

# Analysis of the Effect of Parameters on Fracture Toughness of Bioplastic-Based Starch Reinforcements in Washintonia Fiber and Date Palm Fiber Using Taguchi Method

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Received: 22/10/2023, Accepted: 02/01/2024

## Abstract

In this study, Washingtonian and date palm fibers of different lengths and weights were used as reinforcement materials for starch-based bioplastics, and then the Toughness module of the manufactured samples was measured. It has been discovered that composites reinforced with Washingtonia fibers have greater continuity of Toughness module and resistance compared to composites reinforced with date palm fibers. The difference becomes more apparent when compared to the starch matrix, as the Toughness module of the composites reinforced with Washingtonian fibers improved by 2.3 compared to the starch matrix (Biop). As for the composites reinforced with date palm fiber, it improved by 1.64 compared to the starch matrix (Biop). The Taguchi method was applied to optimize the fiber length, and fiber weight ratio, of bioplastic starch-based fiber reinforced composite. The effect of fiber weight percentage at three levels (2, 5, and 8 wt%) and fiber length at three levels (5, 15, and 25 mm) on fracture toughness was studied using L9 orthogonal arrays. Optimal conditions were found with 2 wt% fiber and 5 mm fiber length. The fracture toughness under optimal conditions was 32,7944 kJ/m<sup>2</sup> for specimens reinforced with Washingtonian fibers (BWS2), and the value reached 23,8722 kg/m<sup>2</sup> for specimens reinforced with date palm fibers (BPS2).

**Keywords:** Bio-Composite, Palm fiber, Starch, Taguchi method, Toughness module, Washingtonian Fiber.

Tob Regul Sci.™ 2024; 10(1): 109 – 125

DOI: doi.org/10.18001/TRS.10.1.8

## Introduction

Composites constructed from synthetic fibers are currently used in a variety of applications by people all over the world. However, these compounds are dangerous to human health and do not biodegrade. As a result, there is ample opportunity to produce composite materials made of natural fibers [1]. The use of fibers extracted from vegetables, plants, and animals is being endorsed due to the growing environmental consciousness to replace synthetic fibers [2]. The development of new materials and technologies is leading to the transition of societies to an environmentally sustainable world because of the need to reduce non-biodegradable waste [3-5]. Natural fibers can more easily replace traditional metallic materials and synthetic fibers due to their many beneficial qualities, including biodegradability, non-toxicity, light weight, high specific strength, strong wear resistance, and inexpensive [2, 3, 6]. Natural fibers are employed as reinforcement in polymer composites for a variety of industrial applications because of these massive properties [2, 7]. Several investigators have been working hard in recent years to meet the materials (high mechanical properties) needs of transportation and industry [8, 9]. Several studies have been conducted to investigate the mechanical properties of natural fibers when mixed with thermoplastics, like PHA and PLA, and thermosets, like polyester, phenol- formaldehyde, and epoxy. Because the efficacy of the reinforcing is determined by the stress transfer between the matrix and fiber, these studies have shown that the bonding between the matrix and the fiber significantly affects the overall mechanical properties of the final biocomposite [10-12]. Natural fiber reinforced composites are employed in a variety of applications in the building, automotive, aerospace, and packaging industries [10, 11]. The characterization of cellulosic fibers, specifically those of the Washingtonia, date palm, sisal, flax, and jute, has been the subject of numerous research projects. The Washingtonia palm, also called the desert palm or California palm, is a flowering plant of the palm family (Arecaceae), developed in colonies in the valleys of arid regions. It is native to the southwestern regions of the United States (California and southwestern Arizona) and northwestern Mexico. It has waxy fan-shaped leaves and a strong vertical stem, reaching a height of 15 to 20 meters and a width of 3 to 6 meters. Furthermore, there are several species of Washingtonia, such as Robusta, Filifera, and Washingtonia filifera (Petticoat Palm) [13-16]. Washington's palm is a fast-growing species that produces a large amount of biomass each year, which is generally disposed of in landfills or burned on site without being exploited, although its fibers have great potential for engineering applications such as furniture and construction [13, 17]. The date palm tree, or Phoenix dactylifera L, is one of the oldest fruit-producing trees in cultivation, having been domesticated in arid regions of North Africa, the Middle East, and the Persian Gulf nations since more than 3,500 years ago, date palm trees can withstand and grow in any climatic zone because they have good resistance to bad climatic conditions, and they can withstand temperatures ranging from -6 to 50 °C, a date palm tree's productive lifespan is 40 to 50 years on average, with a few of them exceeding 100 years [18]. A natural fiber called date palm fiber (DPF) is extracted from the waste material of date palm trees, as a process by-product, DPF is widely available and has a low

processing cost in many countries with significant date production because DPF is environmentally friendly and sustainable, it is becoming more and more accepted as a fiber material in a variety of composites, including bricks, mortar, gypsum composites, concrete, and clay composites [18]. Fracture toughness is the term used to determine how resistant materials are to the extension and propagation of cracks [4, 19]. Due to the increasing demand for biocomposites in various applications, their fracture toughness is expected to play an essential role in the future [10, 20]. Toughness is a crucial mechanical property that must be investigated for all materials used in structural applications [21]. The strength of the cracked structures or components is mostly related to fracture toughness [19]. When compared to the application of fracture mechanics concepts to metals, polymers, and composites is still in a very early stage [10]. For design purposes and in many material failure studies, quantifying fracture is crucial because it is one of the main failure mechanisms of fiber-reinforced composites [22]. The primary obstacle to designing materials for fracture applications is that cracks can alter local stresses to the point where the designer's analysis of elastic stress is not adequate [19]. The fracture toughness values are useful for engineering structures like petrochemical structures, gas and oil pipelines, nuclear pressure vessels, and piping, ships, aircraft, and automotive structures in performance evaluation, material characterization, and quality assurance [10, 12]. The fracture toughness of a composite material has a significant impact on its impact characteristics, because the fracture toughness governs the impact response of the compact material, under impact loading, the composite material can absorb a significant amount of energy in the full spectrum of damage modes, this evaluation is done using the impact test [6]. One of the natural fibers that several researchers have developed for many applications is date palm fiber and Washingtonia, particularly as a material for reinforcement in composites [14, 17]. Genechi Taguchi proposed an orthogonal array-based experimental design in which process variations are reduced and parameters are systematically studied through the conduct of minimal practical experiments [23]. Abdelaziz Lekrine et al [13]. This research examined the mechanical, thermal, and physical properties of WF fiber-reinforced High Density Polyethylene (WFRHDPE) biocomposites at varying WF fiber mass ratios (10%, 20%, and 30%) during the production process. In comparison to 30% WF-based composites, it was found that biocomposites containing 20% of the mass ratio of WF fibers exhibit good mechanical and thermal properties. Malek Ali [24]. Composites made of epoxy-date palm fiber (DPF) have been created using different reinforced DPF ratios. The outcomes of the mechanical tests showed a notable improvement when the DPF ratio in the epoxy matrix was raised. When compared to other composite specimens, the 15% weight of percentage Epoxy-DPF composites shows a high degree of hardness. Manjunath Shettar et al. [25] investigated the impact of nano-fillers on the mechanical characteristics of epoxy-based composites. It was statistically demonstrated that the addition of different filler weight percentages and filler types has a significant impact on the mechanical characteristics of epoxy-based composite materials.

This work aimed to investigate the fabrication of green composites material composed of a Starch matrix derived from corn reinforced with Washintonia and Date Palm fibers, with differences in fiber length and mass percentages in terms of the biocomposition of the fibers used. To characterize the composites, a fracture toughness test was conducted. By performing a methodical analysis, we intend to ascertain whether there are any notable variations in the improvement of toughness between the various composite formulations. The Taguchi method has been used to optimize the parameters of biocomposites to maximize the composites' fracture toughness.

## Materials and Methods

**Matrix.** Corn starch (food grade) supplied in powder by Corn Products NOUNOURS (Algeria) and glycerol (PRODERMA), 100% vegetable, was used to prepare the TPS matrix.

**Reinforcement.** Natural fibers were extracted from the Washintonia and Date Palm plants, which were collected from Ouargla in Algeria. First, Washingtonia filifera fibers are easily extracted manually from the leaves of the Washingtonia tree (Fig. 1).

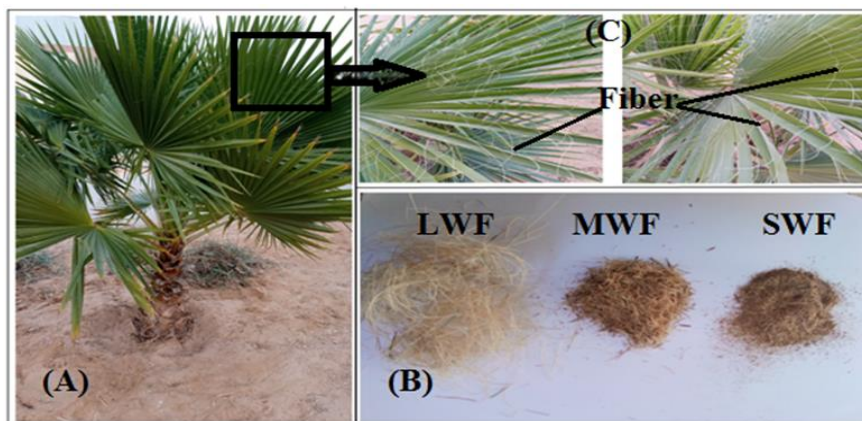


Fig.1. (A): Washingtonian plant; (B): Washingtonian leaf and fibers;

(C): Lengths of fibers used.

As for the date palm fibers, they are extracted from the trunk of a palm tree (Fig. 2). They are then collected and cut into small pieces, then cleaned before being mixed mechanically in an electric blender and sifted to obtain three types of fibers for each type; short fibers (SF) less than 5 mm, medium fibers (MF) less than 15 mm and greater than 5 mm, and long fibers (LF) greater than 25 mm.

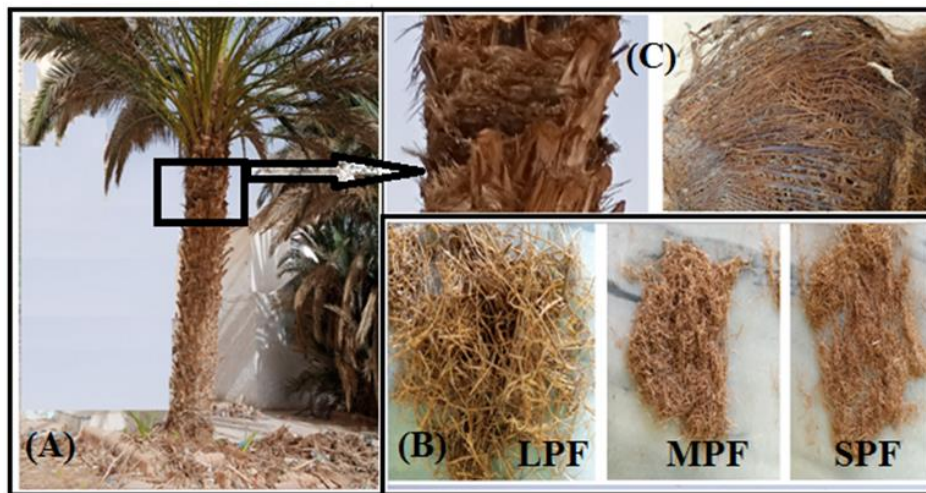


Fig.2 (A): Date Palm plant; (B): Trunk of a palm tree and fibers;

(C): Lengths of fibers used.

**Preparation of bio-plastic and bio-composite.** In this study, bioplastic fibers composites containing 0, 2, 5 and 8% wt/wt of fibers in the matrix were fabricated with the two types of fiber ; Washingtonia fiber (WF) and date-palm fiber (PF). Each type has three different lengths: short fibers (SF), medium fibers (MF), and long fibers (LF). First, to create a bioplastic, glycerol and distilled water were mixed. Next, gradually add the amount of cornstarch and stir until you get a milky white liquid. According to preliminary research, the ideal glycerol content is between 30 and 40% [4, 26, 27]. Next, add the fibers and heat the mixture at 80 °C under continuous stirring for 10 min until it turns into a gelatinous paste [4]. Leave to cool (from 2 to 4 minutes). Then, it is properly kneaded and molded (Fig. 3). Finally, it is left in the open air at room temperature for seven days to dry.



Fig. 3 kneaded and molded.

The following terms (Biop) and (BWS 2, 6, 8), (BWM 2, 6, 8), (BWL 2, 6, 8), (BPS 2, 6, 8), (BPM 2, 6, 8), and (BPL 2, 6, 8) are used to define the Bioplastic non-reinforced (Biop) for

specimens with a composition of 40% glycerol, and Biocomposites containing Washingtonia fiber (WF) and Date-Palm fiber (BP) to matrix as shown in the table 1.

Table 1 Weight percentage (wt%) and fiber length for each composite.

Specimens	Fiber	Tall (mm)	Wight (%)	Specimens	Fiber	Tall (mm)	Wight (%)
Biop	/	/	/	Biop	/	/	/
BWS2	Washingtonia	5	2	BPS2	Palm	5	2
BWS5	Washingtonia	5	5	BPS5	Palm	5	5
BWS8	Washingtonia	5	8	BPS8	Palm	5	8
BWM2	Washingtonia	15	2	BPS2	Palm	15	2
BWM5	Washingtonia	15	5	BPS5	Palm	15	5
BWM8	Washingtonia	15	8	BPS8	Palm	15	8
BWL2	Washingtonia	25	2	BPS2	Palm	25	2
BWL5	Washingtonia	25	5	BPS5	Palm	25	5
BWL8	Washingtonia	25	8	BPS8	Palm	25	8

**Charpy impact tests.** The specimens were prepared in accordance with ASTM-A370 standards. The impact strength was measured using a Charpy hammer pendulum on an impact tester in a machine known as "Karl Frank GmbH Weinheim Birkenau" type "53565" on a 4J hammer. The average of four samples was used to determine the results.

**Taguchi method.** The relative importance of different filler weight ratios, lengths, and fiber types in fracture toughness is statistically examined using Taguchi analysis, using Minitab 19 software. Two control factors that could impact fracture toughness were chosen to create Taguchi's orthogonal array table. The levels and parameters used in this experiment are displayed in Table 2. The planning of the experiment is carried out according to the L9 orthogonal array, and interactions between control variables are taken into account during the analysis of experimental data.

Table 2 process parameters and levels.

parameters	Level 1	Level 2	Level 2
Length (mm)	5	15	25
mass percentages (%)	2	5	8

## Results and discussion

Figure 4 shows the results obtained for the average toughness effect values of the biocomposites for different length and weight ratios of Washington fibers in Charpy tests performed on a series of samples.

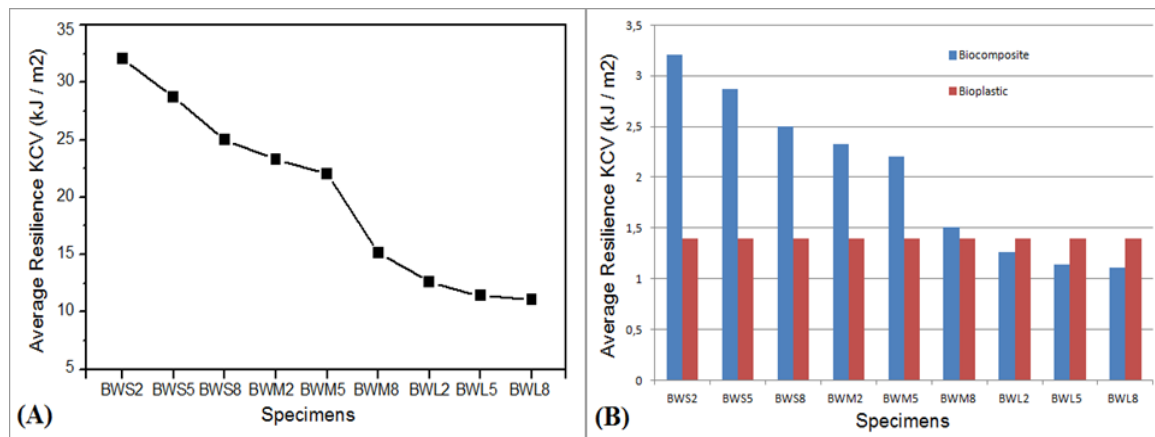


Fig. 4 Average results of the toughness Impact for Starch–WF biocomposites for Washingtonia fiber's various length and weight ratios.

It was revealed that the best results in terms of fiber length were for samples reinforced with short fibers (5 mm), while the best results in terms of weight percentage were for samples reinforced with 2 wt% fibers (Fig. 4. A), This is because of the fibers' excellent random dispersion and strong interfacial adherence to the starch matrix, which prevented cracks from spreading. Also, the increase in the length or weight percentage of the fibers reduces the resistance of the samples to the breaking force. This is evident for samples BWL2, BWL5, and BWL8, where the toughness values were less than the value of the starch matrix (Fig. 4. B). This is due to the high fiber pulling rate of these samples due to the low adhesion between the matrix and the fibers (Fig 5), because there is weak surface bonding in some places and the starch matrix between the fibers has vanished. Similar research revealed that adding WF fibers to the plaster matrix at 2% enhances the novel biocomposite's ductility and mechanical performance [15].



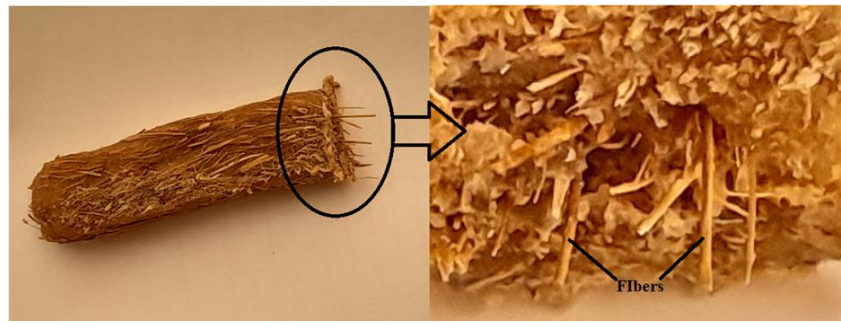


Fig. 5 Macrostructure appearance of sample after impact.

Likewise, Figure 6 displays the findings from Charpy tests conducted on several samples about the average impact hardness values of the biocomposites for various length and weight ratios of date palm fibers.

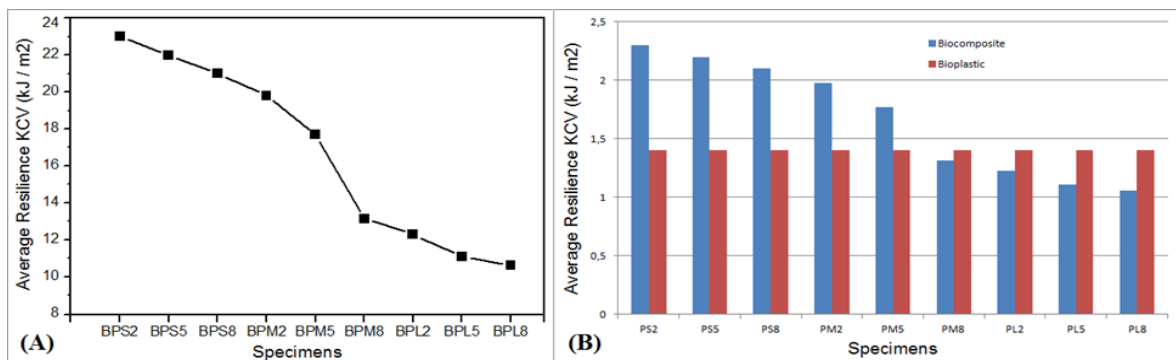


Fig. 6 Average results of the toughness impact for Starch–DPF biocomposites for Date Palm fiber's various length and weight ratios.

It was discovered that samples reinforced with short fibers (5 mm) produced the greatest results in terms of fiber length, while samples reinforced with 2 wt% fibers produced the best results in terms of weight percentage (Fig. 6. A), this is due to the good interfacial adhesion between the starch matrix and the fiber. Higher percentages of the total energy are absorbed when there is strong interfacial adhesion [28]. Additionally, the samples' resistance to the breaking force is decreased by an increase in the length or weight % of the fibers. This can be seen in samples BPM8, BPL2, BPL5, and BPL8 (Fig. 6. B), where the starch matrix value was exceeded by the toughness values. This is due to the presence of weak surface bonding in some places and the disappearance of starch between the fibers. Similar research revealed that when PMMA is mixed with recycled date palm leaves, the resulting samples exhibit improved mechanical and water resistance properties in comparison to composites without these reinforcements [3]. Figure 7 shows the comparative plots of the date palm and Washingtonia fibers' fracture toughness for nine experiments.



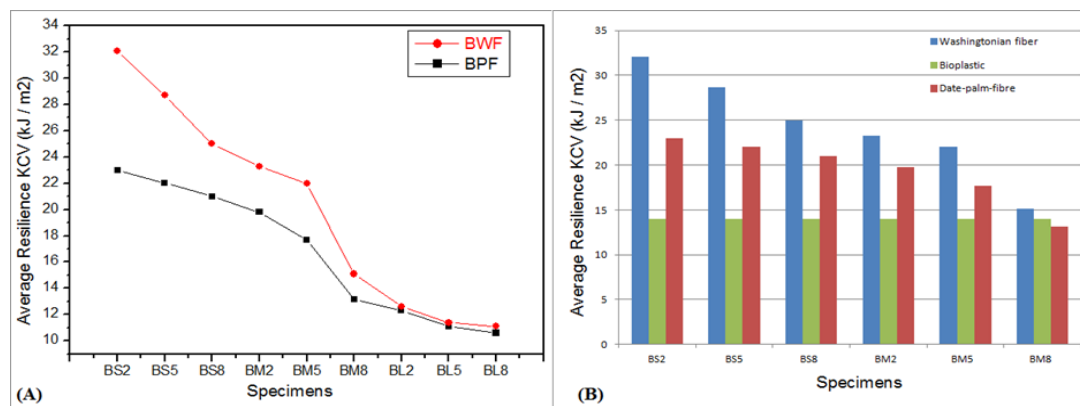


Fig.7 Comparative graph of specimens reinforced (BWF, BPF) with Matrix specimens (Biop).

It was discovered that Washngnonia fiber composite had consistently more durability and resist capability than Date Palm fiber, even when the lengths or mass percentage of the fibers in the samples were changed (Fig. 7. A). The difference becomes more apparent when comparing them to the matrix Biop (Fig. 7. B), as it appears that the greatest improvement was in samples with fiber lengths of 5 mm and a mass percentage of 2 wt%, where the fracture resistance value reached 32.1 kJ/m<sup>2</sup> for the samples reinforced with Washingtonia fibers (BWS2), i.e. an improvement of 2.3 compared to the starch matrix (Biop). The value of 23 kJ/m<sup>2</sup> for samples reinforced with date palm fiber (BPS2), i.e. an improvement of 1.64 compared to the starch matrix (Biop). This is due to the elastic structure of Washingtonia fibers, unlike the hard structure that characterizes date palm fibers, which allows Washingtonia fibers to adhere well to the bioplastic starch matrix without forming clumps or the presence of small air bubbles, thus improving the transfer of stresses. Similar research revealed that the toughness modulus for the Diss fiber-reinforced starch matrix was around 31.25 (KJ/m<sup>2</sup>), which is 2.1 times greater than the value obtained from the starch matrix [4]. In another study, it was found that the addition of different filler weight percentages and filler types has a significant impact on the fracture toughness of epoxy-based composite materials [10].

Following the acquisition of the experimental values by the Taguchi experiment plan, the outcomes underwent analysis. After analyzing the impact of process parameters that can be controlled, such as the length of the fiber (L), and the weight ratio of reinforcement (WR), Tables 3 and 4 present the ranking of the factors that support the signal-to-noise response.

Table 3 Response Table for Signal to Noise Ratios for specimens reinforced with WF Larger is better.

Level	Length	Weight Ratio
1	29,08	26,49

2	25,93	25,71
3	21,35	24,15
Delta	7,73	2,35
Rank	1	2

Table 4 Response Table for Signal to Noise Ratios for specimens reinforced with DPF

Larger is better.

Level	Length	Weight Ratio
1	26,84	24,99
2	24,42	24,24
3	21,07	23,11
Delta	5,77	1,88
Rank	1	2

Tables 3 and 4 show the processing parameters that have an effect on the fracture toughness of the samples reinforced with Washingtonia fiber and date palm fiber, respectively. The most significant effect corresponds to fiber length with an effect of 7.73 for BWF and 5,77 for BPF, and in second place comes the fiber weight ratio with an effect of 2.35 for BWF and 1,88 for BPF. By Taguchi's method, the WR and L levels correlate with the optimum of the response (KCV). Thus, the smallest Weight Ratio WR = 2% and the smallest Length of Fiber L = 5 mm are the levels of the parameters expected under the ideal conditions of the Toughness module (KCV). Figure 8 shows the optimal toughness fracture parameters highlighted in circles.

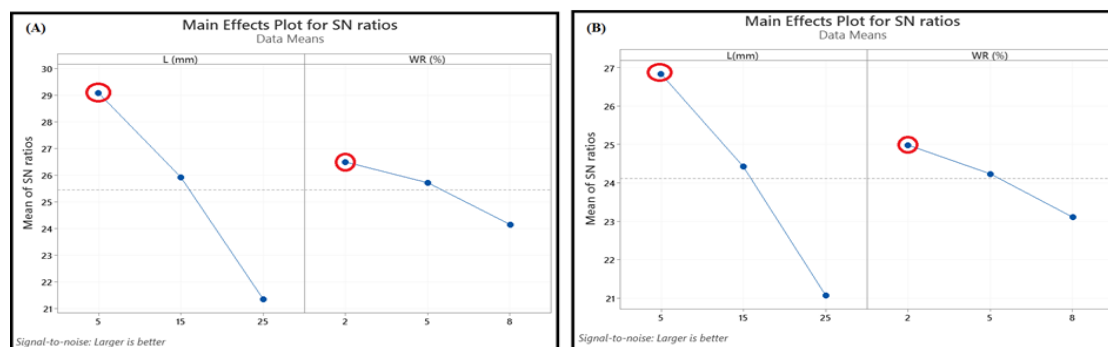


Fig.8 Main effects plot for S/N ratios Toughness module KCV (A: BWF; B: BPF)

By fitting a linear equation to the measured data, multiple linear regression models are developed to provide the relationship between an independent variable (predictor variable) and a response variable. The regression equation for the biocomposite reinforced with Washingtonia fiber (BWF), and reinforced with date palm fiber (BPF) with R-squared  $R-sq=97,88\%$  and  $R-sq=95,51\%$  respectively were generated is

$$KCV_w = 40,99 - 1,078 L - 1,633 WR + 0,0467 L*WR \quad (1)$$

$$KCV_p = 27,80 - 0,546 L - 0,612 WR + 0,0025 L*WR \quad (2)$$

The experimental (measured) toughness values were subjected to an ANOVA that included all potential interactions of the parameters used, as shown in Tables 5 and 6. In this study, a 95% statistical confidence level was employed. This suggests that variables or interactions are deemed insignificant if their P-value is greater than 0.005.

**Table 5 Variance analysis BWF**

Source	DF	Adj SS	Adj MS	F-Value	P-Value
<b>Regression</b>	3	483,295	161,098	77,10	0,000
L (mm)	1	135,035	135,035	64,63	0,000
WR (%)	1	32,928	32,928	15,76	0,011
L (mm)*WR (%)	1	7,840	7,840	3,75	0,110
<b>Error</b>	5	10,447	2,089		
<b>Total</b>	8	493,742			
S = 1,44549      R-Sq = 97,88%      R-Sq(adj) = 96,61%					

Table 5 shows that the first model,  $KCV_w$  (toughness module for BWF), has a P value of 0,000, which is less than 0.05. This indicates that the model fits. Table 5 presents the results, which show that the toughness module (i.e., response) is significantly influenced by the model terms L and WR, with P values of 0,000 and 0,011, respectively. Additionally, the model's R-Sq value demonstrates that 97,88% of the variation in the data was explained by the model, confirming the high reliability of the model that was developed.

Table 6 Variance analysis BPF

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	3	188,543	62,8476	35,48	0,001
L (mm)	1	34,599	34,5988	19,53	0,007
WR (%)	1	4,630	4,6305	2,61	0,167
L (mm)*WR (%)	1	0,022	0,0225	0,01	0,915
Error	5	8,856	1,7712		
Total	8	197,399			
S = 1,33086 R-Sq = 95,51% R-Sq(adj) = 92,82%					

Table 6 demonstrates that the toughness module for BWP, or KCVp, the second model, has a P value of 0.001, which is less than 0.05. This suggests that the model fits the data. The results in Table 6 demonstrate that the model term L has a significant impact on the toughness module (i.e., response) with a P value of 0.007. Furthermore, the model's R<sup>2</sup> value indicates that 95.51% of the variation in the data was explained by the model, demonstrating the high degree of reliability of the developed model.

The predicted toughness strength values for each of the eighteen experimental operations, nine for BWF, and nine for BPF were computed and compared to the measured experimental values.

Table 7 Differences between the toughness strength's measured and predicted values.

Length (mm)	Weight Ratio %	Biocomposite BWF			Biocomposite WPF		
		Measured Experimental (kj/m <sup>2</sup> )	Model Predicted (kj/m <sup>2</sup> )	Error (%)	Measured Experimental (kj/m <sup>2</sup> )	Model Predicted (kj/m <sup>2</sup> )	Error (%)
5	2	32,1	32,79	2,14	23	23,87	3,78
5	5	28,7	28,59	0,38	22	22,07	0,31
5	8	25	24,39	2,44	21	20,27	3,47
15	2	23,3	22,94	1,54	19,8	18,46	6,76

15	5	22	20,14	8,45	17,7	16,73	5,48
15	8	15,1	17,34	14,83	13,15	15,01	14,14
25	2	12,6	13,09	3,88	12,3	13,05	6,09
25	5	11,4	11,69	2,54	11,1	11,4	2,7
25	8	11,1	10,29	7,29	10,6	9,75	8,01
Average				4,83	Average		5,63

Table 7 clearly shows that the average percentage error for the model's prediction of the toughness strength values for BWE and BPF were 4,83% and 5,63%, respectively, from the experimental values. This indicates that for BWF and BPF, the two linear models have prediction accuracy of 95,17% and 94,37%, respectively. This indicates that the toughness results predicted by the models were very close to the actual experimental values.

The interaction effect of the combinations of the independent variables on resilience was further evaluated as shown in Figures 9 and 10. These treatment combinations of independent variables based on experimental design include the interaction effect between the length and weight ratios of fiber. We note that the maximum toughness values obtained are in the range 5 and 7,5 for length (mm) and 2 to 4 for weight ratios (%), for biocomposites reinforced with Washingtonian fibers (BWF). It ranges between 5 and 7.5 in length (mm) and from 2 to 4 by weight percentages (%), for biocomposites reinforced with date palm fibers (BPF).

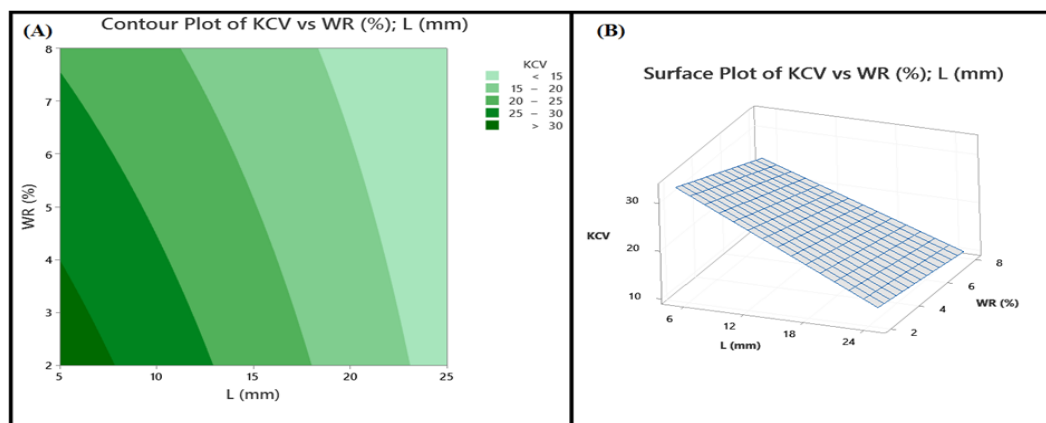


Fig.9 Variation in resilience depends on the Washingtonian fiber's various length and weight ratios (A: contour plot; B: Surface plot).

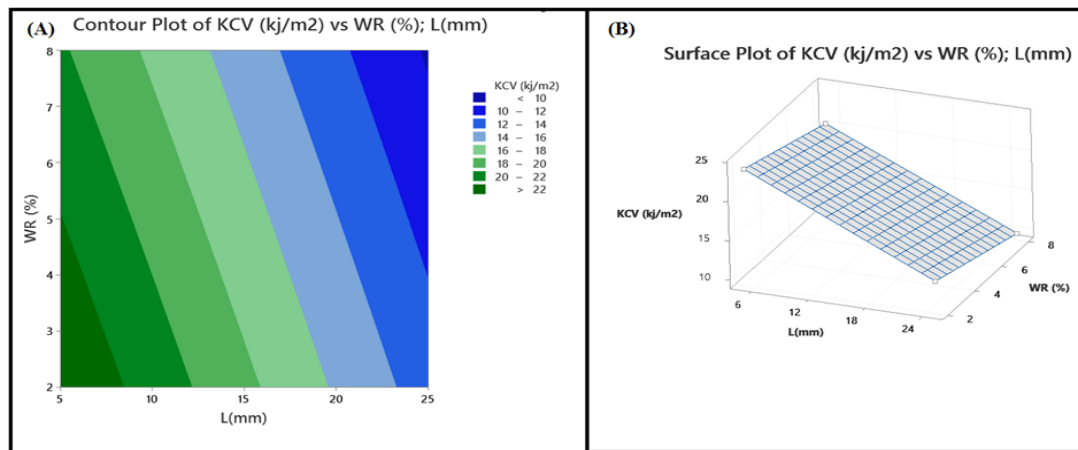


Fig. 10 Variation in resilience depends on the Date Palm fiber's various length and weight ratios (A: contour plot; B: Surface plot).

The execution of the optimization led us to find the optimum values mentioned in Table 8

Table 8 Optimum values.

Biocomposite	Optimal Length L (mm)	Optimal Weight Ratio WR (%)	KCV Fit (kJ/m²)
BWF	5	2	32,7944
BPF	5	2	23,8722

## Conclusion

Washigntonina and date palm fibers reinforced with a bioplastic matrix were used in this study to compare their toughness characteristics.

It was discovered that samples reinforced with short fibers (5 mm) produced the best results in terms of fiber length, while samples reinforced with fibers making up 2% of the sample weight produced the best results in terms of weight percentage. The samples' resistance to breaking force decreases as the length or weight percentage of the fibers increases beyond these points.

It has been found that Washingtonia fiber composites exhibit higher resistance capacity and fracture toughness in comparison to date palm fiber composites. When compared to the starch matrix (Biop), the toughness value (BWS2) increased by 2.3. In comparison to the starch matrix (Biop), the date palm fiber-reinforced samples (BPS2) improved by 1.64.



The most significant effect of the processing parameters that affect the fracture toughness of the samples reinforced with Washingtonia fiber and date palm fiber corresponds to fiber length and in second place comes the fiber weight ratio.

The range of maximum toughness values obtained for biocomposites reinforced with Washingtonian fibers (BWF) is between 5 and 7,5 for length (mm) and 2 to 4 for weight ratios (%). For biocomposites reinforced with date palm fibers (BPF), it varies from 2 to 4 in weight ratio (%) and from 5 to 7.5 in length (mm).

Optimal conditions were found with 2 wt% fiber and 5 mm fiber length. Under ideal circumstances, the fracture toughness of specimens reinforced with Washingtonian fibers (BWS2) was 32,7944 kJ/m<sup>2</sup>, while the value for specimens reinforced with date palm fibers (BPS2) was 23,8722 kg/m<sup>2</sup>.

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