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# Optimization of Bending and Compressive Strength Behavior of *Agave americana* Fiber Reinforced Cementitious Composite Using Response Surface Methodology

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## Abstract

The purpose of this research is to optimize several variables, including fiber length, volumetric fiber percentage, and sodium hydroxide (NaOH) solution concentration, which influence the bending and compression behavior of cementitious composites reinforced with plant-based fibers from *Agave americana*. Samples of composites based on fibers extracted from *Agave americana* and a cement matrix were manufactured and prepared according to a reference mortar. Box-Behnken Design (BBD) of Response Surface Methodology (RSM) was employed to build an experimental design to explore their mechanical behavior and to construct a set of mathematical models predictive of their behavior. The first stage of the work includes the extraction and processing of *Agave americana* fibers and the preparation of cement and *Agave americana* mortar specimens. The second phase aims to establish mathematical models to forecast the workability of the cement mortar and its strength at 28 days while emphasizing the correlations and interactions between the different components through the Design Expert software.

## Keywords

eco-friendly composite, mechanical behavior, mortar cement, extraction, experimental design

# **1** Introduction

Many sectors, including the automotive industry, aerospace companies, building sector, and medical field, incorporate composite materials. A composite material is defined as any alloy or raw material comprising reinforcement in the intimate association of at least two components: the reinforcement and the matrix, which must be compatible with each other and become united. Because the material and the product are generated simultaneously, composite's mechanical properties differ from normal raw materials in that they are only really known after manufacture. The requirements of the construction sector have resulted in significant waste generation, material use, and energy consumption for built infrastructure [1, 2]. The building and construction sector consumes a significant amount of land, energy, and raw materials, as well as notably contributing to environmental pollution, including greenhouse gas emissions [3]. In conjunction with this, the construction sector should consider the use of renewable materials

and industrial by-products to increase sustainability. The reinforcing of concrete with steel is one of the most effective technical applications in civil engineering [4–6]. A cementitious composite is a material comprised of plantbased fibers added to a cement matrix [7-10]. However, due to the prevailing regulations, the survey of these materials kind must increasingly take into account the environmental aspect of their development. Plant fiber and cement matrix composites have been the subject of many investigations in recent years because advantages over asbestos fibers: low cost, healthier processing, renewal, and recycling [8, 10-11], whereas the use of asbestos is challenged by health legislation [12]. For several purposes, including roofing sheets, pipes, wall cladding, and other applications, asbestos cement can be substituted by composite materials reinforced with cellulosic fibers. The choice of reinforcement is still crucial. It must be compatible with the mortar matrix with which it will be used, have acceptable cost,

and have good intrinsic strength properties (elastic modulus, geometry, etc.). Flax, cotton, jute, hemp, sisal, palm, bagasse, ramie, and processed specialty fibers are among the most common plant fibers. As mentioned in the relevant references [13] and [14], natural fibers can be employed in conjunction with their produced counterparts for fiber reinforcing and improving the characteristics of cement mortar. Moreover, plant-based natural fibers can be used in a variety of building industry applications owing to their mechanical and chemical properties [1, 15]. Extensive research that explored the integration of plant-based natural fibers in the concrete industry revealed that the mechanical characteristics of concrete significantly improved [16-18]. Recently researchers have focused on statistical methods for investigating and characterizing the mechanical properties of cement reinforced with vegetable fibers. Khelifa et al. [19] have conducted a very interesting study that aims to carry out an experimental and static study of a mortar reinforced by date palm mesh fibers. In another analysis, the authors proposed an experimental design to statistically analyze the mechano-physical properties of a jute varn-reinforced composite [20]. Moreover, investigations have been made on experimental and statistical analysis based on analysis of variance (ANOVA) of the mechanical properties and the compressive and bending behavior of composites manufactured of cement mortar reinforced with vegetable fibers [21-24]. In Algeria Stipa tenacissima, Ampelodesmos mauritanicus, Cork, and Agave americana are abounding, their harvests and their industrialization are a substantial source of income for some entire populations. In this study Agave americana, fiber is chosen for reasons of availability and economy, comes from a renewable source, and can be integrated rationally in the field of construction. This research was carried out both experimentally and numerically investigations. The work was subdivided into two parts. In the first part, Agave americana fibers were extracted and chemically treated at several concentrations of alkaline. After, specimens of composites based on extracted fibers and a cement matrix were produced and prepared according to a reference mortar. The samples were characterized using standard bending and compression tests. The second part was dedicated to a statistical analysis of obtained results. The effect of some parameters on the mechanical behavior of the processed composites was considered. An experimental design was developed considering the following factors: volumetric fiber length, fiber fraction, and percent of NaOH. Using the response surface methodology (RSM) and based on the Box-Behnken Design (BBD), a set of mathematical models with a predictive character of the essential mechanical properties of the composite has been introduced.

## 2 Materials and methods

## 2.1 Fibers preparation

In this research, we used Agave americana which is among the most responded plant species in Algeria. The fresh blades (Fig. 1(a)) extracted from the plant were collected in the region of Tebessa situated in the extreme east of Algeria. The blades were immersed in a closed water drum for 21 days to separate the fibers from the body of the blade and achieve their extraction. They were then crushed with a blunt knife to remove the outer shell of the plant and release the fibers, as shown in Figs. 1(b) and 1(c). They were subsequently immersed in water for two days at room temperature, then leave to dry for 12 hours at a room temperature of about 25 °C. The extraction was done manually to separate the raw fibers into individual fibers of different diameters and lengths. To improve the physical and mechanical properties of composite systems made of Agave americana fibers and cement mortar matrix, the fibers must undergo several chemical and thermal treatments [25-26]. They were chemically treated with a sodium hydroxide (NaOH) solution, using various concentrations of NaOH (2%. 5%, and 8%), in an attempt to remove impurities, modify their chemical state and their



Fig. 1 Various phases of extraction of *Agave americana*: a) Blades used in fibers extraction, b) Crushing of the fibers, c) Extraction fibers, d) *Agave americana* fibers before drying, e) *Agave americana* fibers after drying

surface state in such a way as to give them better compatibility with the matrix. To get a neutral PH, the fibers were submerged in a 0.5% sulfuric acid solution for 10 minutes and washed at the end with distilled water [27] (Fig. 1(c)). After all these processes, the fibers were dried (Fig. 1(d)) and stored in hermetic bags after being sliced into chunks of 5, 10, and 15 mm in length.

## 2.2 Response surface methodology

Response Surface Methodology is comprised of a set of mathematical and statistical techniques that are based on fitting empirical models to experimental data obtained from the experimental design [28]. It was originally conceived and developed by Box and Wilson [29-30] and has been used with great effectiveness in a wide range of scientific disciplines. RSM has several advantages, such as effectiveness in predicting the model for each response, building a robust model with a small number of experimental data points, assessing the interaction effect between factors, and localizing the optimal response [31]. In this methodology, regression analysis is used in conjunction with statistical techniques to display the overall experimental data set and predict the relationships between a response of interest, y (dependent variable), and several related input or control variables, denoted by  $x_1$  through  $x_k$ (independent variables) [31-33]. This experimental design is characterized by a limited calculation interval. The used levels, labeled by the codes (-1) and (+1), signify, respectively, the minimum and the maximum of the level values assigned to the factors which are centralized about a middle value (0). In this work, bending and compression tests, on a bio-composite consisting of cement mortar reinforced with Agave americana fibers were performed using the parameters listed in Table 1. Based on the methodology RSM, the parameters that were used to build the experimental design were: fiber length (A), volumetric fiber fraction (B), and % of NaOH (C). The Box-Behnken Design used in this study was shown in a previous study to be significantly more effective than the full three-level factorial designs and the central composite designs [34]. The RSM technique provides regression models that may be used to determine the influence of factors and their levels on the

mechanical properties (in this case bending and compressive stresses). In this study, a fitting analysis suggested the implementation of a model involving a quadratic function equation. The second-order polynomial equation most widely used in fitting the experimental data and determining the relevant model terms is expressed as follows:

$$Y = a_0 + \sum_{i=1}^{k} a_i X_i + \sum_{i=1}^{k} a_{ij} X_i^2 + \sum_i \sum_{i=1}^{k} a_{ij} X_i X_j + \varepsilon_i , \qquad (1)$$

where *Y* represents the predicted response, i.e., the bending and compression strength,  $a_0$ , the constant coefficient,  $X_i$ and  $X_g$  the variables,  $a_i$ , the ith linear coefficient of the input factor  $X_i$ ,  $a_{ii}$ , the *i*<sup>th</sup> quadratic coefficient of the input factors  $X_i$ ,  $a_{ij}$ , the different interaction coefficients between input factors  $X_i$  and  $X_j$  (I = 1-3; j = 1-3), and  $\varepsilon_i$ , the error of the model [35]. Instead of performing a test campaign consisting of 27 experiments for the bending test and a further 27 for the compression test, this number can be reduced to just 17 for each of these two tests, by selecting and implementing an experimental design with RSM and BBD Design and using the Design expert software. Table 2 illustrates the levels and factors involved in this experimental design.

# 2.3 Preparation of the bio-composite

The specimens were produced and prepared according to a reference mortar (control) conforming to the European standard EN 196-1 [36]. For the control mortar, the composition of cement, sand, and water is 1:3: 0.5 with  $450 \pm 2$  g of cement,  $1350 \pm 5$  g of sand, and  $225 \pm 1$  ml of water. Different volumetric percentages of fibers: 0.5, 1, and 1.5% treated at different concentrations of alkaline: 2, 5, and 8% and clipped to different lengths: 5, 10, and 15 mm were added to the mixture (decreasing the weight of sand each

 Table 1 Various parameters are used to produce bio-composite

 consisting of cement mortar reinforced with Agave americana fibers

Factors	Unit	Symbol	Min level (–1)	Medium level (0)	Max level (+1)
Fiber length	mm	A	5	10	15
Fiber fraction (% V)	%	В	0.5	1	1.5
NaOH	%	С	2	5	8

Table 2 The levels and factors involved in the construction of the experimental design adopted in this study

Run	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Fiber length (mm)	10	15	5	15	10	5	10	10	10	15	5	10	5	10	10	10	15
Fiber fraction (% V)	0.5	0.5	1	1.5	1	1.5	1.5	1	1.5	1	0.5	1	1	1	0.5	1	1
NaOH (%)	8	5	2	5	5	5	2	5	8	2	5	5	8	5	2	5	8

time). The process of adding the fibers to the mixture was carried out by adding and mixing the treated fibers manually and gradually for five minutes to promote and facilitate the dispersion of the fibers in the mixture. The samples were placed inside steel molds sized  $40 \times 40 \times 160$  mm (Fig. 2(a)). Using a shock table, they were subjected to 60 blows in two phases to eliminate air bubbles (Fig. 2(b)). The specimens were then placed for 24 hours in a humid room at a temperature of 20 °C and a humidity of 90.5% (Fig. 2(c)). After demolding, they were left in the water to cure until ready to be tested after 28 days. According to European Standard EN 196-1, compressive strength (Fig. 3(b)) and three-point bending tests were conducted (Fig. 3(a)). The different types of specimens, as indicated in Fig. 2, were all tested at least three times in both compression and bending. The tests were carried out at room temperature using a universal ToniPrax 1543 machine equipped with a 10 kN load cell at a speed of 2 mm/min. The testXpert software was deployed to collect the results.

# **3** Results and discussion

## 3.1 ANOVA and regression models

The average results from the experimental bending and compression tests (breaking stresses), achieved for the manufactured cementitious composite (*Agave americana* fibers reinforced mortars) and carried out according to the pre-



(a) (b) (c) Fig. 2 Preparation of reinforced mortar: a) Steel prismatic mold of  $40 \times 40 \times 160$  mm, b) Shock table, c) Humid room

established experimental design are reported in Table 3. All the responses (bending and compression stresses) were analyzed separately using the polynomial regression models presented in Table 4, which are: linear, two-factor interaction (2FI), quadratic, and cubic. These models were statistically evaluated by determining the most significant variables that affect the mechanical characteristics of the mortar reinforced with bio-fibers. In Tables 5 and 6, respectively, the initial ANOVA outcomes for the bending and compression strengths can be seen. The sum of squares and mean square values for each parameter are displayed in the ANOVA tables, and the p-value and F-value are defined as the ratio of the respective mean square effect and mean square error. In this research, we used a Box-Behnken Design, which provides for the development of a test campaign made of a set of 17 tests, including five points corresponding to the model's center. The number of trials ranged from 1 to 17. The planning matrix shown in Table 2 covers different combinations of the factors: fiber length (A), volumetric fiber fraction (B), and percentage of NaOH (C). Mathematical models that depict the responses of bending stresses (Y1) and compression stresses (Y2) as a function of the independent variables A, B and C were fitted using the results from the experimental trials conducted on the samples. Before excluding insignificant terms, the predictive models are expressed in terms of coded variables in the following equations:

$$Y_1 = 3.90 - 0.44A - 1.93B - 0.54C + 0.075AB$$
  
-0.15AC + 0.37BC - 0.18A<sup>2</sup> + 1.20B<sup>2</sup> - 0.025C<sup>2</sup>, (2)

and

$$Y_2 = 20.10 - 2.01A - 9.69B - 1.82C + 0.025AB$$
  
-0.55AC + 0.70BC + 0.088A<sup>2</sup> + 7.44B<sup>2</sup> + 0.36C<sup>2</sup>. (3)

The suitability of the regression models to explain the experimental data at the 95% confidence level was explored from the ANOVA results. The significance of the



Fig. 3 Picture of a) 3-point bending setup, b) specimen after bending test, c) compressive strength setup, d) specimen after compressive strength test

Run	Factor 1	Factor 2	Factor 3	Bending (MPa)	Compression (MPa)
	fiber length A: (mm)	fiber fraction B: (% V)	NaOH C: (%)	- · ·	
Control	0	0	0	7.1	37.3
1	10	0.5	8	6.2	34.7
2	15	0.5	5	6.1	35.6
3	5	1	2	4.4	23.8
4	15	1.5	5	2.8	16.1
5*	10	1	5	3.9	20.1
6	5	1.5	5	3.6	19.6
7	10	1.5	2	3.2	19.7
8*	10	1	5	3.9	20.1
9	10	1.5	8	2.7	16.9
10	15	1	2	3.9	20.4
11	5	0.5	5	7.2	39.2
12*	10	1	5	3.9	20.1
13	5	1	8	3.8	21.8
14*	10	1	5	3.9	20.1
15	10	0.5	2	8.2	40.3
16*	10	1	5	3.9	20.1
17	15	1	8	2.8	16.2

Table 3	Experimental	design and	l response	settings for	bending and	compression	tests through RS	M Box-Behnken
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\*Five points in the center of the model.

 Table 4 Fit summary of the two statistical models (bending stress and compressive stress)

Test type	Source	SD	R <sup>2</sup>	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	PRESS	Observation
Bending							
	Linear	0.74	0.8233	0.7825	0.6563	13.98	
	2FI	0.81	0.8399	0.7483	0.2865	29.02	
	Quadratic	0.24	0.9904	0.9779	0.8456	6.28	Suggested
	Cubic	0.00	1.0000	1.000	_	+	Aliased
Compression							
	Linear	4.30	0.7708	0.7180	0.5835	437.52	
	2FI	4.87	0.7739	0.6382	0.1010	944.47	
	Quadratic	0.40	0.9989	0.9976	0.9830	17.88	Suggested
	Cubic	0.00	1.0000				Aliased

SD: Standard deviation

PRESS: Predicted sum of squares

principal effects and interaction effects in the predicting models was considered based on their probability values (p-values). P-values less than 0.05 require rejection of the null hypothesis denoting that the particular term significantly affects the measured response of the system [34].

From the ANOVA Tables 5 and 6, "F-value" of the models is 79.82 for bending and 730.42 for compression respectively, which implies that the models are significant. There is only a 0.01% chance that the model could occur due to noise [19–23]. Probability values less than 0.05 indicate that the model terms are significant [35]. In the case of the bending stresses, the factors A, B, and C, the interaction BC, and the quadratic effects B2, are significant terms in the model. The P-value obtained for the interaction of type AB is 0.5466 (>0.05), type AC is 0.2457(>0.05), type A<sup>2</sup> is 0.1732 (>0.05) and C<sup>2</sup> is 0.8347(>0.05), there is, therefore, no significant effect. As well as the compression, the factors A, B, C, the interaction BC, and the quadratic effect B<sup>2</sup> are significant terms in the model. The P-value obtained for the interaction of type AB is 0.9039 (>0.05), type A<sup>2</sup> is 0.6668 and C<sup>2</sup> is 0.1050, there is therefore no significant effect.

Source	Sum of squares	Df	Mean square	F-Value	P-Value Prob > F	Observation
Model	40.28	9	4.48	79.82	< 0.0001	Significant
A-Length	1.53	1	1.53	27.31	0.0012	
B-Volumetric fraction fibers	29.64	1	29.64	528.70	< 0.0001	
C-NaOH	2.31	1	2.31	41.22	0.0004	
AB	0.023	1	0.023	0.40	0.5466	
AC	0.90	1	0.090	1.61	0.2457	
BC	0.56	1	0.56	10.03	0.0158	
$A^2$	0.13	1	0.13	2.30	0.1732	
$B^2$	6.06	1	6.06	108.13	< 0.0001	
$C^2$	$2.632 \times 10^{-3}$	1	$2.632 \times 10^{-3}$	0.047	0.8347	
Residual	0.39	7	0.056			
Lack of Fit	0.39	3	0.13			
Pure Error	0.00	4	0.00			
Cor Total	40.68	16				
Fit Statistics	Std Dev = 0.24	$R^2 = 0.9904$				
	Mean = 4.37	Adjusted $R^2 = 0.9779$				
	C.V. % 5.42	Predicted $R^2 = 0.8456$				
	Adeq Precision = 2	9.389				

 Table 5 Initial ANOVA results and statistical parameters for bending stress

Table 6 Initial ANOVA results and statistical parameters for compression stress

Source	Sum of squares	df	Mean square	F-Value	P-Value Prob > F	Observation
Model	1049.46	9	116.61	730.42	< 0.0001	Significant
A-Length	32.40	1	32.40	202.96	< 0.0001	
B-Volumetric fraction fibers	750.78	1	750.78	4702.88	< 0.0001	
C-NaOH	26.64	1	26.64	166.90	< 0.0001	
AB	$2.500 \times 10^{-3}$	1	$2.500  imes 10^{-3}$	0.016	0.9039	
AC	1.21	1	1.21	7.58	0.0284	
BC	1.96	1	1.96	12.28	0.0099	
A <sup>2</sup>	0.032	1	0.032	0.20	0.6668	
$B^2$	232.91	1	232.91	1485.95	< 0.0001	
$C^2$	0.55	1	0.55	3.47	0.1050	
Residual	1.12	7	0.16			
Lack of Fit	1.12	3	0.37			
Pure Error	0.000	4	0.000			
Cor Total	1050.59	18				
Fit Statistics	Std Dev = 0.40	$R^2 = 0.9989$				
	Mean = 23.81	Adjusted $R^2 = 0.9976$				
	C.V. % 1.68	Predicted $R^2 = 0.9830$				
	Adeq Precision = 7					

The  $R^2$ , adjusted  $R^2$ , and predicted  $R^2$  determination coefficients were assessed to accurately analyze the regression analysis.  $R^2$  represents the percentage of overall response variation that the models have predicted. The models' suitability and the precision of the derived values are evidenced

by correlation coefficients that are close to 1 [19]. When a new term is introduced, adjusted  $R^2$  can be used to avoid probability errors and is a useful tool for comparing the explanatory power of models with various numbers of predictors. Regression analysis uses the predicted  $R^2$  to

illustrate how well the model predicts responses for new observations. Because it is derived from observations not included in the model estimation, the predicted R<sup>2</sup> could be more useful for comparing models than the adjusted R<sup>2</sup>. The coefficients of determination R<sup>2</sup> and adjusted R<sup>2</sup> are indicative of the adequacy of the polynomial fit and must be approximately 0.20 of one another, to be in reasonable agreement [19]. The two models have high coefficients of determination ( $R^2 = 0.9904$  for bending stresses and  $R^2$ = 0.9989 for compression stresses). The adjusted  $R^2$  will commonly decrease if statistically insignificant factors are incorporated into the model. When the R<sup>2</sup> and adjusted  $R^2$  are significantly different, there is a strong probability that non-significant terms are included in the model [30]. According to this study, for the first response  $(Y_1)$ , the predicted and adjusted R<sup>2</sup> values are close to 1.00, 0.8456, and 0.9779, respectively, suggesting that the predicted and experimental bending stresses are in excellent agreement. This model can reveal 99.04% of the variability, according to the excellent agreement between the  $R^2$  and the experimental values, which is 0.9904. Likewise, the values of R<sup>2</sup>, predicted R<sup>2</sup>, and adjusted R2 for the second response (Y<sub>2</sub>) are, respectively, 0.9989, 0.9830, and 0.9976, indicating a good correlation between the predicted and observed values.

A model is generally regarded as credible if its coefficient of variation (CV) is not higher than 15% [30]. The (CV) is calculated as the ratio of the standard error of the estimate to the mean value of the observed response. Because of this, the obtained coefficient of variation values in this work of 5.42% for bending stresses and 1.68% for compression stresses indicate that the experiments were highly precise and reliable.

The ANOVA was then re-run after removing non-significant terms and the results for bending and compression are reported in Tables 7 and 8. The adequacy of the regression models to interpret the experimental data at the 95% confidence level was examined using the ANOVA results. Finally, based on the final ANOVA for two responses  $Y_1$  and  $Y_2$ , as well as interactions with significant effects, a fitted regression model in terms of coded variables with statistical significance can be reported in the following equations:

$$Y_1 = 15.4375 - 0.0875A - 14.6111B - 0.42917C$$

$$+0.25BC + 4.75556B^2$$
(4)

and

$$Y_2 = 77.09167 - 0.21917A - 81.10833B - 0.70833C$$
  
-0.036667AC + 0.46667BC + 29.85B<sup>2</sup>. (5)

Figs. 4(a) and (b), respectively, show the normal probability plot of the residuals for the two responses  $(Y_1)$ and  $(Y_2)$ . The difference between the expected values and the actual values (residuals), which are predicted to follow a normal distribution, should be used to estimate the model's accuracy. Figs. 4(a) and (b)'s data should be distributed uniformly out along a 45-degree line. The positions [30–31] are rather near to a straight line. The models are appropriate for all evaluated responses, as shown by the straight lines derived for the curves, which show that the studied residual follows a normal linear distribution.

Source	Sum of squares	Df	Mean square	F-Value	P-Value Prob > F	Observation
Model	40.04	5	8.01	137.86	< 0.0001	Significant
A-Length	1.53	1	1.53	26.36	0.0003	
B-Volumetric fraction fibers	29.64	1	29.64	510.41	< 0.0001	
C-NaOH	2.31	1	2.31	37.79	0.0004	
BC	0.56	1	0.56	9.68	0.0158	
$\mathbf{B}^2$	5.99	1	5.99	108.07	< 0.0001	
Residual	0.64	11	0.058			
Lack of Fit	0.64	7	0.091			
Pure Error	0.000	4	0.000			
Cor Total	40.68	16				
Fit Statistics	Std Dev =0.24	$R^2 = 0.9843$				
	Mean =4.37	Adjusted $R^2 = 0.9772$				
	C.V. % 5.51	Predicted $R^2 = 0.9377$				
	Adeq Precision = 3	6.319				

Table 7 Final ANOVA results and statistical parameters for bending stress

			-	-					
Source	Sum of squares	df	Mean square	F-Value	P-Value Prob > F	Observation			
Model	1049.46	6	174.81	1015.60	< 0.0001	Significant			
A-Length	32.40	1	32.40	188.24	< 0.0001				
B-Volumetric fraction fibers	750.78	1	750.78	4361.84	< 0.0001				
C-NaOH	26.64	1	26.64	154.80	< 0.0001				
AC	1.21	1	1.21	7.03	0.0243				
BC	1.96	1	1.96	11.39	0.0071				
$\mathbf{B}^2$	235.86	1	235.86	1370.28	< 0.0001				
Residual	1.72	10	0.17						
Lack of Fit	1.72	6	0.29						
Pure Error	0.000	4	0.000						
Cor Total	1050.58	4							
Fit Statistics	Std Dev = 0.41	$R^2 = 0.9984$							
	Mean = 23.81	Adjusted $R^2 = 0.9974$							
	C.V. % 1.74	Predicted $R^2 = 0.9913$							
	Adeq Precision = 90.385								

Table 8 Final ANOVA results and statistical parameters for compression stress



The distribution of data points around the response variable's average may also be used to choose an acceptable model. A data point with a uniform distribution around the response variable's mean shows that the model is appropriate (Fig. 5). Using a predicted versus real plot, the relationship between the expected values of the response based on the model equation and the actual values acquired in the experiment were examined. With an R<sup>2</sup> value of 0.9843 and 0.9984 for bending and compression stresses, respectively, it is evident that the linear regression fit is appropriate and that the model accurately fits the experimental data. Moreover, the observed results and the corresponding projected results are contrasted in Table 9. Fig. 6 shows

the residuals against predicted values graphs for the final ANOVA for the two responses within the examination. The residuals for the two responses do not exhibit a significant degree of dispersion, as can be seen in this figure. Consequently, it appears that the suggested model was appropriate.

# 3.2 D response surfaces

Figs. 7 and 8 display the 3D response surfaces in the case of bending stress and compression stresses responses respectively. According to the quadratic models given in Eqs. (4) and (5) and Tables 5 and 6, only one interaction combination between the three manufacturing parameters is



	Table 9 Ten best solutions for bio-composite manufacturing parameters influencing bending and compression stresses											
Number	Fiber length (mm)	Volumetric fiber fraction (%)	NaOH (%)	Bending stress (MPa)	Compression stress (MPa)	Desirability						
1	5	0.5006	2.0001	8.2749	41.4372	1.00						
2	5.0004	0.5	2.00203	8.27438	41.1361	1.00						
3	5	0.500386	2.0007	8.27136	41.4179	1.00						
4	5.00453	0.500001	2.00004	8.27458	41.4361	1.00						
5	5.00002	0.500007	2.00364	8.27382	41.4347	1.00						
6	5.00824	0.500004	2	8.27424	41.4349	1.00						
7	5.00024	0.500817	2	8.26734	41.3961	1.00						
8	5.00016	0.500001	2.00754	8.27268	41.4324	1.00						
9	5.00081	0.501342	2.00004	8.26237	41.3693	1.00						
10	5.00005	0.500003	2.01452	8.27055	41.4278	1.00						



Fig. 6 The plot of the residual and predicted values for (a) bending, (b) compression



Fig. 7 3D response surface plot for the binary interaction (B-C) effect of the bio-composite manufacturing parameters on the bending response



Fig. 8 3D response surface plot for the binary interactions (a) (A-C) and(b) (B-C) effects of the bio-composite manufacturing parameters on the compressive response

significant for the bending stress (B-C) and two interactions combinations (A-C) and (B-C) for the compressive stress. Considering a fixed value for parameter A = 10 mm (fiber length), the maximum value for the bending test response was found to be around 8.2 MPa and to be depending on the combination of processing conditions. This value was achieved for a bio-composite that had been treated with 2% NaOH and reinforced with 0.5% volumetric fibers (Fig. 7). The maximum value of the response in the compression test for interaction (A-C) depends on the combination of the treatment conditions and fiber length and is approximately 40.3 MPa for a fixed value for parameter B = 0.5%V of fiber. This value was reached for a bio-composite that has 10 mm of fiber length and 2% NaOH treatment (Fig. 8(a)). Also, this same maximum value (40.3 MPa) of the response in the compression test and for interaction (B-C) which depends on the combination of the treatment conditions and volumetric fiber % and is approximately 40.3 MPa for a fixed value for parameter A (fiber length A = 10 mm). This value was reached for a bio-composite that has endured 2% NaOH treatment and 0.5% volumetric fiber reinforcement (Fig. 8(b)).

# 3.3 Optimization

To pick the processing parameters that would lead to the optimized flexural and compressive properties of the biocomposite, the surface response methodology (SRM) was carried out. Three factors were used to perform the optimization: fiber length (A), volumetric fiber percentage (B), and NaOH % (C). Based on the experimental findings reported in Table 9, and confirmed in Fig. 9, the optimal values of the responses, with target values of the objective function close to 1 (or 100%) for both tests (i.e., bending and compression), are selected as the most significant parameter values concerning the response factor. As can be seen in Table 9, the SRM process tends to find, among a multitude of solutions, the 10 best cases. In this context, the most efficient response values recorded for bending and compression stresses are respectively equal to 8.2749 MPa and 41.4372 MPa. They were recorded for the bio-composite with 0.5% volumetric fiber, a fiber length of 5 mm, and treated with 2% NaOH as depicted in Fig. 9. Compared to the control specimen, a 16.55% increase in bending resistance was registered as well as an 11.10% increase in compressive resistance. The histograms in Fig. 10 show the variation in: a) bending and b) compression responses respectively, depending on the involved factors (fiber length (A), volumetric fiber fraction (B), and percentage of



Fig. 9 Histograms of the variation of the responses a) bending and b) compression as a function of the factors involved (fiber length (a), fiber volume fraction, (b), and percentage of NaOH, (c), provided by the predictive statistical models



Fig. 10 The optimum conditions for the bio-composite manufacturing parameters given the results of the bending and compression tests

NaOH (C)), provided by the predictive statistical models. As can be seen, the combination that leads to the optimal values of the two responses, respectively 8.2755 Mpa for bending and 41.9041 Mpa for compression is: 0.5% volumetric fiber, 5 mm fiber length and 2 % NaOH.

# **4** Conclusions

In this study, a cement matrix and fibers extracted from the *Agave americana* were used to produce bio-composites, and an experimental design was built using Box-Behnken Design (BBD) of Response Surface Methodology (RSM). The effect of three main parameters (fiber length (A), volumetric fiber percentage (B), and NaOH% (C), upon bending and compressive properties were explored. This investigation leads to the following findings:

- The mean values of the bending and compressive stresses of the reference mortar are around 7.1 MPa and 37.3 MPa, respectively.
- In both cases of bending and compression tests, analysis of variance ANOVA demonstrated strong coefficients of determination, which led to a successful fitting of the regression models to the experimental data.
- The optimized contributions of variables on the flexural and compressive properties were determined using contour plots.
- The most efficient response values recorded for bending and compression stresses are respectively equal to 8.2749 MPa and 41.4372 MPa, which correspond to an increase of 16.54% and 11.09% compared to the reference mortar. They were recorded for the bio-composite with 0.5% V fiber, a fiber length of 5 mm, and treated with 2 % NaOH.

- The results show that the major improvements in flexural and compressive strength are obtained in the low levels of fiber length, volumetric fiber percentage, and NaOH% (C).
- The ANOVA reveals that the proposed quadratic models permit, among other possibilities, to determine the best selection of manufacturing parameters for mortars reinforced with Agave americana fibers, leading to the optimization of their mechanical characteristics under bending and compression.
- These models also show that the manufacturing parameters, length of fiber (A), % of volumetric fibers (B), and % of NaOH are significant for the bending and compression stresses respectively.

#### References

 Labib, W. A. "Plant-based fibres in cement composites: A conceptual framework", Journal of Engineered Fibers and Fabrics, 17, pp. 1–12, 2022.

https://doi.org/10.1177/15589250221078922

- [2] Khan, M., Cao, M., Ai, H., Hussain, A. "Basalt Fibers in Modified Whisker Reinforced Cementitious Composites", Periodica Polytechnica Civil Engineering, 66(2), pp. 344–354, 2022. https://doi.org/10.3311/PPci.18965
- [3] Wescott, C. G. "Building information-management systems for developing countries", In: Policy Issues in Microcomputer Applications for Developing Countries, Taylor & Francis, 2019, pp. 211–216. ISBN: 9780429301964 https://doi.org/10.4324/9780429301964
- [4] Callaghan, B. G. "The use of 3CR12 as reinforcing in concrete", Construction and Building Materials, 7(3), pp. 131–136, 1993. https://doi.org/10.1016/0950-0618(93)90049-I
- [5] Wiśniewski, D. F., Cruz, P. J. S., Henriques, A. A. R., Simões, R. A. D. "Probabilistic models for mechanical properties of concrete, reinforcing steel and pre-stressing steel", Structure and Infrastructure Engineering, 8(2), pp. 111–123, 2012. https://doi.org/10.1080/15732470903363164
- [6] Dayyani, M., Mortezaei, A., Rouhanimanesh, M. S., Marnani, J. A. "Experimental Study on the Effect of Fibers on Engineered Cementitious Composite Short Square Columns", Periodica Polytechnica Civil Engineering, 66(3), pp. 798–808, 2022. https://doi.org/10.3311/PPci.19612
- [7] Lin, H.-J., Liao, C.-I. "Compressive strength of reinforced concrete column confined by composite material", Composite Structures, 65(2), pp. 239–250, 2004. https://doi.org/10.1016/j.compstruct.2003.11.001
- [8] Ombres, L. "Flexural analysis of reinforced concrete beams strengthened with a cement based high strength composite material", Composite Structures, 94(1), pp. 143–155, 2011. https://doi.org/10.1016/j.compstruct.2011.07.008
- [9] Morton, J. H., Cooke, T., Akers, S. A. S. "Performance of slash pine fibers in fiber cement products", Construction and Building Materials, 24(2), pp. 165–170, 2010. https://doi.org/10.1016/j.conbuildmat.2007.08.015

• The RSM analysis procures a comprehensive view of the feasibility and effectiveness of the bio-fiber reinforced mortar design and optimization approach using three influencing parameters

## Declaration

The authors declare that there is no conflict of interest.

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[10] Zollo, R. F. "Fiber-reinforced concrete: an overview after 30 years of development", Cement and Concrete Composites, 19(2), pp. 107–122, 1997.

https://doi.org/10.1016/S0958-9465(96)00046-7

- [11] Dehghani, A., Ardekani, S. M., Al-Maadeed, M. A., Hassan, A., Wahit, M. U. "Mechanical and thermal properties of date palm leaf fiber reinforced recycled poly (ethylene terephthalate) composites", Materials & Design (1980-2015), 52, pp. 841–848, 2013. https://doi.org/10.1016/j.matdes.2013.06.022
- [12] Koronis, G., Silva, A. "Green Composites Reinforced with Plant-Based Fabrics: Cost and Eco-Impact Assessment", Journal of Composites Science, 2(1), 8, 2018. https://doi.org/10.3390/jcs2010008
- [13] Alawar, A., Hamed, A. M., Al-Kaabi, K. "Characterization of treated date palm tree fiber as composite reinforcement", Composites Part B: Engineering, 40(7), pp. 601–606, 2009. https://doi.org/10.1016/j.compositesb.2009.04.018
- [14] Ochi, S. "Mechanical properties of kenaf fibers and kenaf/PLA composites", Mechanics of Materials, 40(4–5), pp. 446–452, 2008. https://doi.org/10.1016/j.mechmat.2007.10.006
- [15] Mahjoub, R., Yatim, J. M., Sam, A. R. M., Hashemi, S. H. "Tensile properties of kenaf fiber due to various conditions of chemical fiber surface modifications", Construction and Building Materials, 55, pp. 103–113, 2014.

https://doi.org/10.1016/j.conbuildmat.2014.01.036

- [16] Lemita, N., Deghboudj, S., Rokbi, M., Rekbi, F. M. L., Halimi, R. "Characterization and analysis of novel natural cellulosic fiber extracted from Strelitzia reginae plant", Journal of Composite Materials, 56(1), pp. 99–114, 2022. https://doi.org/10.1177/00219983211049285
- [17] Holbery, J., Houston, D. "Natural-fiber-reinforced polymer composites in automotive applications", JOM, 58, pp. 80–86, 2006. https://doi.org/10.1007/s11837-006-0234-2
- [18] Felekoğlu, B. "Effects of loading conditions and specimen thickness on the flexural behavior of fiber-reinforced cementitious composites", Periodica Polytechnica Civil Engineering, 58(3), pp. 279–291, 2014.

https://doi.org/10.3311/PPci.7486

[19] Khelifa, H., Bezazi, A., Boumediri, H., Garcia del Pino, G., Reis, P. N. B., Scarpa, F., Dufresne, A. "Mechanical characterization of mortar reinforced by date palm mesh fibers: Experimental and statistical analysis", Construction and Building Materials, 300, 124067, 2021.

https://doi.org/10.1016/j.conbuildmat.2021.124067

- [20] Saaidia, A., Bezazi, A., Belbah, A., Bouchelaghem, H., Scarpa, F., Amirouche, S. "Mechano-physical properties and statistical design of jute yarns", Measurement, 111, pp. 284–294, 2017. https://doi.org/10.1016/j.measurement.2017.07.054
- [21] Achara, B. E., Mohammed, B. S., Liew, M. S. "Bond behaviour of nano-silica-modified self-compacting engineered cementitious composite using response surface methodology", Construction and Building Materials, 224, pp. 796–814, 2019. https://doi.org/10.1016/j.conbuildmat.2019.07.115
- [22] Mazloom, M., Mirzamohammadi, S. "Computing the fracture energy of fiber reinforced cementitious composites using response surface methodology", Advances in Computational Design, 6(3), pp. 225–239, 2021.

https://doi.org/10.12989/acd.2021.6.3

[23] Mohammed, B. S., Achara, B. E., Liew, M. S., Alaloul, W. S., Khed, V. C. "Effects of elevated temperature on the tensile properties of NS-modified self-consolidating engineered cementitious composites and property optimization using response surface methodology (RSM)", Construction and Building Materials, 206, pp. 449–469, 2019.

https://doi.org/10.1016/j.conbuildmat.2019.02.033

- [24] Khed, V. C., Mohammed, B. S., Liew, M. S., Zawawi, N. A. W. A. "Development of response surface models for self-compacting hybrid fibre reinforced rubberized cementitious composite", Construction and Building Materials, 232, 117191, 2020. https://doi.org/10.1016/j.conbuildmat.2019.117191
- [25] Madhu, P., Sanjay, M. R., Jawaid, M., Siengchin, S., Khan, A., Pruncu, C. I. "A new study on effect of various chemical treatments on Agave Americana fiber for composite reinforcement: Physicochemical, thermal, mechanical and morphological properties", Polymer Testing, 85, 106437, 2020. https://doi.org/10.1016/j.polymertesting.2020.106437
- [26] Mylsamy, K., Rajendran, I. "Investigation on physio-chemical and mechanical properties of raw and alkali-treated Agave americana fiber", Journal of Reinforced Plastics and Composites, 29(19), pp. 2925–2935, 2010. https://dxi.org/10.1177/0721/0844102/2817

https://doi.org/10.1177/0731684410362817

- [27] Bezazi, A., Belaadi, A., Bourchak, M., Scarpa, F., Boba, K. " Novel extraction techniques, chemical and mechanical characterisation of Agave americana L. Natural fibres", Composites Part B: Engineering, 66, pp. 194–203, 2014. https://doi.org/10.1016/j.compositesb.2014.05.014
- [28] Said, K. A. M., Amin, M. A. M. "Overview on the response surface methodology (RSM) in extraction processes", Journal of Applied Science & Process Engineering, 2(1), pp. 8–17, 2015. [online] Available at: https://publisher.unimas.my/ojs-training/index.php/ JASPE/article/ view/161
- [29] Khuri, A. I., Mukhopadhyay, S. "Response surface methodology", Wiley Interdisciplinary Reviews: Computational Statistics, 2(2), pp. 128–149, 2010.

https://doi.org/10.1002/wics.73

- [30] Myers, R. H., Khuri, A. I., Carter, W. H. "Response surface methodology: 1966-1988", Technometrics, 31(2), pp. 137–157, 1989. https://doi.org/10.1080/00401706.1989.10488509
- [31] Hassan, M. Z., Sapuan, S. M., Roslan, S. A., Abdul Aziz, S., Sarip, S. "Optimization of tensile behavior of banana pseudo-stem (Musa acuminate) fiber reinforced epoxy composites using response surface methodology", Journal of Materials Research and Technology, 8(4), pp. 3517–3528, 2019. https://doi.org/10.1016/j.jmrt.2019.06.026
- [32] Ruangmee, A., Sangwichien, C. "Statistical optimization for alkali pretreatment conditions of narrow-leaf cattail by response surface methodology", [pdf] Songklanakarin Journal of Science & Technology, 35(4), pp. 443–450, 2013. Available at: https://www. thaiscience.info/journals/Article/SONG/10890989.pdf
- [33] Kandar, M. I. M., Akil, H. M. "Application of Design of Experiment (DoE) for Parameters Optimization in Compression Moulding for Flax Reinforced Biocomposites", Procedia Chemistry, 19, pp. 433– 440, 2016.

https://doi.org/10.1016/j.proche.2016.03.035

- [34] Maruyama, S. A., Palombini, S. V., Claus, T., Carbonera, F., Montanher, P. F., de Souza, N. E., Visentainer, J. V., Gomes, S. T. M., Matsushita, M. "Application of box-behnken design to the study of fatty acids and antioxidant activity from enriched white bread", Journal of the Brazilian Chemical Society, 24, pp. 1520–1529, 2013. https://doi.org/10.5935/0103-5053.20130193
- [35] Mongomery, D. C. "Design and Analysis of Experiments", Wiley, 2017. ISBN978-1-119-49244-3
- [36] CEN "BS EN 196-1 Methods of testing cement Part 1: Determination of strength", European Committee for Standardization, Brussels, Belgium, 2016.