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Application of Response Surface Methodology for Optimizing Tensile Strength of Rice Husk Fiber-Reinforced Polylactic Acid Composites †

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Abstract: Filler/fiber loading and surface modification significantly influence tensile properties of natural fiber-reinforced plastic composites. It is therefore pertinent that they are suitably selected in order to yield the optimum tensile properties. Fiber-reinforced PLA composites were prepared using compression molding with Box-Behnken Design experimental design approach. Factors, namely clay filler loading (1−5 wt.%), rice husk fiber loading (10−30 wt.%), alkali concentration (0−4 wt.%), rice husk variety (K85, K98) and alkali type (NaOH, $Mg(OH)_2$) were varied. ANOVA determined significance of the factors affecting composites' tensile strength. ANOVA results revealed the reduced cubic model as best fit for tensile strength response. The desirability function revealed that variable values leading to optimum tensile strength (33.67 MPa) were 4.97 wt.%, 11.16 wt.% and 3.99 wt.% for filler loading, fiber loading and alkali concentration, respectively.

Keywords: clay; optimization; polylactic acid; PLA composites; rice husks; RSM; tensile strength

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1. Introduction

In recent times, there are growing interests in the use of agricultural fibers as reinforcement in plastics so as to minimize the environmental challenges associated with disposal of non-biodegradable polymer [1,2]. A traditional approach to experimental work for developing plastic composites is to vary one factor at a time, holding all other factors fixed but this is time consuming and unable to produce satisfactory results in a wide range of experimental settings [3]. As such, the complexity of developing fiber-reinforced PLA composites with the required tensile properties can be solved by employing Response Surface Methodology (RSM) [4]. RSM is one of Design of Experiments (DOE) techniques that implements a statistical approach to optimize factors necessary to enhance tensile properties of fiber-reinforced PLA composites. This is done by applying multiple regression analysis to the responses to form a model for the desired response term [5,6]. Besides, Analysis of Variance (ANOVA), which is embedded in RSM provides statistical results and diagnostic checking tests to the developed model. These are extremely important in evaluating the model's adequacy.

RSM approach requires fewer experimental runs compared to single factor experiments, making it less costly and less time consuming while enhancing accuracy in experimentation [7,8]. Box-Behnken Design (BBD) is more efficient than Central Composite Design (CCD), Doehlert Design (DD) as well as 3-level factorial Design [5]. The higher efficiency is because Box-Behnken Designs are rotatable and do not contain combinations for which all factors are simultaneously at their highest or lowest levels. As such, these designs avoid experiments performed under extreme conditions which would otherwise yield unsatisfactory results [9]. BBD is appreciated for its fast and economic convergence compared to other traditional approaches as well as for its ability to provide 3 to 7 factors, along with three levels of experimental conditions (low, medium and high levels) [10–12].

Some researchers have previously used BBD to optimize tensile strength of fiber-reinforced composites. Hassan et al. [13] used BDD to design an experiment to investigate the mercerization effect of bamboo fiber-reinforced epoxy composites. It was found that the treatment factors (NaOH concentration, soaking time and drying time) had a significant impact on the tensile properties of the developed composites. The optimum mercerization condition for bamboo fiber was reported as a NaOH concentration of 5.81 wt.%, a soaking time of 3.99 h and a drying time of 72 h which yielded a tensile strength of 338.95 MPa. Tharazi et al. [14] used BBD to identify the cause-and-effect relationship between processing parameters and composites' tensile strength of kenaf fiber-reinforced PLA composites. Hassan et al. [6] studied the optimization of fiber length, NaOH concentration and fiber loading in developing banana pseudo-stem fiber-reinforced epoxy composites. They reported that BBD accurately optimized independent conditions to obtain optimal composites' tensile strength and flexural modulus. Soundhar et al. [15] used BBD to confirm that addition of natural fibers to polyurethane foam matrix enhances the matrix's tensile properties due to enhanced adhesion between the fibers and matrix. The effects of hot-pressing parameters on tensile strength of aligned kenaf fiber-reinforced PLA composites were investigated using BBD by Tharazi et al. [3]. They suggested that the best combination of optimum hot press process parameters wherefor maximum tensile strength were 200 °C (temperature), 3 MPa (pressure) and 8 min (heating time).

Experiment design is an effective tool for optimizing the developed PLA composites' tensile strength since it depends on a number of factors. In this study therefore, parameter optimization using Design Expert Software (version 13, Stat-Ease Inc., MN, USA) was employed to characterize the tensile strength of the developed PLA/clay/rice husk composites. The experimental runs were based on the Box-Behnken design with five factors namely: alkali concentration, fiber loading, filler loading, rice husk variety and alkali type.

2. Materials and Methods

2.1. Materials

Rice husks (K98 and K85) of 13% moisture content were obtained from Tororo district (latitude 0.45°, longitude 34.05°, ≈208 km from Kampala city center). Polylactic acid in pellet form with a specific gravity 1.24 g/cm^3 and melt flow index (MFI) of 7 g/10 min (210 °C/2.16 kg) was purchased from Huaian Ruanke Trade Co. Ltd., Huaian, China. Kaolin was obtained from Buwambo clay deposits (latitude 0.50°, longitude 32.55°, ≈24 km from Kampala city center) and used as received. Magnesium Hydroxide ($Mg(OH)_2$) (CAS number 2917-11-90) with a molecular weight of 58.53 g/mol and Sodium Hydroxide (NaOH) (CAS number 1310-73-2) with a molecular weight of 40 g/mol were both supplied by Lab Access Uganda Ltd., Kampala, Uganda.

2.2. Methods

2.2.1. Surface Modification

Part of the husks was used as received (raw rice husks samples). Alkaline surface modification was done for the other part using $Mg(OH)_2$ and NaOH (modified rice husks samples). This involved soaking 1000 g of rice husks in 2 wt.% and 4 wt.% concentrated solutions for 3.5 h at ambient temperature using a 20:1 liquor ratio. Samples were then washed in reverse osmosis water until a neutral PH was obtained. They were then dried for 48 h at room temperature and then 60 °C over-night before storage [16].

2.2.2. PLA Composites Fabrication

Prior to fabrication, PLA pellets, rice husks and clay were held in an oven at 100 °C for 1 h. Rice husks and clay were then ground to ≤100 μm using a using a Retsch Planetary Ball mill PM 100 machine, Hann, Germany. PLA was melted and mixed with ratios shown in Table 1 in a compression molding machine to obtain tensile strips [17]. The residence time for composites preparation at 195 °C during compression molding was 10 min. Compression was effected by use of a hand-screw jerk for 10 min under ≈7 MPa loading. The strips were then air-cooled for 10 min and stored before tensile testing.

2.2.3. Tensile Tests

Tensile testing for the developed PLA composites were performed according to ASTM D638 at 25 °C using a Universal Testing Machine (Testometric FS300CT-2032, England, UK) with a 0–300 kN load cell range at a constant crosshead speed of 10 mm/min. Tests were conducted for three samples in each run and their average was used.

2.2.4. Design of Experiments

The response (tensile strength) was optimized using a standard RSM design in Design of Experiments (BBD) which consists of a central point and middle points of the edges [6]. Clay filler loading, rice husks fiber loading, alkali concentration, rice husk variety and alkali type were employed as the five input factors in this study. Table 1 shows the low, medium and high levels of each. The numeric factors (k_1); filler loading, fiber loading and alkali concentration were elucidated in a range of 1–5 wt.% [18], 10–30 wt.% ([6,19] and 0–4 wt.% [20], respectively, as tabulated in Table 1.

Table 1. Actual and coded factors for experimental design.

Factor	Code	Variation Levels		
		Low (−1)	Medium (0)	High (+1)
Filler loading (wt.%)	A	1	3	5
Fiber loading (wt.%)	B	10	20	30
Alkali concentration (wt.%)	C	0	2	4
Rice husk variety	D	K85	-	K98
Alkali type	E	NaOH	-	$Mg(OH)_2$

Randomized order runs were planned factors using Design Expert Software (version 13, Stat-Ease Inc., MN, USA) to minimize the effects of the uncontrolled. The runs consisted three numeric level factors (k_1), two categoric level factors (k_2) and five replicated central points (c_p). This produced 68 total required experiments based on Equation (1).

$$N = \left(2k_1(k_1 - 1) + c_p\right) \times (k_2)^2 = \left((2 \times 3)(3 - 1) + 5\right) \times (2)^2 = 68 \quad (1)$$

The influence of the factors on PLA composites' tensile strengths can be modelled using a second-order polynomial shown in Equation (2).

$$\text{Tensile strength (MPa)} = \beta_0 + \sum_{j=1}^{k} \beta_j x_j + \sum_{i<j} \sum \beta_{ij} x_i x_j + \sum_{i=0}^{k} \beta_{jj} {x_j}^2 + e \quad (2)$$

where β_0 is the model constant, β_j is the linear coefficient, β_{ij} is the interaction coefficient, β_{jj} is the quadratic coefficients, x is the independent factors in coded values, k is the number of studied and optimized factors, and e is the experimental error.

3. Results and Discussions

The experimental design and results obtained at different combinations of factors are shown in Table 2. The main effect and interaction of the experimental factors on the tensile strength response, analysis of variance (ANOVA) and regression analysis were discussed. Optimal combination of process factors was found based on the contour analysis.

Table 2. Experimental Design and response results.

Experimental Factors				NaOH Modified K85	$Mg(OH)_2$ Modified K85	NaOH Modified K98	$Mg(OH)_2$ Modified K98
Runs	A: Filler Loading (wt.%)	B: Fiber Loading (wt.%)	C: Alkali Concentration (wt.%)	Response-Tensile Strength (MPa)			
1	1	10	2	22.64	20.05	21.15	20.66
2	5	20	4	32.08	33.63	32.44	32.44
3	3	20	2	24.81	24.42	24.61	25.29
4	1	30	2	25.58	25.82	26.51	25.43
5	3	20	2	24.67	24.5	25.72	25.65
6	5	10	2	25.51	25.85	25.79	26.41
7	5	20	0	22.91	22.91	22.37	21.23
8	1	20	0	30.22	30.22	29.19	29.52
9	3	20	2	24.23	24.05	25.35	26.35
10	3	10	4	25.59	25.29	25.09	25.1
11	3	20	2	25.53	25.75	25.87	24.44
12	5	30	2	26.43	25.82	27.61	25.94
13	3	20	2	24.31	25.04	25.52	25.55
14	1	20	4	19.54	19.64	21.15	20.25
15	3	30	4	22.8	23.23	23.56	23.3
16	3	30	0	29.29	28.32	27.38	27.09
17	3	10	0	23.27	23.33	21.17	21.35

3.1. Analysis of Variance (ANOVA)

Table 3 shows ANOVA results for the tensile strength of the developed PLA composites based on the experimental results. The table presents results on statistical significance of the factors as well as respective model coefficients. Additionally, statistical data for tensile strength variance analysis is presented.

Table 3. Analysis of Variance results for reduced cubic model.

Source	Sum of Squares	*df*	Mean Square	*F* Value	*p* Value	
Model	605.85	12	50.49	130.9	<0.0001	significant
A-Filler loading	54.6	1	54.6	141.57	<0.0001	
B-Fiber loading	40.25	1	40.25	104.37	<0.0001	
D-Rice husk variety	2.17	1	2.17	5.63	0.0212	
AB	17.22	1	17.22	44.65	<0.0001	
AC	397.4	1	397.4	1030.37	<0.0001	
BC	60.72	1	60.72	157.44	<0.0001	
CD	5.02	1	5.02	13.01	0.0007	
A^2	6.9	1	6.9	17.89	<0.0001	
B^2	13.58	1	13.58	35.22	<0.0001	
C^2	4.39	1	4.39	11.39	0.0014	
ABE	1.88	1	1.88	4.87	0.0316	
C^2D	5.05	1	5.05	13.09	0.0006	
Residual	21.21	55	0.3857			
Lack of Fit	15.52	39	0.3981	1.12	0.4186	not significant
Pure Error	5.69	16	0.3555			
Cor Total	627.06	67				

R^2 = 0.9662	Adjusted R^2 = 0.9588
Predicted R^2 = 0.9464	Adequate precision = 47.03
C.V. % = 2.46	Mean = 25.2

The model *F* value of 130.90 corresponding to the reduced cubic model implies the model is significant (p value < 0.05). There is only a 0.01% chance that an *F* value this large could occur due to noise. The lack of fit of the developed model has an *F* value of 1.12, which shows that there is a 41.86% chance that lack of fit values occurred due to noise. The model was statistically inspected using the lack of fit p value (0.4186). For interaction between factors, p values < 0.05 indicate model terms are significant and p values > 0.10 must be removed to improve model accuracy. In this case, significant model terms were A, B, D, AB, AC, BC, CD, A^2, B^2, C^2, ABE and C^2D. Among these, the interactive effect between filler loading and alkali concentration (*F* value of 1030.37) was found to be of most influence, followed by the interactive effect between fiber loading and alkali concentration (*F* value of 157.44). The least influence was the interactive effect between filler loading, fiber loading and alkali type (*F* value of 4.87).

The coefficient of regression value (R^2) of 0.9662 suggested that the model competently represented the relationship between significant model terms and the tensile strength of the developed PLA composites. Predicted R^2 value (0.9464) was in reasonable agreement with adjusted R^2 value (0.9588) with a difference of less than 0.2 [4]. The low C.V. values (2.46%) show preciseness of the estimate since it is much less than 10% [21]. Moreover, the adequate precision ratio of 47.03 indicated adequate signal since it was much greater than 4, suggesting that the model can be used to navigate the design space defined by the BBD [22].

Diagnostics plots of (a) normal probability distribution vs. studentized residuals, (b) studentized residuals and predicted as well as (c) studentized residuals residual and experimental runs for tensile strength are shown in Figure 3. The randomness observed in Figure 3b,c proved that the residuals follow normal distribution and fit the data well [3].

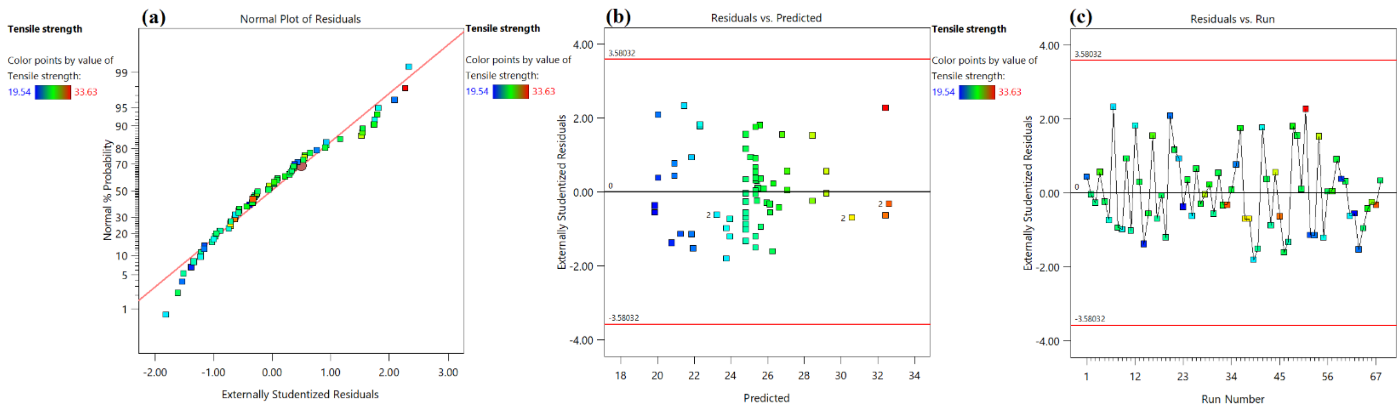

Figure 1. (**a**) Normal probability-residual; (**b**) externally studentized residuals-predicted response and (**c**) externally studentized residuals-run following tensile test.

3.2. Regression Analysis

Multiple linear regression analysis was used to generate a mathematical model for variation of tensile strength with significant process factors. The tensile strength response surface reduced cubic model for the developed PLA composites is shown in Equation (3). It was obtained after neglecting insignificant model terms (p values > 0.1).

$$\begin{aligned} Tensile\ strength\ (MPa) &= 25.08 + 1.31A + 1.12B + 0.25D - 1.04AB + 4.98AC - 1.95BC \\ &+ 0.40CD + 0.64A^2 - 0.90B^2 + 0.51C^2 - 0.34ABE - 0.55C^2D \end{aligned} \quad (3)$$

where A: filler loading (wt.%), B: fiber loading (wt.%) and C: alkali concentration (wt.%).

3.3. Contour Plots

Figure 2 presents relationships between tensile strength and the three numeric experimental factors (fiber loading, filler loading and alkali concentration). Each contour plot shows the effects of two numeric factors within the experimental range, while keeping one numeric factor and the two categoric factors constant at center points. Figure 2a shows the interaction effect between rice husks fiber loading and clay filler loading on tensile strength. It can be observed that increase in both filler and fiber loading led to increasing tensile strengths. This is attributed to better interlocking between the fiber/filler and PLA matrix [23].

The interaction effect between alkali concentration and clay filler loading process factors on tensile strength is shown in Figure 2b. At 1 wt.% filler levels, tensile strength reduced due to alkali modification of rice husks while at the 5 wt.% filler loading conditions, tensile strength of the developed PLA composites increased with rice husks alkali modification. It is clear that when low filler amounts are used, it is not necessary to modify rice husks while when higher filler amounts are used, higher alkali concentrations are required to modify rice husks so as to enhance their compatibility with PLA matrix.

Figure 2c shows the interaction effect between alkali concentration and fiber loading. When raw rice husks were used, tensile strength increased with increasing fiber loading from while with 4 wt.% alkali concentrations, no striking effect was noted. It should also be noted that at low fiber loading, alkali modification led to increased tensile strengths, while an opposite trend was observed at high fiber loading.

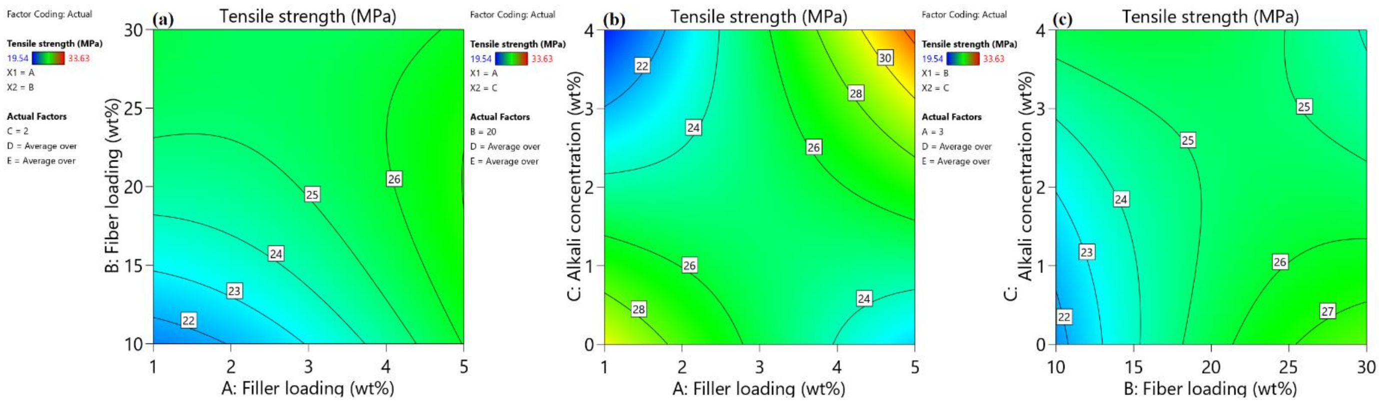

Figure 2. Response contour plots for PLA composites' Tensile strengths.

3.4. Optimization Responses

The optimization of process parameters in range, for maximizing tensile strength of the developed PLA composites was done by Design-Expert software using the regression equation (see Equation (3)). As such, for a maximum tensile strength (33.70 MPa), the suggested optimum combination variables were (4.97 wt.%) filler loading, (11.16 wt.%) fiber loading and (3.99 wt.%) alkali concentration with a desirability value of 1.

4. Conclusions

The effect of clay filler loading, rice husks fiber loading and alkali concentration on tensile strengths of PLA composites was evaluated using RSM. As shown in the experimental results, clay filler loading had the most significant individual effect on tensile strength of the developed PLA composites. Tensile strength was also highly influenced by interaction effects between filler loading and alkali concentration. The quadratic effect of alkali concentration had the least influence on tensile strength. Based on the R^2 value (0.9662), the results showed good agreement between experimental and predicted tensile strength values. The combination of optimum factors where tensile strength is maximum (33.70 MPa) were obtained as (4.97 wt.%) filler loading, (11.16 wt.%) fiber loading and

(3.99 wt.%) alkali concentration. The optimization results indicated that a BBD is a very effective tool for optimizing individual factors in a new process.

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