Optimization of Chemical Treatment Conditions of Ampelocissus Cavicaulis Fibre Using RSM

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Abstract- Ampelocissus Cavicaulis fibre was treated with NaoH, acetic anhydride, nitric acid, and zinc chloride. The influence of these chemicals, their concentrations, and pretreatment times on the resultant tensile strength of the fibre was studied. Response surface methodology was used to optimize the numeric and categoric factors involved. The results showed that the single and interaction effects of the chemical types and their concentrations were significant. Two factors interaction (2FI) model was proposed for predicting the ultimate tensile strength of the fibre. The optimum conditions obtained for the different chemical used are; 6% NaoH for 50minues, 14% Acetic anhydride for 70minutes, 6% Nitric acid for 50minutes, and 3% Zinc chloride for 70minutes. These optimum conditions were validated with little errors of less than 2.0%

Keywords- Ampelocissus cavicaulis fibre, Acetic anhydride, Nitric acid, RSM, Sodium hydroxide, Zinc chloride

1 INTRODUCTION

Natural fiber reinforced polymer composites have raised great interest among material scientists and engineers in recent years due to the need for developing an environmentally friendly material, and partly replacing currently used synthetic fiber for composite reinforcement [1], [2]. Composite material is made of the combination of two different materials to achieve certain properties different from each material on its own. One of the two materials acts as a matrix, while the other acts as reinforcing material. The reinforcing material is imbedded in the matrix material to improve its mechanical and physical properties [3]. Natural fibers have advantages over synthetic fiber due to these reasons; low cost, low densities, acceptable specific strength properties, ease of separation, carbon dioxide sequestration and biodegradability [4]. However, these natural fibers are not problem free as reinforcement on composites. In natural fiber reinforced composites, there is a lack of good interfacial adhesion between the hydrophilic fibers and hydrophobic resins due to their inherent incompatibility [5]. Chemical treatment on the natural fibers directly influences the fiber structure and changes their composites thereby facilitating better bonding with the matrix materials. Many researchers have worked on chemical treatments of natural fiber on their mechanical properties, but have not optimized the conditions of the treatments. This study was aimed at optimizing the conditions for the chemical treatments of Ampelocissus cavicaulis fiber on its mechanical property.

Central composite design (CCD), a type of response surface methodology (RSM) was used to optimize the process conditions. The novelty of this research lies on the fact that the CCD incorporated not only the numerical factors, but also the categorical factor. The categorical factor is the types of chemical used for the pretreatment. The work developed a good model by diagnosing and validating the model.

2 EXPERIMENTAL DESCRIPTIONS

2.1 Raw Materials Preparation

Ampellocissus cavicaulis fibre was obtained from well defined locations in Ebonyi State of Nigeria. This plant fibre was extracted from the plant stem using water retting extraction process, giving fibre of different lengths and diameters. Before usage, the fibre was visually selected in order to verify the absence of defects along the length of the fibre.

2.2 Alkali treatment

The Ampelocissus cavicaulis was treated at 6% NaoH in accordance with work done by nural and Ishak with slight modifications [9]. The fiber was immersed in the alkali solution for 50 minutes, then neutralized with acetic acid and washed with distilled water repeatedly until all sodium hydroxide was eliminated. Finally, the fiber was washed with distilled water and dried at room temperature for 48h

2.3 Acetic anhydride treatment

The acetylation process was in accordance with work done by A.K bledzki, with slight modifications [10]. The Ampelocissus cavicaulis fiber was soaked in distilled water for an hour, filtered and placed in a round bottom flask containing 10% acetic acid solution for 30 minutes. After which it was placed on flask containing 14% acetic anhydride solution. The process temperature of acetylation was 30^0C and duration was 70 minutes. After modification, the fiber was washed periodically with distilled water until acid free. Finally, modified fiber was air dried for certain time before analysis.

2.4 Nitric acid treatment

The nitric acid treatment was according to F. Vautard etal 2013 [11] and W. 2 etal 2010 [12] with modifications. The size reduced ampleocissus cavicaulis fiber was oxidized with 6% nitric acid. The prepared oxidizing solution was boiled to a temperature of 60^0C and the fiber immersed in the solution at maintained said temperature for 50 minutes. It was then neutralized with NaoH solution and washed with distilled water repeatedly until all the nitric acid was eliminated. Finally, the fiber was washed again with distilled water and dried to a constant weight temperature.

2.5 Zinc chloride treatment

Zinc chloride treatment was done in accordance with the work done by V. Nadanthangam etal 2013 with modification [13]. The fiber was soaked in 3% zinc chloride solution for 70 minutes after which it was washed with distilled water until the washing solution became chloride free. The fiber was washed with distilled water and dried at room temperature for 48 hours.

2.6 Tensile Strength

Tensile strength is a measurement of the ability of material to withstand forces that tend to pull it apart. It determines to what extents the material stretches before breaking. The fiber tensile strength tests were performed by a computer controlled Hounsfield tensometer testing machine (model 5566) with a gauge length of 40mm and a crosshead speed of 5mm/min. The round bars were covered with surgical glove fingers, and the flax was clamped at the top and bottom. The fiber bundle was wrapped one revolution around each of the two bars and was spread out over the entire gauge length in a parallel. The machine was wet up to display a force deformation curve at loading and to read the load at maximum or the break point.

2.7 Determination of lignin content by gravimetric method.

This was done according to work done by G.N Onyeagoro 2012 [13]. 2.0g of the sample were weighed and placed inside a beaker. 72% H_2SO_4 was added and allowed to stand for 2 hours. 8% H_2SO_4 was later added and the solution refluxed for 3 hours. The residue was filtered with purpling cloth and washed severally with hot water. A crucible was weighed and the sample was scraped into it. The sample was oven dried at 110^0C for 1 hour and then cooled inside desiccators after which the weight was taken. The sample was ashed in a furnace at 500^0C for 3 hours. It was then cooled inside desiccators and finally weighed. The % lignin was calculated using equation

$$\% \text{ Lignin} = \frac{W_2 - W_1}{W_S} X\ 100 \qquad (1)$$

Where,

W_1 = weight of ash sample + crucible

W_2 = weight of oven dried sample + crucible

W_S = initial weight of dried sample

2.8 Determination of cellulose content

This was done according to work done by G.N Onyeagoro 2012 [13]. 1.5g of fiber sample was weighed into a beaker followed by addition of 20ml of 80% acetic acid, 1ml of concentrated nitric acid and 3 glass beads. The content was refluxed for 30 minutes. The sample was cooled and washed into 50ml centrifuge tube containing hot 95% ethanol, and then centrifuged at 15,000rpm for 5 minutes. Thereafter, the liquid was decanted and 95% ethanol was added, stirred and filtered by suction. The sample was washed three times with hot benzene, two times with 95% ethanol and once with ether. The sample was placed inside a weighed crucible and placed in the oven maintained at 1100C for 1 hour. The crucible was then cooled in desiccators and weighed. For ash content determination, the crucible and its content was placed inside a furnace maintained at 5000C for 3 hours after which it was cooled in desiccators and weighed. The % cellulose was calculated from equation 2.

$$\% \text{ cellulose} = \frac{w_2 - w_1 x100}{ws} \qquad (2)$$

Where,

W1 = weight of crucible + sample after ashing

W2 = weight of crucible + sample after drying

Ws = weight of sample

2.9 Holocellulose content

This was according to the work done by A.K Bledzki et al 2008 [10].

Three grams of air dried fibre was weighed and placed in an Erlenmeyer flask and then, 160 ml of distilled water, 0.5 ml of glacial acetic acid and 1.5 g of sodium chloride were added successively. The flask was placed in water bath and heated up to 75°C for an hour and then additional 0.5 ml of glacial acetic acid and 1.5 g of sodium chloride were added. The additions of acetic acid and sodium chloride were repeated two times hourly. The flask was placed in an ice bath and cooled down below 10°C. The holocellulose was filtered and washed with acetone, ethanol and water respectively and at the end, sample was dried in oven at 105°C before weighed.

2.10 α-cellulose content

This was according to the work done by A.K Bledzki et al 2008 [10]. Two grams of holocellulose were placed in a beaker and 10 ml of sodium hydroxide solution (17.5%) was added. The fibre was stirred up by glass rod so that they could be soaked with sodium hydroxide

solution vigorously. Then sodium hydroxide solution was added to the mixture periodically (once every five minutes) for half an hour and the mixture temperature was kept at 20°C. About 33 ml of distilled water was added in the beaker and kept it for an hour. The holocellulose residue was filtered and transferred to the crucible and washed with 100 ml of sodium hydroxide (8.3%), 200 ml of distilled water, 15 ml of acetic acid (10%) and again water successively. The crucible with α-celluloses was dried and weighed.

2.11 Hemicellulose content

The content of hemicelluloses of flax fibre was calculated from Equation below

Hemicelluloses = Holocellulose – α-celluloses [10]. (3)

2.12 Optimizing process using RSM

The optimization was done using central composite design (CCD) with full factorial core encompassing two numerical factors (chemical concentration and time) and one categorical factor (chemical type). With the categorical factor added, the experimental runs were multiplied by the number of the categoric factor making the experiment fifty-two runs.

These numeric factors had factorial, axial and center points, but categoric factor had only four levels but nothing in between as center point. The four levels of the categoric factor are NaoH, Acetic anhydride, Nitric Acid and zinc chloride. The location of the axial points from the factorial points was "two" making the design rotatable. The experiments were randomized to protect against an unknown bias distorting the result of the experiment. The design matrix used for the experiment with the experimental values for the fiber is shown in table 3. The experiment was strictly based on the design matrix in table 3.

3 RESULTS AND DISCUSSIONS

3.1 Properties of natural Fiber

To better understand the effect of chemical treatments on the fiber, the chemical and mechanical properties of the natural fibre was analysed prior to chemical treatment. Fibre properties directly influence the physical and mechanical properties of the fibre reinforced composites. Natural fibre consists of cellulose, hemicelluloses, lignin, wax, water content water soluble substances. These compositions differed with the different species of the plant. Physical and mechanical properties of the fibre, depends on these chemicals compositions, grooving conditions (soil features, climate, aging conditions) and extraction/processing method conditions (Bongarde and Shinde 2014).

The chemical and mechanical properties of the natural fibre used in thus study are shown in table 1 and 2 respectively.

TABLE 1

CHEMICAL PROPERTIES OF FIBRE

Cellulose Content (%)	Hemicellulose content (%)	Lignin content (%)	Ash content (%)	Wax (%)	Moisture content (%)
48.967	21.221	31.33	2.43	0.21	0.514

TABLE 2

MECHANICAL PROPERTIES OF FIBER

Density (g/cm3)	Tensile Strength (Mpa)	Elastic Modulus (Mpa)	Ultimate Elongation at break (%)
1.28	238.28	3,971.33	3.00

TABLE 3

RSM DESIGN MATRIX WITH THE EXPERIMENTAL VALUES

Std Order	Run Order	Chemical concentration (%)	Time (Mins.)	Chemical type	Tensile Strength (Mpa)
11	1	10.00	70.00	NaOH	330.176
27	2	6.00	50.00	Acetic Anhydride	318.4
8	3	10.00	110.00	NaOH	315.936
38	4	10.00	70.00	Acetic Anhydride	329.52
7	5	10.00	30.00	NaOH	328.088
32	6	18.00	70.00	Acetic Anhydride	340.8
5	7	2.00	70.00	NaOH	321.576
35	8	10.00	70.00	Acetic Anhydride	328.296
46	9	10.00	30.00	Zncl	319.912
4	10	14.00	90.00	NaOH	416
40	11	6.00	50.00	Zncl	319.016
9	12	10.00	70.00	NaOH	305.6
3	13	6.00	90.00	NaOH	320.208
37	14	10.00	70.00	Acetic Anhydride	328.312
28	15	14.00	50.00	Acetic Anhydride	328.632
48	16	10.00	70.00	Zncl	316.176
36	17	10.00	70.00	Acetic Anhydride	306.04
47	18	10.00	110.00	Zncl	202.168

52	19	10.00	70.00	Zncl	330.224
16	20	6.00	90.00	Nitric Acid	316.576
33	21	10.00	30.00	Acetic Anhydride	327.496
41	22	14.00	50.00	Zncl	240.416
34	23	10.00	110.00	Acetic Anhydride	329.6
17	24	14.00	90.00	Nitric Acid	205.848
6	25	18.00	70.00	NaOH	378.288
49	26	10.00	70.00	Zncl	336
29	27	6.00	90.00	Acetic Anhydride	320.632
45	28	18.00	70.00	Zncl	220.712
19	29	18.00	70.00	Nitric Acid	193.864
30	30	14.00	90.00	Acetic Anhydride	336.256
13	31	10.00	70.00	NaOH	331.464
24	32	10.00	70.00	Nitric Acid	240.176
1	33	6.00	50.00	NaOH	316.264
12	34	10.00	70.00	NaOH	320.784
39	35	10.00	70.00	Acetic Anhydride	318.976
10	36	10.00	70.00	NaOH	240
15	37	14.00	50.00	Nitric Acid	208.576
43	38	14.00	90.00	Zncl	208.096
2	39	14.00	50.00	NaOH	408
20	40	10.00	30.00	Nitric Acid	320
14	41	6.00	50.00	Nitric Acid	314.024
31	42	2.00	70.00	Acetic Anhydride	312.8
23	43	10.00	70.00	Nitric Acid	244.616
25	44	10.00	70.00	Nitric Acid	216.72
44	45	2.00	70.00	Zncl	314.304
42	46	6.00	90.00	Zncl	34`.048

18	47	2.00	70.00	Nitric Acid	312.576
50	48	10.00	70.00	Zncl	270.488
22	49	10.00	70.00	Nitric Acid	238.664
26	50	10.00	70.00	Nitric Acid	249.376
51	51	10.00	70.00	Zncl	320
21	52	10.00	110.00	Nitric Acid	200.576

3.2 Selection of a good predictive model.

Like the ANOVA, the sequential model sum of square was used to compare different models. It shows the statistical significance of adding new model terms step by step in increasing order. It provided accounts of variation and associated P-values (Prob>F) so that one can see how far it is worth going in degree of polynomial. The objective was to add a higher level source of term only if it explains a significant amount of variation beyond what was already accounted for.

The model was selected based on the highest order model that was significant (P-value small) and not aliased, on lack of fit (P-value > 0.10) and reasonable agreement between Adjusted R-squared and predicted R-squared (within 0.2 of each other). The summary table of the sequential model sum of square for the fibre is shown in table 4.4

TABLE 4

MODEL SUMMARY TABLE FOR AMPELOCISSUS CAVICALIS

Source	**Sequential P-Value**	**Lack of fit P-Value**	**Adjusted R-Squared**	**Predicted R-Squared**
Linear	L0.0001	0.0034	0.3945	0.2902
2FI	L 0.0001	0.0756	0.8575	0.7315 suggested
Quadratic	0.9384	0.0559	0.6402	0.4691
Cubic	0.2707	0.0575	0.6711	-0.4085 aliased

The lack of fit tests were included because extra design points beyond what was needed for the model were involved and some points were replicated (center points) to provide estimate of pure error. It compares the residual's error mean square to the pure error's mean square. Since it is a measure of risk, it is not desirable, so a small F value and probability greater than 0.1 were desired. From table 4, the fiber displayed non significant lack of fit for the suggested models. The predicted R-square was in close range to the adjusted R-squared for the model. The suggested model for Ampelocissus cavicaulis was two factor interaction models (2FI), addition of quadratic terms to the model did not improve the model. Although the linear model had low p-value for fibre, it was discarded due to significant lack of fit. The linear model been insignificant in the sequential model sum of square means that the error term at that stage still contains variation that can be explained by higher order terms, in

this case the 2FI. Therefore, it will be a mistake to say that the class of terms was not significant. As will be seen later on ANOVA, all the three linear terms A, B, and C may be significant at the 0.05 probability level. Even if all linear terms were insignificant according to ANOVA, one or more of them would be included in the final model to maintain model hierarchy. Notice from table 4, that adding cubic terms would not significantly improve the model for the fibre because it have P-value above 0.05. Even if it did, the central composite design lacks the design points needed to fit all terms required for the cubic, thus was labeled as being aliased.

3.3 Inspection of selected model

It is important to examine the suggested model if it provides an adequate approximation of the true response surface. The analysis of variance (ANONA) was used for this purpose.

TABLE 5

ANOVA TABLE FOR AMPELOCISSUS CAVICAULIS
R^2 = 0.9020, Adj-R^2 = 0.8575, pred-R^2 = 0.7315 adeq – precision = 16.1333.

ANOVA was used to interpret the relative contribution of each factors to the total variations "equally, R-squared, predicted R-squared and adjusted R-squared values were used to ascertain if the model selected will produce good prodiction for average outcome.

Attention was focused on predicted R-squared and adjusted R-squared because the regular R-squared can be artificially inflated by simple continuing to add terms to the model, even if the terms are not statistically significant. The adjusted R-squared plateaus when insignificant terms are added to the model, and the predicted R-squared will decrease when there are too many insignificant terms. A role of thumb is that the adjusted and predicted R-square values should be within 0.2 of each other. There is no "cut off" value for R-squared [7].

The model was deemed appropriate in this study based on the significance of the model p-value, insignificant lack of fit test, good agreement between adjusted and predicted $R^{\Lambda}2$, adequate precision over 4 and well behaved residuals. Insignificant lack of fit was desired because significant lack of fit means that the variation of the replicates about their mean values is less than the variation of the design points about their predicted values. Either the runs replicated well or their variance is small, or the model does not predict well, or some combination of the two. Adequate precision measures the signal-to- noise ratio. It compares the range of the predicated values at the design points to the average prediction error. The ANOVA for the fibre is shown on table 5.

The model F-value of 12.66 implied that the selected model

Source	Sum of Square	df	Mean Square	F Value	P-value prob>F
Model	99551.65	8	12443.96	12.66	<0.0001 suggested
A-Chemical Conc.	4247.45	1	424745	4.32	0.0436
B-Time	4862.12	1	4862.12	4.95	0.314
C-Chemical type	55258.35	3	18419.45	18.74	<0.001
AC	35183.73	3	11727.91	11.93	<0.0001
Residual	42268.59	43	982.99		
Lack of Fit	327529	27	123.01	2.04	0.0694 not significant
Pure error	9517.21	16	594.83		
Cur Total	1418E+005	51			

was significant. There is only 0.01% chance that a model F-value" this large could occur due to noise. Values prob< F less than 0.0500 indicated that the model term were significant. Values greater than 0.1000 indicated the model terms were not significant. For Ampelocissus cavicaulis fiber, linear effect of chemical concentration (A), Linear effect of time (B), linear effect of chemical type (C) and interaction effects of chemical concentration and chemical types (AC) were significant.

The lack of fit F-value of 2.04 implied it was not significant and there was a 6.94% chance that a lack of fit F-value" this large could occur due to noise. Insignificant lack of fit is desirable because it is a measure of risk. The predicted R-squared of 0.7315 is in agreement with the Adj R-square of 0.8575. 90.2% of the variability of the response data around it's mean was explained by the model. Adequate precision of 16.133 indicated an adequate signal.

The ANOVA confirmed that the model selected was adequate to predict the response well.

3.4 Predictive model in coded and actual form

Predictive model is mathematical representation of the chemical treatment process. The model equation was presented in both coded and actual values. Regardless of the form of the model, it is only an approximation, not the real truth. It is good enough to help you move in the proper direction, but not to make exact prediction particularly outside the actual experimental region.

Typically, a categoric factor's level are represented by indicator "dummy" variables in regression analysis. The value of the dummy variables are "0" if that types is not present in that treatment/run, and "I" if it is present, therefore, the four chemical types were represented by 100, 010, 001, and -1-1-1 respectively

The coded equation involving categoric factor can be seen as being four equations one comprising C[1] with its interactions, two comprising C[2] with its interactions, three comprising C[3] with all its interactions, and C[4] is seen as the reference level of the categorized factors. The Equation for C [4] is one with all the C terms and interaction terms were eliminated. Each chemical type adjusts the intercept by the amount of its coefficient, while its effect on interaction with other factors affects the slope due to the factor.

Final Equation in terms of coded factors:

Tensile strength (Mpa) = 2.99.02 – 941A – 10.06B+ 33.54 C[1] – 48.13 C[2] +26.03 C[3] + 35.99 AC[1] – 28.39AC[2] +16.23AC[3] (4)

Final equation in terms of actual factors:

NaOH :

Tensile strength (Mpa) = +301.34542+6.644.83 chemical concentration – 0.50323 Time (5)

Nitric acid:

Tensile strength (Mpa) = + 380.61744 -9.45000 Chemical concentration - 0.50323 Time (6)

Acetic anhydride:

Tensile strength (Mpa) = +343.23.23688+1.70533 chemical concentration – 0.50323 Time (7)

Zinc Chloride:

Tensile strength (Mpa) = + 405.87729 – 8.30700 chemical concentration – 0.50323 Time (8)

Using model equations involving categoric factors in predicating responses is always a complex issue, the final equation in terms of coded value in equation 4 can be seen as being four equations, one for each type of chemical as thus;

Tensile strength (Mpa) - +299.02 – 9.41A – 10.06B +33.54C[1] + 35.99AC[1] (9)

Tensile strength (Mpa) = +299.02 – 9.41A – 10.06B +48.13 C[2] + 28.39 AC[2] (10)

Tensile strength (Mpa) = +299.02 – 9.41A – 10.06B +26.03 C[3] + 16.23AC[3] (11)

Tensile strength (Mpa) = +299.02 – 9.41A – 10.06B (12)

In equation 9, the presence of the chemical type "100" adjusted the intercept positively by 33.54 and also, its effect affected the slope due to chemical concentration (A). It increased the sensitivity of tensile strength due to chemical concentration by 35.99. The presence of chemical type "010" adjusted the intercept negatively by 48.13 and decreased the sensitivity of tensile strength due to chemical concentration by 35.99. The presence of chemical type "010" adjusted the intercept negatively by 48.13 and decreased the sensitivity of tensile strength due to chemical concentration by 28.39 as indicated in eqn.10. The presence of chemical type "001" adjusted the intercept positivity by 26.03 and increased the sensitivity of tensile strength due to chemical concentration by 16.23.

Equation 12 is the reference level and was used when all the chemical types and their interaction with other factors were eliminated.

3.5 Diagnosing residuals to validate statistical assumptions

A good way to check your model is to enter factor levels from the design and generate the predicated response. When the predicated values are with the actual (observed) value, one will always see a discrepancy. This is called the residuals (noise). Residuals are differences between the predicated values and the actual values.

For statistical purpose, residuals were assumed to be independent of each other and distributed according to a normal distribution with constant variance [7]. Fig.1 below showed how the residuals were obtained.

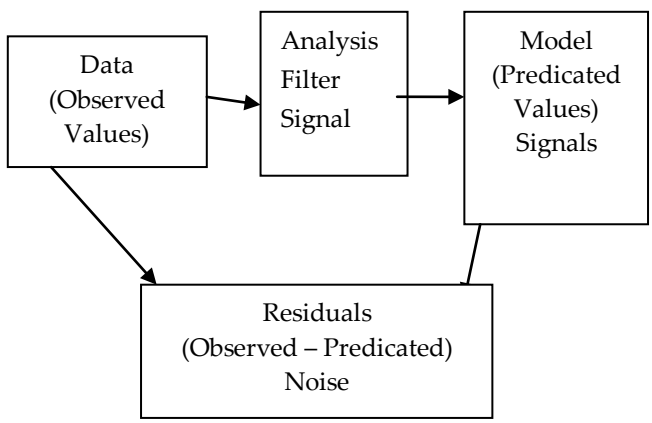


Fig.1 Derivation of residuals [7]

These residuals generated from fig. 1 were examined for patterns that indicate that some something other than noise was present.

TABLE 6

THE ACTUAL VALUES, PREDICATED VALUES AND THE RESIDUALS

Standard order	Actual values	Predicted values	Residuals
1	316.26	316.05	0.21
2	408.00	369.21	38.79
3	320.21	295.92	24.28
4	416.00	349.08	66.92
5	312.58	279.41	33.17
6	378.29	385.73	-7.44
7	328.09	352.70	-24.61
8	315.94	312.44	3.50
9	305.60	332.57	-26.97
10	240.00	332.57	-92.57
11	330.18	332.57	-2.39
12	320.78	332.57	-11.78
13	331.46	332.57	-1.10
14	314.02	298.76	15.27
15	208.58	223.16	-14.58
16	316.58	278.63	37.95
17	205.85	203.03	2.82
18	312.58	326.49	-13.92
19	193.86	175.29	18.57
20	320.00	271.02	48.98
21	200.58	230.76	-30.19
22	238.66	250.89	-12.23
23	244.62	250.89	-6.28
24	240.18	250.89	-10.72
25	216.72	250.89	-34.17
26	249.38	250.89	-1.52
27	318.40	328.30	-9.90
28	328.63	341.94	-13.31
29	320.63	308.17	12.46
30	336.26	321.82	14.44
31	312.80	311.42	1.38
32	340.80	338.70	2.10
33	327.50	345.19	-17.69
34	329.60	304.93	24.67
35	328.30	325.06	3.24
36	306.04	325.06	-19.02
37	328.31	325.06	3.25
38	329.52	325.06	4.46
39	318.98	325.06	-6.08
40	319.02	330.87	-11.86
41	240.42	264.42	-24.00
42	341.05	310.75	30.30
43	208.10	244.29	-36.19
44	314.30	354.04	-39.73
45	220.71	221.13	-0.41
46	319.91	307.71	12.20
47	202.17	267.45	-65.28

48	316.18	287.58	28.59
49	336.00	287.58	48.42
50	270.49	287.58	-17.09
51	320.00	287.58	32.42
52	330.22	287.58	42.64

A quick but effect tool for diagnosing residuals is the normal plot of residuals, residuals versus predicated level and predicated values versus actual values [7].

3.5.1 Normal plot of residuals

Normal plot of residuals indicates whether the residuals followed a normal distribution; in which case the points will follow a straight line, one should expect some moderate scatters which are normal even with normal data. The normal plot of residuals for the fibre is shown on fig. 2.

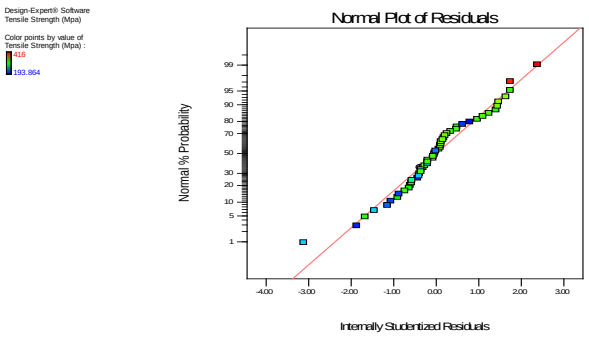


Fig. 2 Normal plot of residuals

From the plot, it showed that some of the plots lined up nicely as expected from a normal distribution while some had minor deviation from linear. In all, it obeyed the "pencil test" which is the effective way to assess the normal plot of residuals. Signal may be detected if clearly non linear, Pattern, such as "S" shape is detected on the plot [7].

3.5.2 Residual versus predicated levels.

This plot of residuals versus predicated response values was used to test the assumption of constant variance. The plot should be a random scatter (constant range of residuals across the graph). The plot of residuals versus predicated levels for the fiber is shown in fig. 3 below;

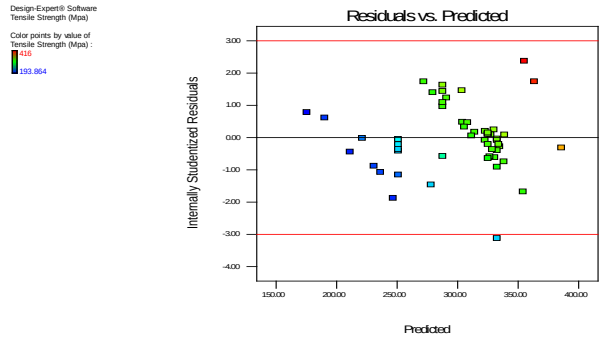
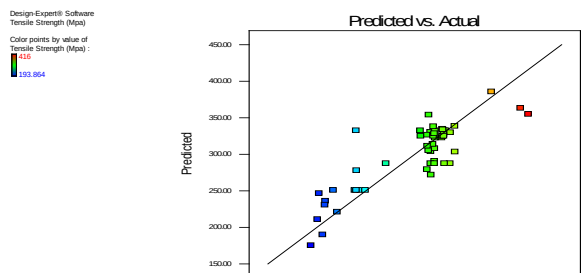


Fig. 3 Plot of Residuals vs. Predicted values

This plot provided a handy diagnostic for non-constant variance. From this plot, the patterns exhibited the hoped-for random scatter. Therefore, there is no definite increase in residuals with predicated level, which supports the underlying statistical assumption of constant variance. Signal will be present only when megaphone (<) pattern is detected, where the residuals increase with predicated value.

3.5.3 Predicated versus actual values.

A graph of the actual response value versus the predicated response values was used to detect a value, or group of values that were not easily predicated by the model. The condition is that the data point should be split evenly by the 45 degree line [15]. Fig 4 shows the plot of predicated versus actual values for the fibre.



From the plot, it showed that the data points were split evenly by the 45 degree line. This means that all the values where well predicated by the model.

The results of the diagnosis revealed no problem, which showed that the model met the assumptions of ANOVA and can be used to navigate the design space.

3.6 Effect of chemical type on the tensile strength of the fiber

Chemical types been a categoric factor was used to compare the magnitude of the effects of different chemicals on the tensile strength of the fiber. The graph of the effect of chemical types on the tensile strength of Ampelocissus cavicaulis is shown in fig. 5. From the graph, it can be seen that NaoH had highest effect on the tensile strength of the fiber, followed by acetic anhydride, zinc chloride and lastly nitric acid.

The effects were ascertained at time of 70mins and 10% chemical concentration.

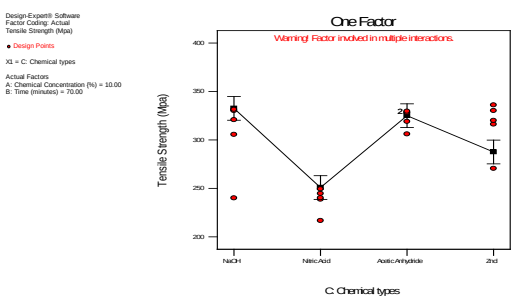


Fig. 5 Effect of chemical type on the tensile strength of the fibre.

3.7 Process optimization

Numerical optimization was used to search the design space using the model created during analysis to find factor settings that met the defined goal. Maximization of tensile strength was set as goal to be met for the optimization. The factor setting used for the optimization was selected based on the highest desirability.

The Optimum Conditions based on the Categoric Factors involved for Ampelocissus cavicaulis is as follows:

1. 6% Na0H for 5minutes with predicated ultimate tensile strength of 369.21 2Mpa
2. 14% Acetic anhydride for 70minute with predicated ultimate tensile strength of 341.944 Mpa.
3. 6% Nitric acid for 50 minutes with predicated ultimate strength of 298.25% Mpa.
4. 3% zinc chloride for 70 minutes with predicated ultimate strength of 330.84 Mpa.

3.7.1 Validation of optimum conditions

The optimum conditions obtained based on the predicated models were validated to confirm the predicated tensile strength and to obtain the percentage deviation (error) from the predicated ultimate conditions.

TABLE 7

VALIDATION OF THE OPTIMUN CONDITIONS

Model desir.	Chem. strength (%)	Chem. type	Time (mins)	Tensile strength (Mpa)		Error (%)
				Pred. Values	Exp. values	
1.00	6.0	NaOH	50.0	369.212	365.62	0975

0.928	14.0	Acetic anhydride	70.0	341.944	338.52	1.0
0.829	6.0	Nitric acid	50.0	298.756	295.111	1.22
0.723	3.0	Zncl	70.0	330.874	326.407	1.35

Table 7 shows the model desirability's, the optimum conditions, predicated and experimental ultimate strength with their percentage errors for the fiber.

4 CONCLUSIONS

This study inspected the effect of different types of chemicals, the strength of the chemicals, and the pretreatment time on the tensile strength of Ampellocissus cavicaulis fibre. A model equation was generated and optimized. The following conclusions can be drawn:

1. The type of chemical used in treating the fiber affects the resultant tensile strength.
2. The chemical concentrations equally affect the resultant tensile strength.
3. The interaction effects of the chemical and its concentrations affect the resultant tensile strength.
4. Two factors interaction model was deemed appropriate based on the diagnosis for predicting the tensile strength.
5. The optimum conditions using different chemicals were obtain as thus;
   - 6% NaoH for 50minues
   - 14% Acetic anhydride for 70minutes
   - 6% Nitric acid for 50minutes
   - 3% Zinc chloride for 70minutes
6. The optimum conditions were validated with little errors of less than 2.0%.

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