IMPACT OF FIBERS WEIGHT PROPORTION AND SURFACE TREATMENTS ON THE RELIABILITY OF HYBRID EPOXY COMPOSITE

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Abstract

Natural fiber-reinforced composites (NFRCs) are attracting growing interest in many technical applications owing to their environmentally sustainable and economical properties. This study investigates the impact of weight proportion of fibers and surface treatments on the tensile strength of hybrid epoxy composites reinforced with hemp, jute, and bamboo. Prior to composite fabrication, the fibers underwent alkali and saline treatments at different concentrations (3, 5, and 7 wt. %). The composites were fabricated via the hand lay-up technique by varying wt. proportion of fibers (10, 15, and 20 wt. %). The tensile strength is optimized using Response Surface Methodology (RSM), which yields highest tensile strength of 63.11 MPa at 14.24 wt. % of fibers fraction and 5.40 wt. % concentration of alkali treatment. Findings of this study demonstrate that reliability of the hybrid composite significantly enhanced due to improvement in strength of the composite. The treated hybrid composites have favourable properties, rendering them a feasible and sustainable option for automobile components and other lightweight structural applications.

Keywords: *Natural fibers, hybrid composite, Surface treatments, Tensile strength, Reliability*

I. Introduction

Natural cellulose fibers are extensively used in various engineering applications, such as construction materials and vehicle structural components, where lightweight materials are essential. These plant-based fibers offer numerous advantages, including cost-effectiveness, low density, wide availability, excellent mechanical strength, thermal insulation, electrical resistivity, and environmental sustainability [1, 2, 3]. Today, a wide range of natural fibres is recognized and utilized in lightweight structural applications across the construction, automotive, and aerospace industries. Economically viable fibers such as hemp, palm, okra, kapok, silk, coconut, alfa, bamboo, jute, and abaca are extensively used in various products, including textiles, household items, ropes, bags, automotive components, and lightweight parts for the aviation sector [4, 5].

Despite their apparent advantages, natural fibers have certain disadvantages, such as lower strength than synthetic materials, inconsistent size and diameter, extreme sensitivity to water, intolerance to high temperatures, and reduced resistance to fungal and bacterial infections. To address these obstacles and enhance the mechanical characteristics of NFRCs, researchers employ various strategies, including chemical treatments, modifications to fiber alignment, stacking

sequences, hybridization techniques, and other innovative methods [6, 7, 8]. Hybrid natural fiber composites use the favorable qualities of one fiber to counterbalance the negative attributes of another fiber. For example, hemp fiber exhibits more flexural and tensile strength than jute fiber, but jute fibers show higher impact strength than hemp fibres. Therefore, the combination of hemp and jute fibers in hybrid laminates results in decreased brittleness and improvement in the mechanical characteristics of the composite[9]. Hybrid NFRCs leverage the strengths of different fibers to compensate for their respective weaknesses. Hemp fibers, known for their brittleness and toughness, contrast with jute fibres, which exhibit excellent elongation properties [10, 11]. These composites achieve reduced brittleness and enhanced mechanical strength by integrating hemp and jute fibers in hybrid laminates. Bamboo fiber exhibits a high specific tensile strength that surpasses that of steel [12].

Reinforcing hybrid epoxy composites with snake grass and mudar ranging from 10 to 40 wt. % is possible. Reinforcement of fibers at 30 wt. % produced the best results. The optimal strength was achieved with a blend of 30 wt. % flax and 10 wt. % jute fiber [13]. The results showed that the impact strength improved by 40.6% and the flexural strength by 35% at 30 wt. % fiber content when banana fiber reinforcement was added to low-density polyethylene at a weight percentage ranging from 10 to 30 wt. % [14]. The hand lay-up method was used to create hybrid epoxy composite reinforced date palm fibers with varied weight percent of fiber. The mechanical characteristics of the composite were observed to be the highest at a fiber concentration of 50 wt. percent [15]. In contrast to jute fibers, which have exceptional elongation qualities, hemp fibers are infamously strong and brittle. Hybrid laminates that have hemp and jute fiber reinforcement are less brittle and have greater mechanical strength. Hemp and jute fiber hybridization greatly improves the composite's strength, according to the study. The composite with the perfect combination of hemp fiber (at 10% by weight) and jute fiber (at 40% by weight) demonstrated the highest levels of flexural strength, impact resistance, and tensile strength [16]. Highest mechanical and tribological characteristics were obtained at 15 wt. % reinforcement in hybrid epoxy composite [17].

Numerous researchers have highlighted in the literature that various surface treatments can further enhance the properties of composites. Among these, alkali and saline treatments have proven to be the most effective in enhancing composites' mechanical properties [18]. Alkaline surface treatment is often used to increase natural fibres' characteristics in composite materials [19, 20, 21]. This is achieved by chemically pre-treating natural fiber with NaOH. The surface treatment removes waxes and hemicellulose from the fiber surface, exposing more bare surface area. Which enhances adhesion, resulting in improved bonding and, consequently, better mechanical strength of the composite [22]. Yusuf et al., The flexural strength of epoxy reinforced with kenaf fiber increased by 36% following alkali surface pretreatment at a 6% concentration [23]. Vekateshwaran et al., Fabricated Epoxy composites reinforced with banana fibres, with NaOH pretreatment, applied at different concentrations. The study depicted that the highest strength was achieved at a 1% NaOH concentration, with the treated composites showing improved strength across all concentrations [24]. Goriparthi et al., fabricated a composite by reinforcing jute fibers into PLA, followed by alkali treatment. It was observed that the treated composite exhibited enhanced mechanical properties compared to the untreated one [25]. Sisal fibers were treated with NaOH solutions at concentrations of 3%, 6%, and 9%, thenrinsed with distilled water and dried. The highest tensile strength was achieved with the 3% NaOH treatment [26].

II. Methodology

The experiment was created with the help of the Design Expert13 program. Evaluating the effect of different variables on many replies allows this program to adequately examine diverse experimental

designs and further improve their findings. Based on previous research and results of trial and error, three variables were picked from the literature and their values were determined. Because of its superior capacity for higher-order modeling, enhanced efficiency in estimating quadratic terms, and backing from previous research, the RSM investigation utilized the optimal (custom) design approach. The study used an optimum (custom) design approach using RSM to produce twenty different trial combinations. A predictive model was developed by taking process parameters as inputs and hardness as a response variable. After that, we optimized the process parameters until we reached the maximum tensile strength. Figure 1 shows the approach that was used.

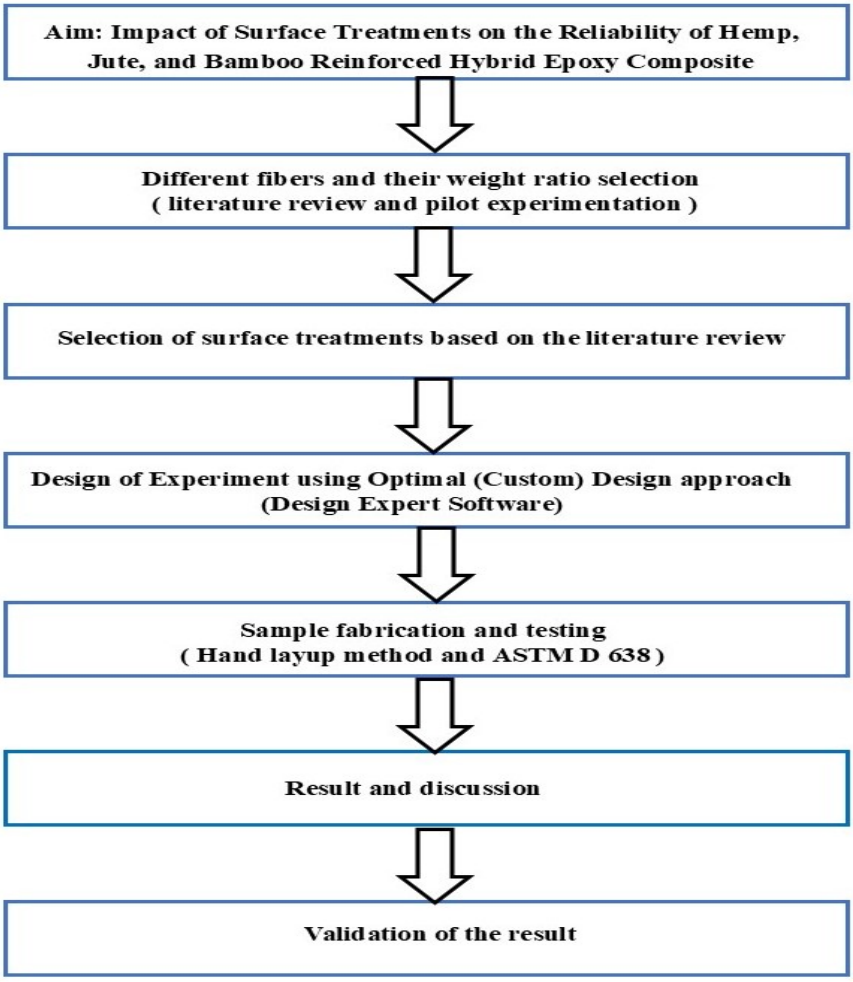

Figure 1: *Methodology implemented*

I. Experimentation

Hemp Organization Pvt. Ltd. in Delhi, India, provided the hemp fibers, while Himanshu Jute Fab in Ashok Vihar, Delhi, provided the jute fibers. The supplier of bamboo fibers was Vruksha Composites, located in Guntur, Andhra Pradesh, India. These natural fibers were used in their long fibrous forms, and were selected for their advantageous mechanical properties in composite manufacturing. The matrix material used in this study consisted of epoxy resin LY556 mixed with hardener HY 951 in a precise ratio of 10:1. Herenba Instruments & Engineers, Chennai, India, supplied both materials. The hardener was crucial for curing the epoxy, ensuring proper setting and improving the durability of the final composite. Additionally, wax from Jai Shree Balaji Sales

Corporation, New Delhi, was used as a releasing agent to ensure smooth demolding during fabrication.

The properties of NFRCs are largely influenced by the type of surface treatment and the concentration levels used. This study investigates the effects of alkali and saline surface treatments. Sodium hydroxide (NaOH) and (3-Glycidyloxypropyl) silane were selected for these treatments. Fibers were immersed in NaOH solutions at concentrations of 3 wt. %, 5 wt. %, and 7 wt. % for 30 minutes. After soaking, the fibers were thoroughly washed with distilled water until the pH of the water reached 7, then dried in an oven and subsequently straightened through hammering and combing before composite fabrication. For the saline and acetylation treatments, the same fiber types were soaked in saline and acetylation solutions at identical concentrations for 1 hour, followed by drying in an oven at 70°C for 24 hours. This procedure was repeated for each treatment concentration.

Using the hand lay-up process, the hemp, jute, and bamboo fiber-reinforced hybrid epoxy composite was produced. The mold, constructed from mild steel, has cavity dimensions of 200×180 mm, with the upper part slightly smaller at 199.5×179.5 mm. In this study, hybrid epoxy composites were fabricated by varying weight proportions of fibers (10, 15, and 20 wt. %) and with parameters as depicted in Table 1. These fibers were taken in long fibrous form. The fibres underwent different surface treatment process, followed by drying in an oven and subsequent straightening by hammering and combing before composite fabrication. Initially, PVC polythene was applied to coat both the upper and lower mold surfaces, followed by the application of wax to prevent resin adhesion. The prepared fibres were then carefully arranged in the mold cavity. Epoxy resin, pre-mixed with hardener at a 10:1 ratio and thoroughly blended, was poured onto the fibers. A metallic roller was employed to remove bubbles and gases from each fiber layer meticulously. The composite was then left to cure for 24 hours at room temperature while a 20 kg dead weight was positioned on the mold. After curing, the composite was demolded, and Specimens measuring 115 × 19 × 4 mm and having a gauge length of 25 mm were manufactured for tensile testing in accordance with ASTM D 638 standard as depicted in Figure 2.

Figure 2: T*esting setup for tensile test and specimens according to ASTM standard*

Table 1: *Requirement for single-objective optimization of tensile strength*

Input variables	Units	Level 1	Level 2	Level 3
A: Fibers fraction	Wt. %	10	15	20
B: Solution concentration	Wt. %	3	5	7
C: Treatment type	-	1	2	3

II. Statistical Analysis

Design-Expert 13 was used to evaluate the data collected from the experiments. For the purpose of tabulating the combination of input variables at their distinct levels, centre composite design was employed. In order to select the best polynomial model with a significance level of $p<0.05$, several quantitative features, including coefficients of deviation, lack of fit, computed and shifted multiple correlation coefficients, were thoroughly examined. We used the DX-13 program to make interaction, factor, and three-dimensional graphs so we could see how the input variables affected the responses.

III. Results and Discussion

Specimens were manually produced using the hand lap up method and evaluated in accordance with ASTM standards. Table 2 presents the calculated tensile strength results. A mathematical model was developed utilizing twenty permutations of three input factors and the hardness of the resultant nanocomposite. Equation illustrates the mathematical relationship derived from the calculation of tensile strength coefficient values.

$$\text{Tensile strength} = +62.01 - 2.36\,A + 1.03\,B + 0.8057\,C[1] - 0.1781\,C[2] + 0.2262\,AB - 0.2549\,AC[1] - 0.2249\,AC[2] - 0.1144\,BC[1] - 0.2463\,BC[2] - 8.47\,A^2 - 2.18\,B^2$$

Where, C[1], C[2] and C[3] are alkaline, saline and acetylene surface treatment respectively.

Table 2: *Experimental results for tensile strength*

Run	Factor 1	Factor 2	Factor 3	Response 1
	A:Fiber Fraction (wt. %)	B:Treatment Concentration (wt. %)	C:Treatment Type -	Tensile Strength
1	20	7	2	49.76
2	20	5	3	50.48
3	15	3	2	58.45
4	20	3	2	47.76
5	10	5	1	57.06
6	15	3	1	59.48
7	10	5	3	54.78
8	10	5	3	54.86
9	15	5	1	62.82

10	20	5	1	52.14
11	10	7	2	54.28
12	15	3	3	57.45
13	15	5	1	63.78
14	20	5	1	50.98
15	10	7	1	55.12
16	10	3	1	53.98
17	15	5	2	61.64
18	10	3	2	53.56
19	15	7	3	60.85
20	20	3	3	47.86

Table 3 shows the results of an ANOVA test conducted with a 95% confidence level, which was used to determine the model's appropriateness. Results demonstrated the usefulness of the model and its potential for use in predicting the shore hardness of the produced nanocomposite in the absence of actual experimental data. Statistical relevance and significance are indicated by the model's p-value, which is less than 0.0001. Important model terms in this instance include A, B, C, A^2, and B^2. The model's R2 is 0.998, indicating that approx. For all tensile strengths, the input parameters account for 99.8 percent of the variance.

Table 3: *ANOVA test results for tensile strength*

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	432.45	11	39.31	99.86	< 0.0001	significant
A-Fibers fraction	52.17	1	52.17	132.51	< 0.0001	
B-Treatment concentration	13.79	1	13.79	35.03	0.0004	
C-Types of treatment	8.46	2	4.23	10.75	0.0054	
AB	0.2517	1	0.2517	0.6394	0.4470	
AC	1.18	2	0.5899	1.50	0.2801	
BC	0.5941	2	0.2970	0.7545	0.5010	
A^2	303.30	1	303.30	770.39	< 0.0001	
B^2	14.92	1	14.92	37.90	0.0003	
Residual	3.15	8	0.3937			
Lack of Fit	2.01	5	0.4026	1.06	0.5133	non significant
Pure Error	1.14	3	0.3789			
Cor Total	435.60	19				

As depicted in Figure 3, the projected tensile strength values are in agreement with the experimental ones, and the difference between the two sets of values is also illustrated. Figure 2D and 3D

surface plots show the impact of changing various input factors on the tensile strength of the hybrid epoxy composite that was produced. The results reveal that as the fiber fraction enhances, the tensile strength of the produced hybrid composite decreases. The tensile strength is greatly enhanced with a rise in the fiber fraction, reaching its maximum at 15 % by weight and a decrease in tensile strength observed when this point is passed. The inadequate soaking of the fibers by the epoxy caused a weak connection between the fibers and the matrix, which resulted in this reduction.

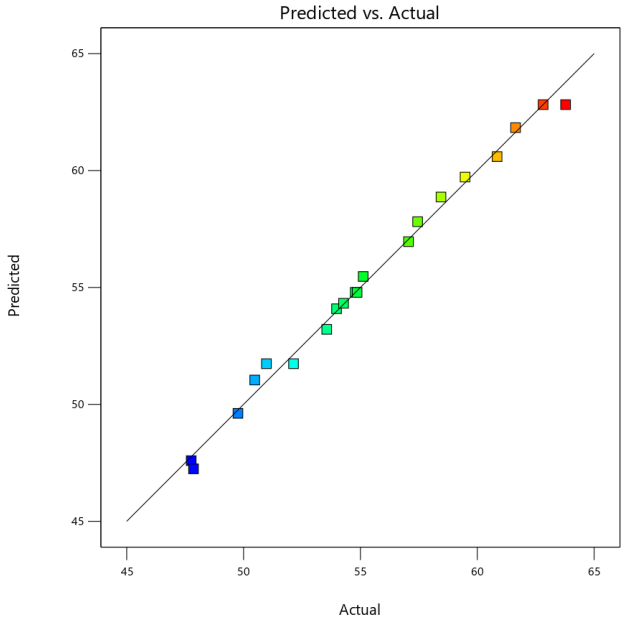

Figure 3: *Predicted v/s actual values for tensile strength*

The section presents 2D and 3D surface plots that show how the tensile strength is affected by weight proportion, surface treatment, and its concentration. Figure 1 shows how the tensile strength of the hybrid composite changes as a function of the weight proportion and the concentration level of the alkali treatment. The red area indicates the zone with the highest tensile strength, while the blue area indicates the zone with the lowest. The green and blue patches, respectively, show regions with moderate and low tensile strength. A maximum tensile strength of 63.78 MPa was achieved in the area contained by the 11–17 wt. % fibers, which also had the strongest reaction. In a similar vein, Figure shows how the tensile strength of the hybrid composite changes as a function of the treatment concentration level of saline (C2) and the alteration of the weight proportion. The area bounded by the 12–16 wt. % fibers showed the most reaction and had the maximum tensile strength value of 61.64 MPa. C3 surface treatment with acetylene resulted in a maximum tensile strength of 60.85 MPa.

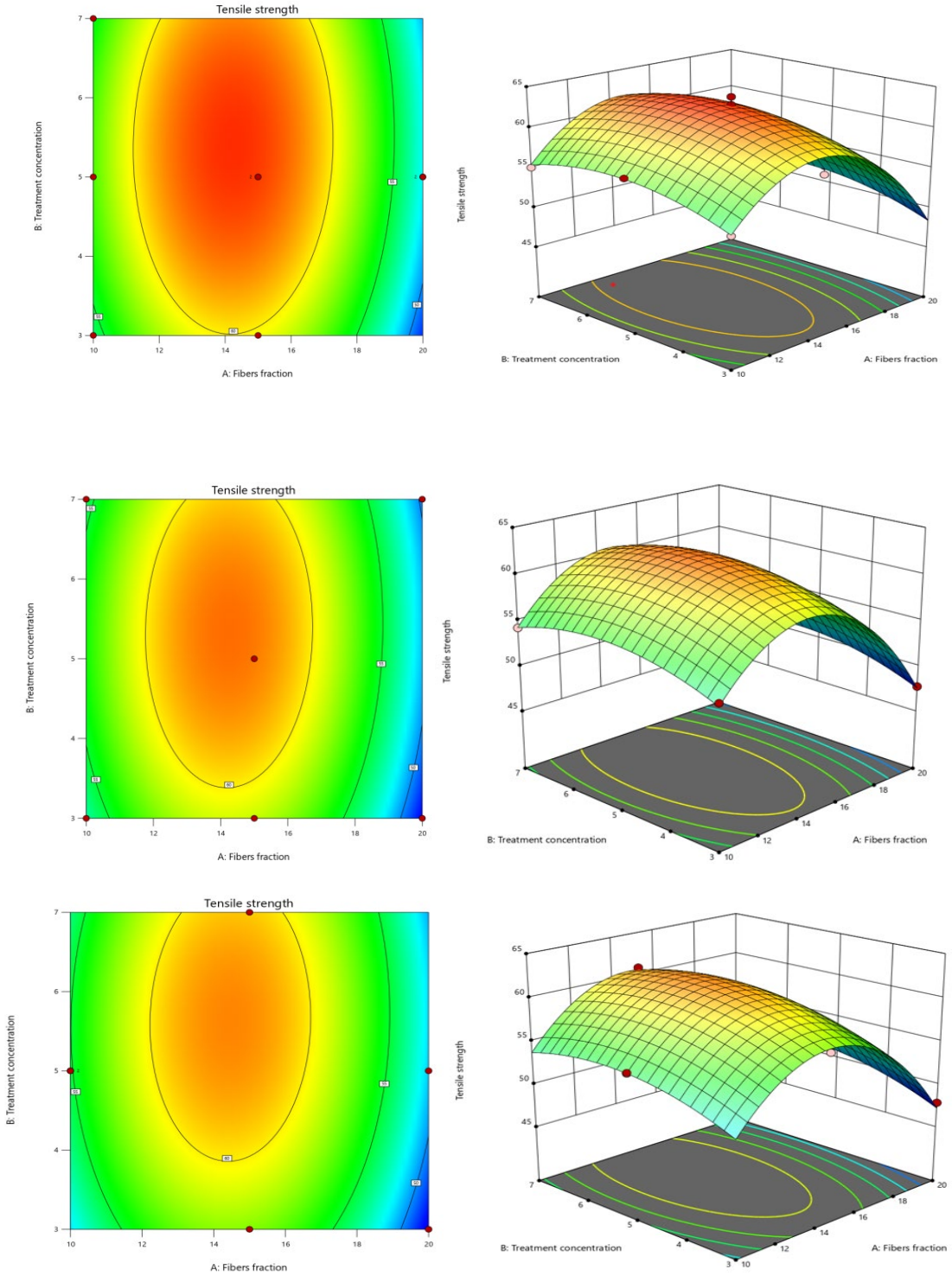

Figure 4: *2D and 3D surface plots illustrating the effect of fiber weight fraction, surface treatment type, and treatment concentration on the tensile strength*

Identical findings were reported for treatment concentration, with the maximum tensile strength recorded at a concentration of 5 wt. %. At this stage, fiber-matrix interaction was enhanced by the elimination of wax and hemicellulose, therefore increasing the exposed fiber surface area. Exceeding this concentration, tensile strength begins to diminish. The alkali surface-treated composite exhibited superior tensile strength at all concentrations due to enhanced surface

roughness, resulting in improved interlocking between the matrix and fibers.

In single-objective optimization of the response parameter, i.e., tensile strength, Table 4 shows the criteria for various process parameters. The objective is to optimize the hybrid epoxy composite's tensile strength within the given parameter range.

Table 4: *Requirement for single-objective optimization of shore D hardness*

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Fibers fraction	is in range	10	20	1	1	***
B:Treatmnt concentration	is in range	3	7	1	1	***
C:Types of treatment	is equal to	1	3	1	1	***
Tensile strength	maximize	47.76	63.78	1	1	***

All responses and criteria were accorded equal significance, and distinct objectives were established for each dimension. The response surface methodology (RSM) produced 76 potential options, from which the ideal solution was chosen based on greatest attractiveness and practical feasibility. The ideal combination of input parameters is determined to be 14.24 wt. % for fiber weight fraction, alkali surface treatment for type, and 5.4 wt. % for concentration level. The optimal values of the response parameters were determined as follows: Tensile strength: 63.11 MPa. The ramp function graphs in Figure 5 clearly depict these conclusions. The model was verified utilizing the optimal process parameters derived from the RSM-generated model. Three specimens were produced for each condition, and experimental findings indicated a maximum error of under 2.3%, therefore validating the model's precision and dependability.

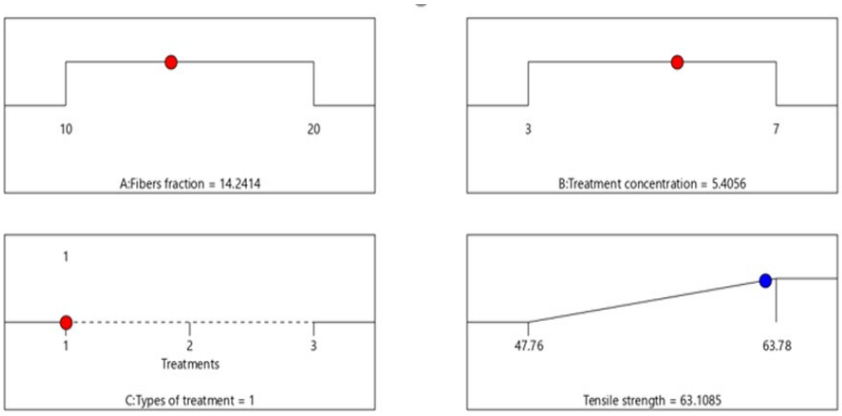

Figure 5: *Ramp function graphs for single-objective optimization of tensile strength*

IV. Conclusion

This study investigates the tensile strength of hybrid epoxy reinforced composites produced via the hand layup method under diverse processing conditions. The results indicate that alterations in process parameters significantly affect tensile strength, with fiber weight fraction and diverse surface treatments at different concentrations identified as critical variables. The tensile strength improves with the rising weight fraction of fibers, up to an optimal threshold. The model's robustness and

reliability are validated by the Response Surface Methodology (RSM), yielding an R2 score of 0.998. RSM optimization effectively maximizes tensile strength, achieving a maximum of 63.11 MPa at a fiber fraction of 14.24 wt. % and an alkali treatment concentration of 5.40 wt. %. The enhanced efficacy of alkali treatment is due to its capacity to eliminate surface contaminants, augment fiber roughness, and strengthen interfacial adhesion between the fibers and the matrix. The enhanced connection between the fiber and matrix facilitates superior stress transfer, hence improving mechanical characteristics. Although saline treatment enhanced fiber-matrix compatibility by altering the fiber surface, its impact was relatively less significant than that of alkali treatment.

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