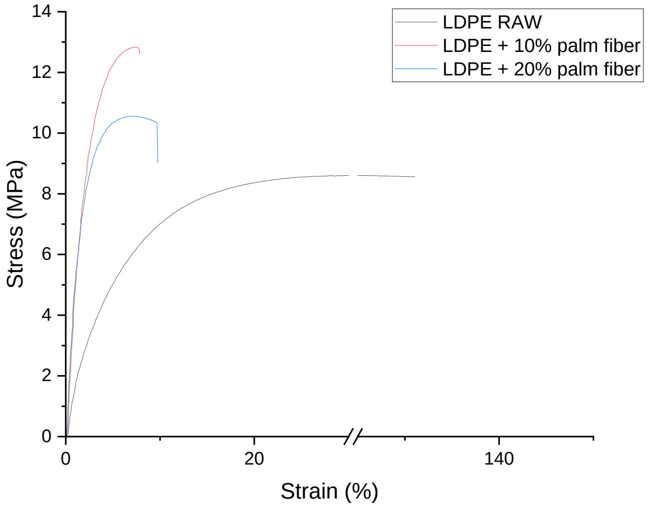

Fig 5. Stress strain curve of the different studied composite ratios

The influence of fibers introduction on the mechanical behavior of the material is noticeable (Fig. 5). Comparing the stress strain curves obtained of the RAW LDPE with the different composites, it can be easily noted that the addition of the filler significantly increased the stiffness of the composites. However, this introduction of the fibers led to a significant reduction in the ductility of the mixtures and to a premature rupture in the plastic zone at low elongation values.

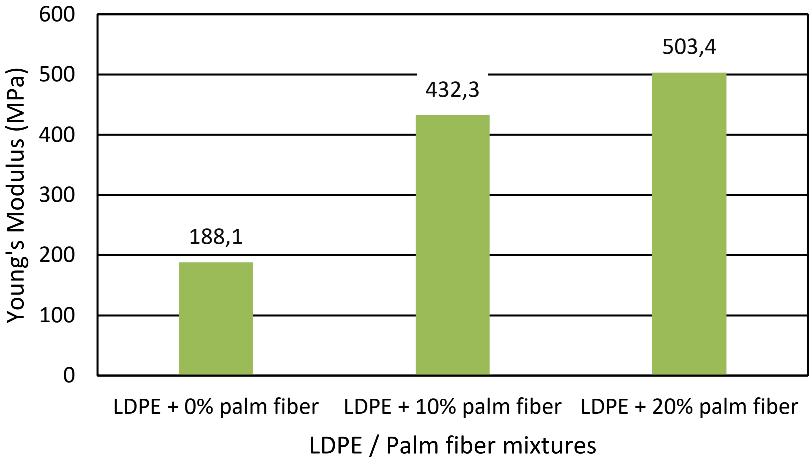

Fig 6. Young’s modulus for different composite ratios

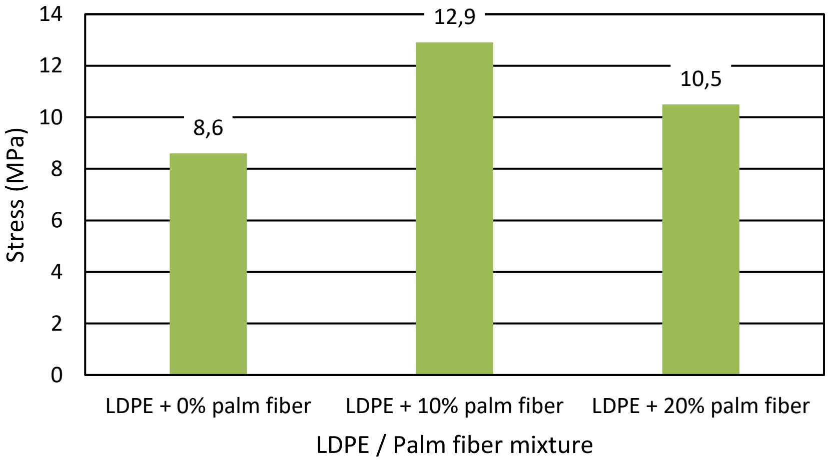

Fig 7. Ultimate strength for different composite ratios

Fig.6 and 7 show histograms of the evolution of the Young's modulus and of the ultimate strength of the different mixtures. It is proven that the addition of palm fibers makes it possible to stiffen the matrix as shown by the increase Young's modulus as a function of the rate of reinforcement. The incorporation of 10% and 20% palm fibers into the LDPE matrix increases the Young's modulus by approximately 230% and 267.5%, respectively. The ultimate strength also goes up from 8.6MPa for the raw LDPE to 13.2MPa and to 10.8MPa for the 10% and 20% mixtures respectively. Finally, the elongation at fracture decreases as the material behavior changes from ductile to less ductile and more fragile.

Composite components effect analysis

The Cox response plot was used to visualize the effects of the components against the reference mixture after being fitted to the model. The plot shows the variations in response as one move along an imaginary line that passes through the reference mixture and the pure component, (Fig. 8)

As the proportion of Palm fiber in the mixture increases, the Young's modulus increases and vice versa.

At a higher proportion of the mixture, the curve behavior indicates a regression in the performance of the mixture. Therefore, it is expected that outside the domain, this response is reversed.

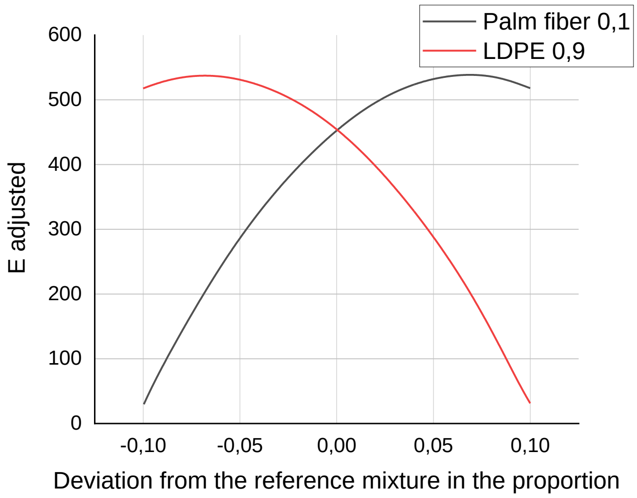

Fig 8. Cox plot

Conclusion

In conclusion, this research dealt with the potential of Palm fiber use as a reinforcement biodegradable material. Relying on the DOE, a comprehensive study of the mechanical behavior of Palm fiber / LDPE bio-composite was conducted.

Numerous outcomes of this work can be outlined. To begin with, an optimized formulation of the proposed composite was achieved. In addition, the substantial increase in the Young's modulus and resulted in a superior mechanical performance and the ultimate strength of the composite. Further, a mathematical model describing the relationship between the Young’s modulus and the bio-composite components proportions was proposed and successfully applied. The model was also validated through statistical analysis to assure the model’s accuracy and reliability.

Future research will involve an in-depth testing of the composite in various electronic devices especially acceleration sensors. In addition, mass production and upscaled manufacturing process can also be subject of further research.

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