Research Article

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Comprehensive Insights of Chemically Treated Jute/Kenaf/Glass Fiber with TiO_2 Nanoparticle using RSM Optimization

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ABSTRACT

In this research, we examined the impacts of silane treatment on jute/kenaf fiber mats. Our main objective was to enhance the bending strength using response surface methodology. Different concentrations of silane (5%, 10%, and 15%) and varying soaking durations (10, 20, 30 min) were examined. Based on the findings, it was discovered that the optimal bending strength was attained by using a 5% silane concentration and soaking for 20 min with 5% TiO_2 nanoparticle. Utilizing Design-Expert software, the study optimized these parameters and examined their impact on flexural characteristics. The model demonstrated impressive correlation coefficients (R^2 and Adjusted R^2) for flexural strength and hardness, suggesting its reliability in predicting properties. Additional experiments were conducted to confirm the accuracy of the model's predictions, and the results showed a minimal discrepancy between the predicted and actual values. This provides strong evidence for the model's reliability in making accurate predictions.

Keywords: RSM Optimization; Flexural properties; Hardness; Silane treatment; TiO_2 nanoparticle.

1. INTRODUCTION

There has been a growing interest in the development of environmentally friendly products that utilize natural raw materials, due to the increase in CO_2 emissions from traditional materials processing (Shahinur *et al*. 2020). There is a growing trend towards the usage of environment-friendly fibers in polymer materials, driven by their positive impact on sustainable development (Sajin *et al*. 2022). Extensive investigation into advanced material development highlights the diverse uses of natural fiber composites in different sectors (Vijay *et al*. 2021 and Khalid *et al*. 2021). Jute and kenaf fiber composites, for example, have the potential to replace traditional materials such as steel and wood, showcasing their exciting potential as composite materials (Sridhar *et al*. 2022). Understanding the characteristics and properties of jute/kenaf fiber is essential in determining its suitability (Tusnim *et al*. 2022). Extensive use has been found for hybrid composites made from jute/kenaf throughout different sectors (Chandekar *et al*. 2020 and Sarker *et al*. 2022).

In this study, we explored the efficacy of several chemical treatments to improve the efficiency of jute fibers in natural rubber laminates. Three diverse surface treatments were used to alter the jute fibers (JFs): alkali treatment, paired alkali/stearic acid treatment, and mixed alkali/silane treatment. Within all of the treatments, the combination of alkali/silane treated JF saturated natural rubber (NR) composite showed remarkable enhancements in mechanical properties. The structural analysis provides additional evidence of improved adhesion among the NR matrix and JFs. This enhanced bonding is a result of the paired alkali/silane treatment, which helps facilitate effective stress transfer. In terms of enhancing the bond between the NR matrix and JFs, the mixed alkali/silane treatment proved to be the most successful approach (Roy *et al*. 2020). This study delves into the recycling of jute fibres obtained from different jute waste materials and their application in creating hybrid composites (Nag and Kumar *et al*. 2023). Composite fabrication involves the use of two types of recycled jute sheets: woven and nonwoven (a blend of jute and polypropylene). The jute sheets are subjected to sodium hydroxide (NaOH) treatment with various concentrations (0%, 2%, 4%, 6%, and 8%) to explore the connections. This study specifically highlights the exceptional properties of JJJ bonded sheets, that are extremely valuable for a wide range of sectors, such as the automotive, and aircraft (Malladi *et al*. 2024).

The bond amongst the jute and coir fibers and the matrix was enhanced by incorporating them into a polycaprolactone solution before fabrication. The mechanical properties of biocomposites reinforced with alkali-silane-treated fibers demonstrated a notable enhancement when compared to untreated fibers (Islam

et al. 2023). In addition, the authors explored characteristics such as moisture absorption. The biocomposites that underwent alkali-silane treatment showed the most positive outcomes in all aspects. In addition, an investigation was carried out to track the breakdown of the biocomposites over 90 days (Hossen *et al.* 2020). Kenaf is used in the furniture and automotive industries to manufacture medium-density fiberboard and different composite materials for structural applications. Kenaf exhibits exceptional mechanical performance due to its cellulose concentration, which typically ranges from 40% to 80%. Before being mixed with the polymer matrix, kenaf fibres undergo chemical treatment to improve their bonding with the polymer framework and boost the performance of the composite. Alkaline treatment using NaOH solution is frequently used among several chemical treatments. Silane treatments on the surface of kenaf fibers and their effect on the production and mechanical characteristics of polymer composites were investigated (Nurazzi *et al*. 2021).

The kenaf fibers, after being treated with NaOH, displayed a pristine surface that was free from any contaminants or impurities. Unexpectedly, the alkali treatment was discovered to adversely impact the mechanical characteristics of kenaf fibers. Composites with unaltered kenaf and modified kenaf fibers improved mechanical properties when compared to neat epoxy, with increases of 72.85% and 12.97%, accordingly (Ismail *et al.* 2021). The use of processed kenaf fibers as reinforcing material resulted in tensile strength drop. Moreover, the performance of the composite was greatly affected by temperature conditions. Treated kenaf fibers exhibited thermal stability and were less affected by temperature changes. This led to a substantial 76% reduction in loss when the composite was reinforced with treated kenaf fibers (Behera *et al*. 2022). The mechanical parameters, including tensile and flexural strengths, as well as tensile and flexural modulus of both modified and unmodified kenaf fiber mat reinforced polypropylene (KFM-PP) biocomposites were evaluated using a universal testing machine (UTM) (Sabri *et al*. 2020). The results demonstrated that the use of peroxide treatment resulted in superior performance in terms of mechanical qualities when compared to alternative chemical treatments. The SEM morphological investigations verified that almost all treated KFM demonstrated excellent adhesion between the fibers and matrix, leading to a decrease in fiber pull-out, void formation, and smaller gaps at the interface. As a result, applying a chemical treatment to KFM enhanced the bonding between the fibers and the matrix, leading to superior physical and mechanical characteristics in comparison to KFM-reinforced PP biocomposites that were not treated (Birniwa *et al*. 2021).

The present study highlights the advantageous weight-to-strength ratios of glass fibres, rendering them appropriate for applications that prioritize lightweight materials. To decrease reliance on synthetic fibers, the study included natural fibers such as jute and kenaf into composites. Literature emphasizes the widespread application of jute in various industries due to its remarkable supporting capabilities, as well as the superior qualities of kenaf when coupled with other substances. In addition, the study investigated the integration of nanoparticles such as titanium dioxide (TiO_2) to improve mechanical strength. Several weight percentages (3wt%, 4wt%, and 5wt%) of nano-filler content were investigated. This work shows that inclusion of nano-filler content improved the strength of composite components, which is supported by relevant research.

2. MATERIALS AND METHODS

2.1 Materials

The study utilized a composite material consisting of a glass fiber mat (Fig. 1a), natural kenaf fiber (Fig. 1b) and a jute fiber mat (Fig. 1c). The fibers were purchased from Hayel Pvt Ltd, Chennai. The fibers were immersed in a solution containing silane at concentrations of 5%, 10%, and 15% for 10, 20, and 30 minutes, respectively, at ambient temperature. Later, the fibers were washed with water and subjected to a drying process in a 100°C oven for 30 minutes. The objective of this procedure was to remove non-cellulosic components to improve the rigidity of the fiber surface and provide better interaction with the matrix. The epoxy resin LY 556 and hardener (HY951), sourced from Jevanthee Enterprises, Chennai, were selected due to their benefits, such as robust bonding to different fibers, exceptional performance under elevated temperatures and remarkable mechanical and electrical characteristics. In addition, the nano-filler (TiO_2) (produced by Ad-Nano Technologies, Bangalore) was chosen because of its outstanding properties, including little shrinkage during the curing process and excellent chemical resistance, which sets it apart from other thermoset polymers.

The outcomes of silane treatment process parameters were investigated using Central Composite Design (CCD) research. Twenty experiments with varied fiber compositions were performed, combining three changing characteristics as stated in Table 1. These experiments were meticulously planned and executed. The procedure was facilitated with the Design Expert software. Table 2 provides comprehensive information about these compositions.

Table 1. Factors of the input parameter and their levels

S. No.	Entity	Units	L [1]	L [2]	L [3]
1	TiO_2	%	3	4	5
2	Silane	%	5	10	15
3	Time	Min	10	20	30

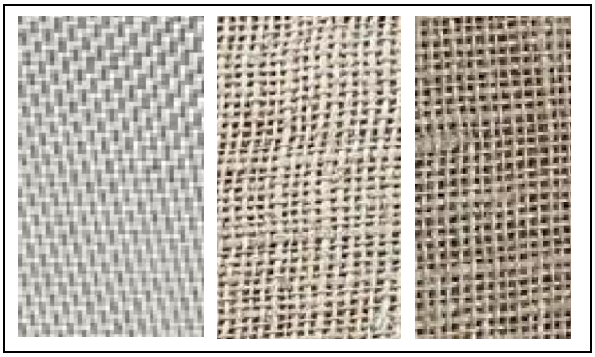

Fig. 1: (a) Glass fiber mat, (b) Kenaf fiber mat, (c) Jute fiber mat

2.2 Surface Treatment of the Fiber

The jute and kenaf natural fibers were extensively purified using multiple rinses with distilled water and then dried for 72 hours in an oven at a temperature of 35℃. This process was essential to remove impurities, such as dust and residual husk in the fibers, which might potentially interfere with material and epoxy bonding. During the washing and drying processes, the untreated jute and kenaf fibers were carefully preserved prior to the next chemical treatment and composite fabrication stages (Jena *et al*. 2023).

2.3 Composite Preparation

The hand lay-up apporach was employed to construct composite laminates by incorporating TiO_2 with fibers such as jute, kenaf, and glass. The quantity of nano-filler varied between 3 and 5 g. To facilitate removal and provide a level surface, a lubricating substance with a high oil content was applied to the bottom of the mould. The jute, kenaf, and glass fibres were precisely cut and arranged in a certain sequence to match the dimensions of the mould. The matrix consisted of a 10:1 blend of epoxy and hardener, with different amounts of TiO_2 nano-filler (3, 4, 5 g). During the lay-up process, a roller was employed to eliminate air bubbles and excess matrix. The G/J/J/K/K/K/K/J/J/G stacking sequence was determined to be the preferred option based on the pre-test findings. Layout of the fabrication process are shown in Fig. 2.

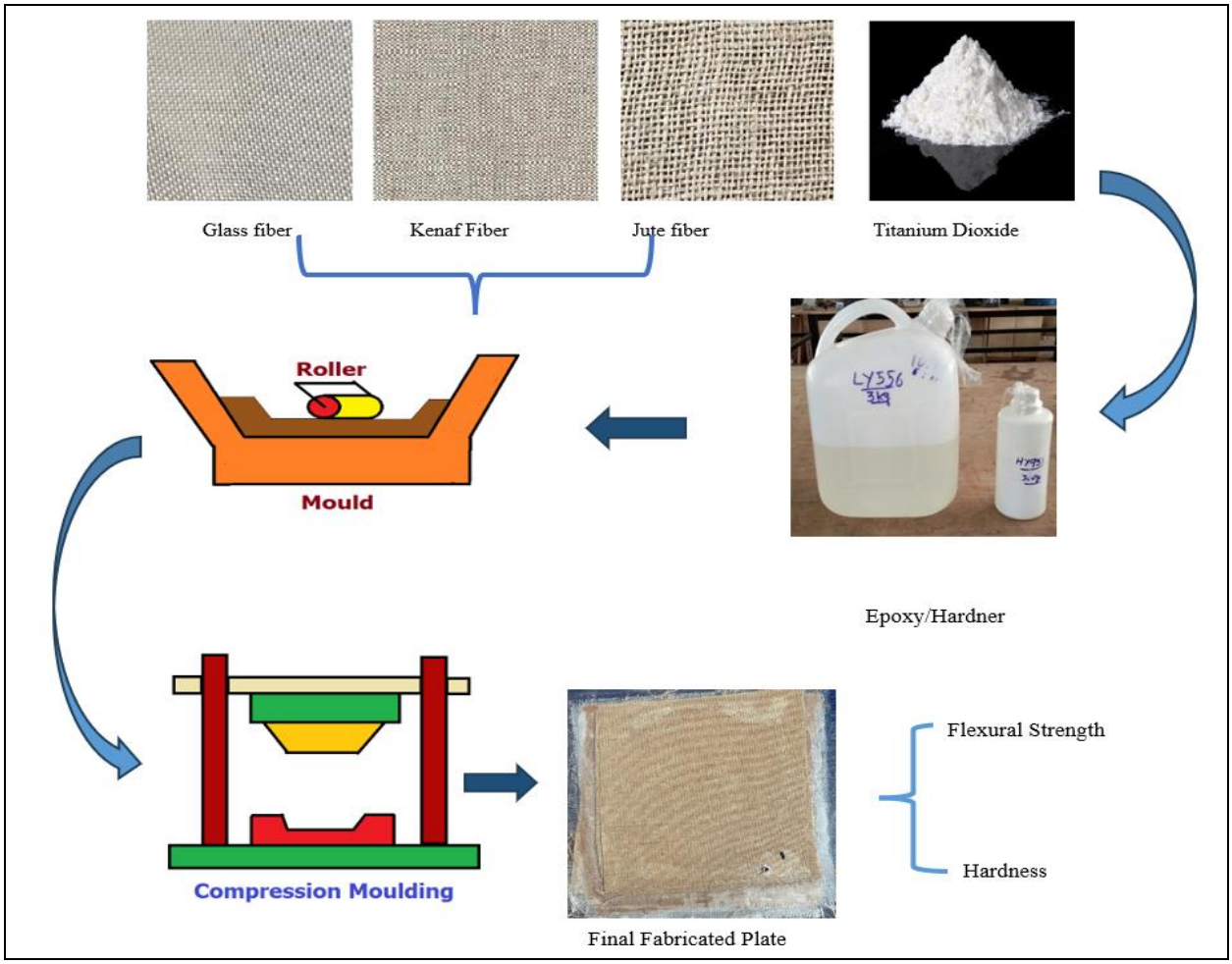

Fig. 2: Layout of composite laminate flow process

2.3.1 Evaluating Techniques for Composite Materials

Flexural testing

The flexural testing of materials was conducted using a UTM following the guidelines of ASTM D790. The specimens, measuring 127 × 12.7 × 3 mm, were subjected to specific parameters, including a load of 58 kN, crosshead speed of 1.3 mm/min, and a span length of 50 mm. The ultimate flexural stresses were determined at a maximum deflection of 6 mm.

Hardness Test

The hardness test was performed using a Shore D hardness tester by ASTM D2240. In each instance, three random experiments were carried out on the surface.

Table 2. Flexural strength and hardness attributes of nano-composites

Std	Run	A: TiO_2 (%)	B: Silane (%)	C: Time (Min)	Flexural Strength (MPa)	Hardness (HRC)
1	1	3	5	10	101	94
7	19	3	15	30	104	95
5	2	3	5	30	106	97
3	14	3	15	10	108	99
9	11	3	10	20	111	101
13	7	4	10	10	118	106
12	8	4	15	20	120	109
20	4	4	10	20	122	110
14	6	4	10	30	123	109
15	16	4	10	20	124	113
18	3	4	10	20	125	116
19	17	4	10	20	125	114
17	9	4	10	20	126	115
11	18	4	5	20	127	117
16	20	4	10	20	128	119
2	5	5	5	10	132	121
4	10	5	15	10	135	123
6	15	5	5	30	136	125
8	13	5	15	30	139	128
10	12	5	10	20	143	131

2.4 Experimental Design

This study employed response surface methodology (RSM) to forecast results and examine the impact of input parameters on both flexural and hardness properties. ANOVA evaluated the significant impacts of silane concentration, time, and nano-filler on the outputs of measured technical parameters. The analysis categorized input parameters according to their effect on the results. The values supplied were obtained from experiments done using Design Expert V13, utilizing a statistical experimental design approach to estimate mixing ratios. The study consisted of 20 experiments, specifically examined two response variables: flexural and hardness properties. The experimental design incorporates three control parameters denoted as (i) TiO_2 nano-filler, (ii) percentage of silane treatment, and (iii) treatment period. Table 1 provides a clear overview of the three control levels associated with components A and B. Table 2 presents the measured values of tensile strength and impact strength obtained under different testing settings. The findings were obtained by the use of an Optimal (custom) Design, taking into account three factors: the percentage of silane, the concentration of silane, and the percentage of nano-filler.

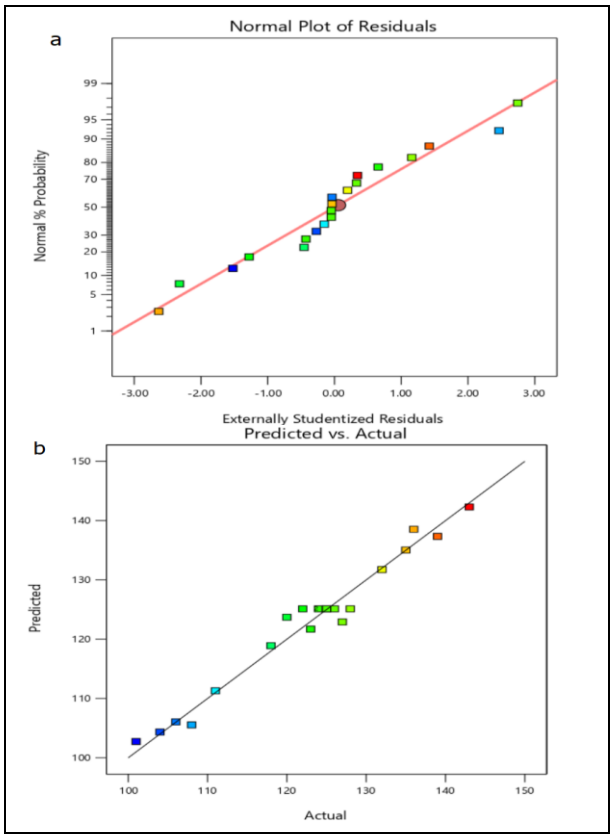

Fig. 3: (a) Normal probability plot of residuals for flexural strength (b) Comparison between predicted and actual values of flexural strength.

Table 3. ANOVA for flexural strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2572.42	9	285.82	39.62	< 0.0001	*
A-TiO2	2402.50	1	2402.50	333.06	< 0.0001	
B-Silane	1.60	1	1.60	0.2218	0.6478	
C-Time	19.60	1	19.60	2.72	0.1303	
AB	0.1250	1	0.1250	0.0173	0.8979	
AC	6.13	1	6.13	0.8491	0.3785	
BC	10.13	1	10.13	1.40	0.2635	
A^2	7.78	1	7.78	1.08	0.3235	
B^2	9.09	1	9.09	1.26	0.2878	
C^2	63.84	1	63.84	8.85	0.0139	
Residual	72.13	10	7.21			
Lack of Fit	52.13	5	10.43	2.61	0.1582	**
Pure Error	20.00	5	4.00			

* significant ** not significant

3. RESULTS AND DISCUSSION

3.1 Assessment of the Residual Plot for Flexural Strength

The residual points identified suggest the existence of a potential curve that closely aligns with the pattern observed in the mathematical model data. There are no apparent issues with the normality of the residual values (Fig. 3a). The graph in Fig. 3b displays the real and anticipated values for flexural strength. The actual values represent the data obtained from experiments, while the projected values are generated using the RSM in grouping with the CCD model. Both data sets exhibit a significantly high degree of proximity, underscoring the suitability of the suggested model. The R^2 values of 0.9727 for flexural strength indicate a strong correlation between the actual and expected responses.

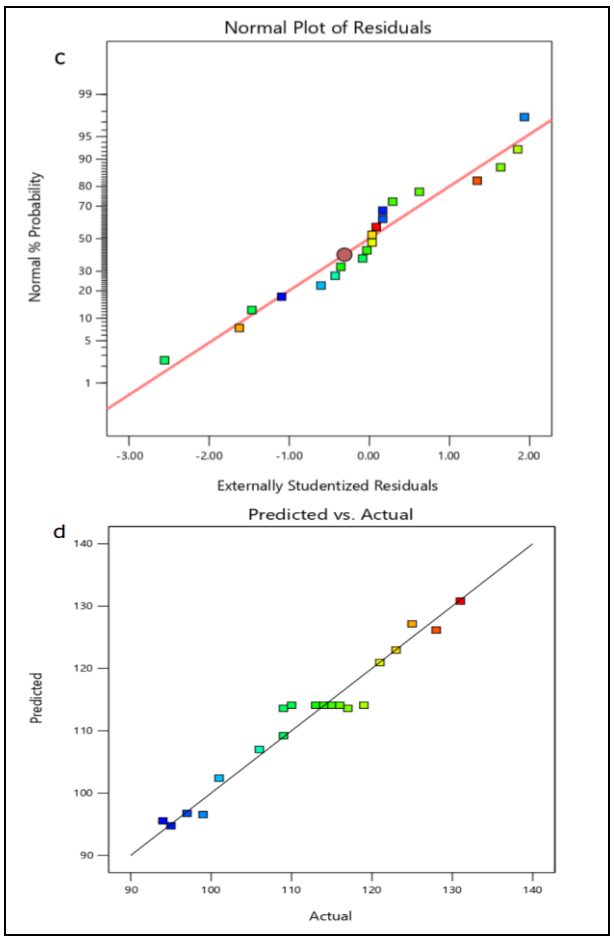

Fig. 3: (c) Normal plot of residuals for hardness (d) Predicted vs actual response for hardness

The residual data points detected indicate the presence of a dependable curve that closely corresponds to the model data. Fig. 3c shows that there are no significant abnormalities in the normality of residual values. In Fig. 3d, the actual values correspond to experimental response data, whereas the predicted values are obtained using response surface methodology. Both sets of data show a significant strong level of conformity, emphasizing the success of the suggested model. The high R^2 value of 0.9566 for hardness demonstrates a strong correlation between the actual and expected responses, highlighting the model's reliability.

3.2 Development of a Model using Mathematical and Independent Factors for Flexural Strength

Flexural strength = 125.127 + 15.5 * A + 0.4 * B + 1.4 * C + 0.125 * AB + 0.875 * AC + -1.125 * BC + 1.68182 * A^2 + -1.81818 * B^2 + -4.81818 * C^2 ……. (1)

Table 3 presents the effective p and f values, confirming the reliability of the ANOVA-analyzed proposed model (f=39.621, p<0.0001). These values are crucial for evaluating the validity of linear terms (A, B, C), interaction terms (AB, BC, AC), and quadratic terms (A^2, B^2, C^2) within the model. Notably, significant p-values for A, and C^2 are <0.0001, and <0.0139, respectively. The difference between R^2 predicted flexural strength and R^2 adjusted flexural strength is within 0.2, affirming the suitability of the model. Equation 1 substantiates these findings through the mathematical representation. Furthermore, three-dimensional (3D) plots visually illustrate the relationship between flexural strength and various process factors (Fig. 5). These plots depict similar trends, showing how flexural strength varies with different combinations of process factors such as silane-modified jute and kenaf, and silane immersion time. Specifically, Fig. 4a and 4b highlight the influence of silane time on silane % and TiO_2 %. The increase in nano-filler content in the hybrid nanocomposite positively correlates with flexural strength, as depicted in the graphs. Additionally, the material's strength is notably affected by the silane dipping percentage. Particularly, when the material undergoes a 20-minute immersion with silane, 10% silane and 5% nano-filler of TiO_2, there is a noticeable enhancement in the material properties.

Fig. 4c and 4d demonstrate the impact of the percentage of silane during immersion on the flexural strength, considering both the nano-filler content and silane duration. The time of silane treatment significantly affects strength metrics, particularly when using a 10% silane concentration. The use of TiO_2 nano-filler enhances the mechanical properties, specifically increasing the flexural strength. Fig. 4e and 4f illustrate the impact of TiO_2 on the proportion of silane and the duration o f silane immersion. Furthermore, they highlight the significance of integrating TiO_2 nano-filler to improve strength, particularly when combined with silane treatment. Additionally, the silane treatment duration proves to be a key component that affects the properties of the material. These results provide valuable information on the intricate relationship among TiO_2, silane content, silane treatment period, and the final material strength.

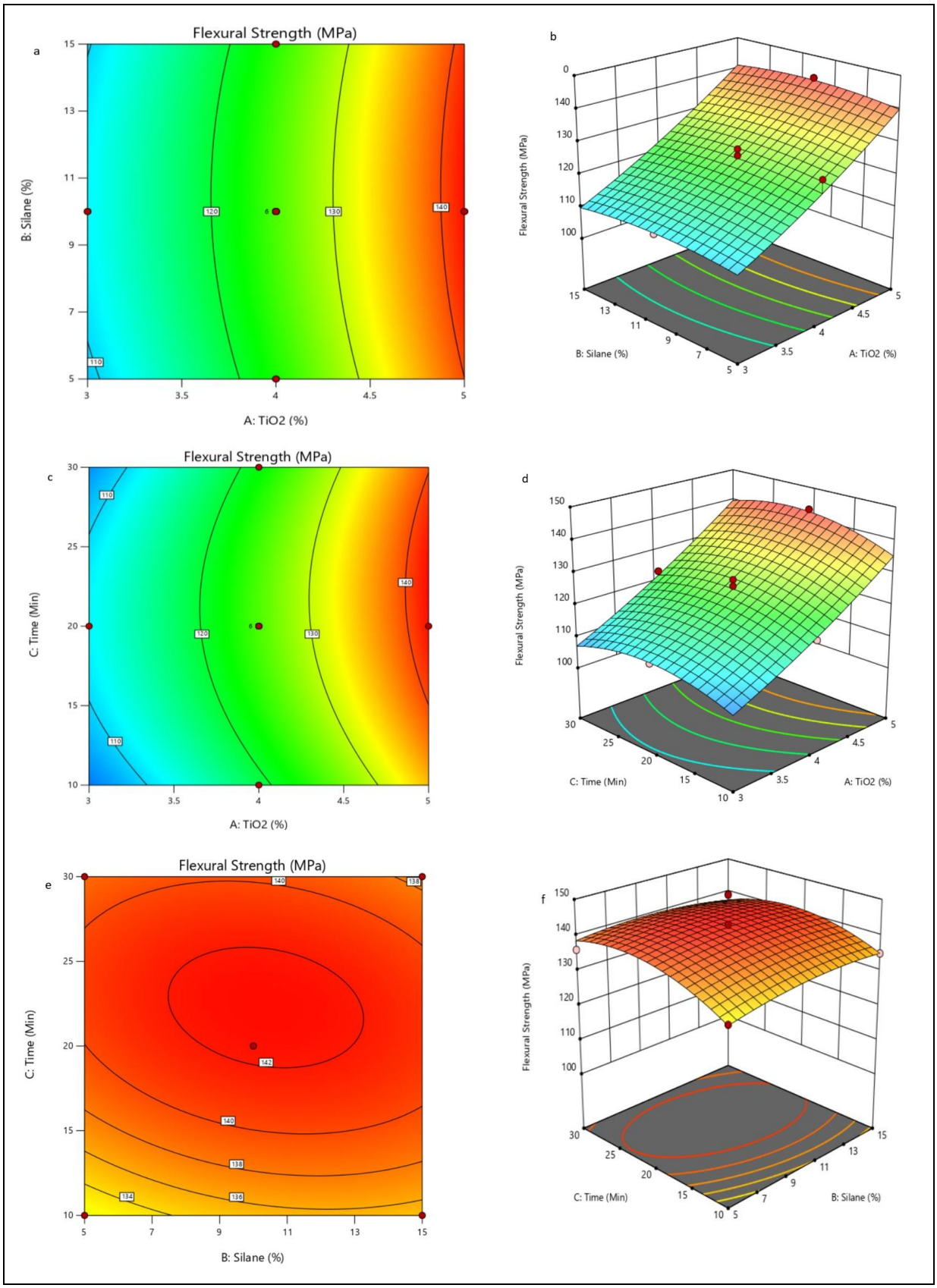

Fig. 4: 2D & 3D Surface plot for flexural strength (a, b) A-B interaction, (c, d) A-C interaction, (e, f) B-C interaction

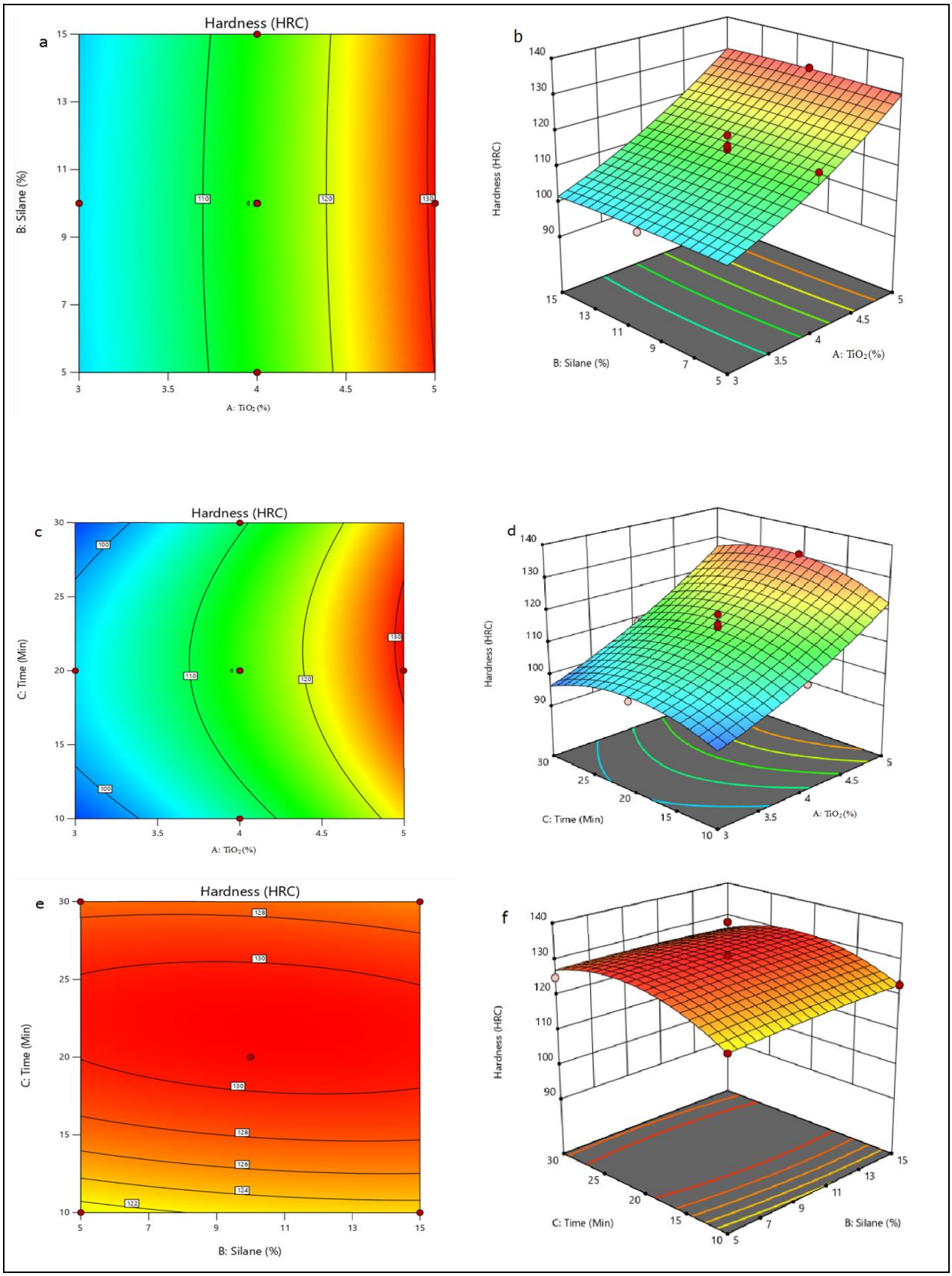

Fig. 5: 2D & 3D Surface plot for hardness (a,b) A-B interaction, (c, d) A-C interaction, (e, f) B-C interaction

3.3 Development of a Model using Mathematical and Independent Factors for Hardness

Hardness = 114.1 + 14.2 * A + 4.08137e-15 * B + 1.1 * C + 0.25 * AB + 1.25 * AC + -0.75 * BC + 2.5 * A^2 + -0.5 * B^2 + -6 * C^2

Table 4. ANOVA for hardness

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	2179.00	9	242.11	24.51	< 0.0001
A-TiO_2	2016.40	1	2016.40	204.09	< 0.0001
B-Silane	0.0000	1	0.0000	0.0000	1.0000
C-Time	12.10	1	12.10	1.22	0.2944
AB	0.5000	1	0.5000	0.0506	0.8265
AC	12.50	1	12.50	1.27	0.2869
BC	4.50	1	4.50	0.4555	0.5151
A^2	17.19	1	17.19	1.74	0.2166
B^2	0.6875	1	0.6875	0.0696	0.7973
C^2	99.00	1	99.00	10.02	0.0101
Residual	98.80	10	9.88		
Lack of Fit	53.30	5	10.66	1.17	0.4332
Pure Error	45.50	5	9.10		
Cor Total	2277.80	19			

** significant ** not significant

Table 4 shows the F value of 24.51, combined with an extremely significant p-value < 0.0001, providing strong evidence supporting the suitability of the CCD-RSM model in predicting hardness. All the process variables (A and C^2) show a p-value below 0.05, which suggests that they are statistically significant. In addition, the significant p-values for each individual variable, along with a small difference between R^2 predicted and R^2 adjusted (within 0.2), provide strong evidence of the significant impact of process variables on the hardness, as indicated by Equation 2. Fig. 5 showcases visual representations of impact strength using 3D response and contour plots. These visuals, featuring a three-dimensional surface map and a two-dimensional contour plot, show the impacts of various arrangements. There was a significant rise in the interaction pattern observed between silane treatment and TiO_2 nano-filler, as shown in Fig. 5a and 5b.

Fig. 5c and 5d investigate comparable patterns in relation to changes in the duration of silane treatment and the concentration of nano-filler. Meanwhile, Fig. 5e and 5f illustrate the impact of the percentage of TiO_2 on the percentage of silane and the duration of silane treatment, indicating that the chemical treatment improves the strength of the nanoparticles. The outcome of this research strongly supports the usage of biodegradable silane-treated jute and kenaf in different industrial sectors. After conducting a comprehensive analysis of various factors, it is clear that the mechanical properties are greatly improved by the application of silane treatment. Of particular interest is the specific configuration that includes a 10% silane treatment for 20 minutes and a 5% nano-filler concentration. This particular configuration has shown superior performance compared to other factors analysed using response surface methodology.

3.4 Conformation through Ramp Numerical Optimization

After conducting a thorough examination of different factors that affect the flexural strength and hardness in the laminate, we have managed to pinpoint the ideal range of parameters that are essential for maximizing these mechanical properties. Based on our findings, we discovered that there is an optimal range for maximizing flexural strength, which is between 101 and 143 MPa. Similarly, for hardness, the ideal range falls between 94 to 131 KJ/M^2, as indicated in Table 2. Fig. 6 provides a visual representation of the results obtained from the optimisation process, illustrating the specific operational conditions necessary to achieve optimal levels of both flexural strength and hardness. After conducting extensive experimentation, we have determined the most effective conditions for this process. These conditions include using a 5% concentration of TiO_2 nano-filler and a silane treatment comprising 10%. It is crucial to immerse the material in silane for 20 minutes, following the prescribed sequence G/J/J/K/K/K/K/J/J/G. The complex process of optimisation is illustrated in Fig. 7, showcasing the meticulous journey taken to determine the most favourable parameters for improving flexural strength and hardness. The parameters were meticulously chosen after thorough analysis to guarantee optimal performance in both flexural strength and hardness. To conform the credibility of our outcome, we demonstrated thorough impact studies using the most optimal conditions. The findings of these investigations clearly validate the effectiveness of our model, suggesting a striking correspondence between the anticipated outcomes and the real experimental results.

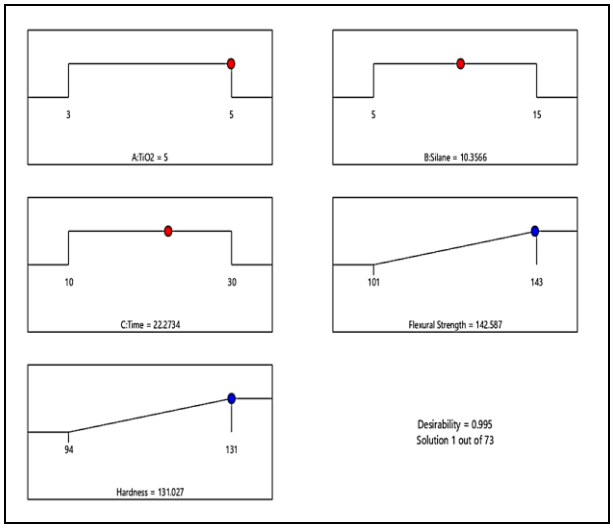

Fig. 6: Ramp numerical optimisation for silane treatment %, silane treatment time and TiO_2 filler

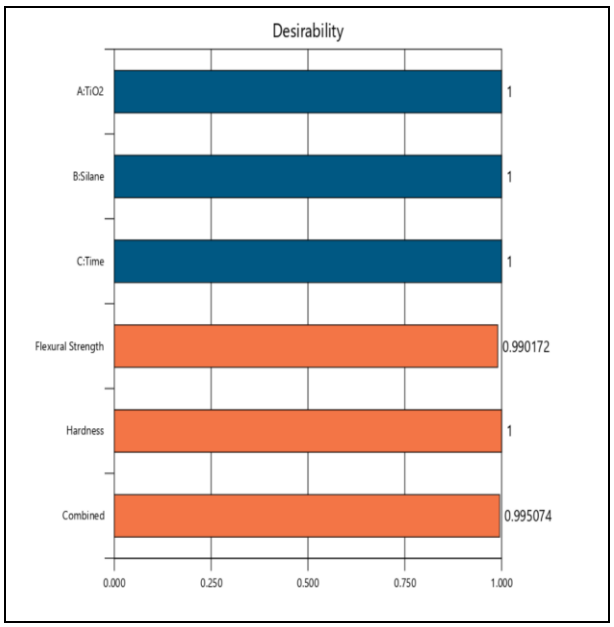

Fig. 7: Desirability plots for individual and combined value of the model

4. CONCLUSION

The research focused on the effects of various levels of nano-filler and silane, along with varying treatment duration on the flexural strength and hardness of composites that were reinforced with jute and kenaf fibres. A thorough investigation was conducted on the preparation parameters of the laminate using CCD within RSM. The study found that the inclusion of treated kenaf and jute fibres, in addition to glass fiber, resulted in a notable enhancement in both flexural strength and hardness. This indicates the successful implementation of a combined reinforcement approach. Furthermore, significant improvements in both flexural strength and hardness were noted when comparing the treated chemicals to the untreated ones, with a 19% increase in flexural strength and a 14% increase in hardness. This highlights the beneficial impact of the silane treatment on the mechanical properties of the composite. In addition, the treatment with silane had a significant impact on the behaviour of the fibres, particularly in the instance of jute. Higher quantity of silane resulted in improved load-bearing capabilities, which were further enhanced by the incorporation of TiO_2 nano-filler, demonstrating its ability to bridge gaps. The RSM approach effectively simplifies the complexity of the problem, providing valuable insights into the intricate relationships within the experimental setup. Ultimately, the study discovered the ideal process conditions for achieving the highest flexural and hardness properties. It emphasized that a combination of 5% TiO_2 filler, 10% silane concentration, and a 20-minute silane treatment duration yielded the best results. It is worth mentioning that the duration of silane treatment applied had a significant impact on the mechanical characteristics of the composite material.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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