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EFFECT OF CITRONELLA FIBER TREATMENT ON TENSILE STRENGTH OF POLYESTER MATRIX COMPOSITES: A 2K FACTORIAL DESIGN ANALYSIS

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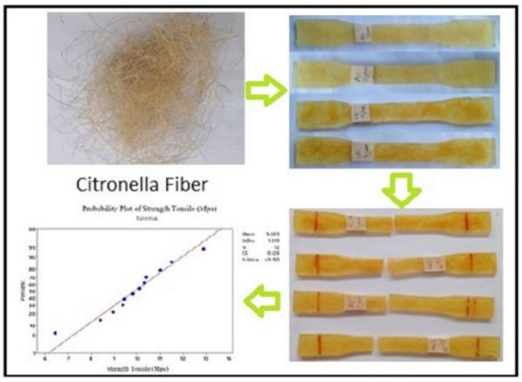
Graphical abstract



Abstract

Using natural fiber-reinforced composites has become a new style in materials technology. The natural fibers used have a significant impact on the mechanical properties of the composite, so the use of natural fibers in composites has the potential to be more sustainable in various industrial fields. However, natural fibers contain several chemical components that can affect the mechanical properties of the composite. Therefore, chemical treatment is needed on the fibers that will be used as reinforcement so that the composite made can produce mechanical properties such as optimal tensile strength. This research aims to see the effect of the type of solution and length of treatment time on citronella fiber on the tensile strength of the resulting composite. The method used in this research is a 2k factorial design. In this research, the tensile testing process used the ASTM D638-01 standard reference, with citronella fiber being soaked using a liquid smoke solution and boiling the fiber using a turmeric solution. The duration of the treatment process for both was 1 hour and 3 hours. The research showed that the highest tensile strength produced was 10.93 MPa when the fiber was soaked in liquid smoke for 1 hour. In contrast, the fiber treatment and length of time for the composite fiber reinforcement treatment did not affect the resulting tensile strength

Keywords: 2^k factorial design; composites; tensile strength; natural fibers; mechanical properties of *composites*

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1. Introduction

Technology in composite materials, particularly composite reinforcements, has developed rapidly in recent decades. Theoretically, composites are materials formed by combining two or more materials to achieve a final material with mechanical properties superior to those of its constituent materials [1], [2]. The advantages of using composites include corrosion resistance, lightweight characteristics, attractive performance, manufacturing without machining processes, and suitability to several applications [3], [4]. Several types of composites are focused on their use as structural materials and the selection of reinforcing fiber composition plays a significant role in determining the properties of the composite. Given the abundance of natural fibers, innovation in composite reinforcements often utilizes them due to their eco-friendly nature, low cost, and potential for widespread application [5]. In addition to being easily accessible, another advantage of using natural fibers as reinforcements is that they usually do not require extensive processing and are biodegradable [6]. This has driven continuous research into natural fibers. Industries also utilize natural fibers based on parameters such as strength and stiffness that meet industry standards, thermal stability, fiber-matrix bonding, dynamic behavior, long-term performance, cost, processing expenses, and availability [7].

Citronella, scientifically known as "Cymbopogon nardus," is a plant that contains natural fibers and is widely cultivated in Indonesia. Citronella contains 0.5-1.5% essential oil, with the remainder consisting of solid waste (residue of raw materials) and water from the distillation process [8]. The study by Afenanda et al. found that the fiber content in citronella is around 25.73%, with lignin content at 27.38%. Meanwhile, the cellulose content in citronella reaches 35.0% [9]. The stalk of the citronella plant can be utilized as composite reinforcement. A composite will have good physical properties or strength if it contains little lignin, as lignin is rigid and brittle [10]. Therefore, to achieve high tensile strength in composites, the fibers used as reinforcement require chemical treatment to significantly improve the mechanical properties of the fiber-reinforced composites, such as tensile and flexural strength (strength and modulus) [11]. Currently, the use of citronella fiber as composite reinforcement is still limited. In a previous study conducted by Afenanda et al., the effect of NaOH fiber soaking treatment on the tensile strength of citronella stalk fiber composites with an epoxy matrix was investigated. The study used NaOH solution concentrations of 3%, 5%, and 7%, and the highest tensile strength was achieved with a 7% NaOH soak, resulting in 50.30 MPa [9]. Many studies have employed essential compounds such as NaOH, $KMnO_4$, H_2O_2, and KOH for fiber treatment, commonly called alkalization treatment.

Research on innovative treatments for composite materials reinforced with natural fibers has been conducted. For example, a study by Mukhlis et al. investigated the effect of soaking coconut coir fibers in liquid smoke for 1, 2, and 3 hours without treatment. The highest tensile strength was obtained with a 1-hour soaking time, reaching 79.655 MPa [12]. Additionally, there is research on the effect of boiling treatment using turmeric solution for 1, 2, and 3 hours on the interfacial shear strength and wettability of single Akaa fibers in an epoxy matrix. This study found that the highest shear strength of 29.48 N/mm^2 was achieved in fibers treated with 1 hour of boiling [13].Based on previous research, it is known that the treatment process applied to fibers used for composite reinforcement affects the composite's mechanical properties, including the composite material's tensile strength. Furthermore, using citronella plant fibers as composite reinforcement still needs to be improved. Therefore, this study will investigate citronella fiber-reinforced composites by applying chemical treatments to citronella fibers using natural compounds, namely coconut shell liquid smoke and turmeric solution, to examine the effects of these treatments on the tensile strength of the resulting composite.

The research will involve soaking the citronella fibers in liquid smoke and boiling the fibers in turmeric solution, using citronella fiber-reinforced composites with a Polyester Yukalac BQTN-157 matrix. By investigating these under-researched combinations (natural treatments and citronella fibers), this study offers novel insights into improving the mechanical properties of eco-friendly composite materials.

The novelty of this research lies in several key aspects related to the treatment of citronella fiber and its use as reinforcement in composite materials:

- Use of Citronella Fiber as Reinforcement: Citronella fiber is relatively underexplored in the context of composite reinforcement. This study contributes to understanding the mechanical properties of composites reinforced with citronella fiber, particularly its tensile strength, which could be a new application of this natural fiber.
- Chemical Treatment with Liquid Smoke and Turmeric Solutions: The research introduces the novel approach of using liquid smoke and turmeric solutions as chemical treatments for natural fiber reinforcement. While chemical treatments of natural fibers are common, using these specific substances is innovative and contributes new insights into their effects on fiber properties and composite strength.
- Comparison of Treatment Durations: The study explores the impact of treatment duration (1 hour and 3 hours) on the mechanical properties of the composite. This focus on both the type of solution and the length of treatment provides a deeper understanding of how these factors interact, adding a new dimension to the optimization of fiber-reinforced composites.

- Factorial Design Method: Employing the 2k factorial design method to study the effects of two factors (type of solution and treatment time) on tensile strength adds a structured experimental approach. The combination of these variables and their effects on mechanical performance has not been widely studied in the context of citronella fibers.

In summary, the novelty lies in the use of citronella fiber, the specific chemical treatments (liquid smoke and turmeric), and the factorial design that evaluates their combined effects on tensile strength, contributing new knowledge to natural fiber-reinforced composite research.

2. Material and Method

The composite was molded using citronella fiber as reinforcement in the experiment. The fibers underwent a chemical treatment process prior to being molded into composites. The chemical treatment involved soaking the fibers in coconut shell liquid smoke for 1 hour and 3 hours and boiling the fibers in a turmeric solution for 1 hour and 3 hours. These treatments aimed to enhance the mechanical properties of the citronella fiber, allowing it to better serve as a reinforcement material in the composite. After the chemical treatments, the fibers were incorporated into the composite, and the resulting material was then tested for its tensile strength and other mechanical properties.

2.1. Material

The supporting equipment used in this research includes an ISO 179-01 tensile test silicone mold, small acrylic boards, bricks, containers for mixing the matrix, measuring cups, calipers, and a Universal Testing Machine, Zwick Roell Model Z20 Xforce K, for tensile testing. The materials employed in this study are citronella fibers, Polyester Yukalac BQTN-157 resin, catalyst, coconut shell liquid smoke, and turmeric solution. The citronella fiber serves as the reinforcement material in the composite, while the Polyester Yukalac BQTN-157 resin acts as the matrix to bind the fibers. The catalyst is used to initiate the curing process of the resin, ensuring proper bonding between the fiber and the matrix. The coconut shell liquid smoke and turmeric solution are natural chemical treatments for the fibers. The citronella fiber used in this study is depicted in Figure 1 below. These materials and equipment are essential to ensure accurate results when testing the mechanical properties of the resulting composite.

Figure 1. Citronella Fiber

- Physical and mechanical properties

Citronella fiber, extracted from the Citronella grass (Cymbopogon nardus or Cymbopogon winterianus), possesses several physical and mechanical properties that make it a candidate for composite reinforcement. However, detailed research specifically on citronella fiber is limited, and its properties might vary depending on the plant's growth conditions and processing methods. Here are the typical physical and mechanical properties based on general studies of natural fibers and those related to citronella:

Physical Properties:

1. Density: Citronella fiber typically has a density of around 1.2–1.3 g/cm³, which is comparable to other natural fibers like jute and hemp.
2. Fiber Diameter: The diameter of citronella fibers ranges from 50 to 200 µm depending on the extraction and processing methods.
3. Moisture Absorption: Citronella fibers, like most natural fibers, have a relatively high moisture absorption rate, which can affect their bonding with resin in composites. This value is typically around 8-12%.

4. Cellulose Content: The fiber contains a relatively high percentage of cellulose (around 60-70%), contributing to its tensile strength. Other components include hemicellulose (10-20%) and lignin (10-15%).

- Mechanical Properties:

1. Tensile Strength: The tensile strength of untreated citronella fibers is generally reported to be around 100-200 MPa, depending on the extraction method, maturity of the plant, and any pre-treatment applied.
2. Young's Modulus: Citronella fibers have a modulus of elasticity of approximately 3-5 GPa, indicating their stiffness and resistance to deformation.
3. Elongation at Break: The elongation at break for citronella fibers is typically around 1-3%, which reflects the fiber’s ability to stretch before breaking, and is on the lower side compared to synthetic fibers.
4. Impact Resistance: Citronella fibers tend to have moderate impact resistance, but this is highly dependent on the treatment and the matrix they are combined with in composites.

- Comparison with Other Natural Fibers:

1. Strength: Citronella fibers are relatively weaker than other natural fibers such as flax or hemp, but treatments such as the use of natural chemicals (e.g., liquid smoke or turmeric) could enhance their tensile properties.
2. Stiffness: The Young’s modulus is in the mid-range compared to natural fibers like jute (which can have a modulus up to 30 GPa) but is still sufficient for certain composite applications, especially in lightweight structures. Overall, citronella fibers are promising due to their moderate mechanical properties and biodegradability. However, mechanical performance, especially tensile strength, can be significantly improved through chemical or physical treatments, which is a major focus of ongoing research.

2.2 Method

Citronella is used as a natural fiber reinforcement material in composites. In this study, chemical treatments on citronella fibers were performed using two types of solutions: liquid smoke and turmeric solution. The fiber treatment with liquid smoke was carried out by soaking the citronella fibers in pure liquid smoke solution for 1 hour and 3 hours. Soaking the fibers in liquid smoke can alter the natural texture of the fibers and modify their mechanical properties due to the carbonyl and acid compounds present in the liquid smoke solution [14]. This treatment enhances the quality of the fibers, making them more suitable for use in composites. For the fiber treatment using turmeric solution, the fibers were boiled in the solution for 1 hour and 3 hours. The turmeric solution used was a mixture of distilled water and pure turmeric powder at 4:1. Boiling the fibers in turmeric solution can increase the cellulose content and reduce a portion of the lignin in the fibers. This occurs because the boiling process generates cellulose acid compounds from the turmeric solution. As a result, this treatment influences the mechanical properties of the fibers, particularly their tensile strength [13].

- Sample

In this study, the preparation of tensile test specimens refers to the ASTM D638-01 testing standard. Figure 2 shows the dimensions of the tensile test specimen based on ASTM D638 Type 1 [15]. This standard specifies the shape and dimensions required for accurate and reliable tensile testing, ensuring consistency in evaluating the mechanical properties of the composite material. The use of ASTM D638 Type 1 provides a benchmark for comparing the tensile strength and elasticity of the citronella fiber-reinforced composites produced in this research.

The process for fabricating tensile test specimens of citronella fiber-reinforced composites using the hand lay-up method with YUKALAC BQTN-157 polyester resin [16] is as follows:

1. Ensure the citronella fibers are clean and have undergone treatment: soaking in coconut shell liquid smoke and boiling in turmeric solution for 1 hour and 3 hours.
2. Clean the mold thoroughly before use to prevent contamination from affecting the specimens.
3. Arrange the fibers along the direction of the mold. Lay them systematically to match the volume and standard dimensions required for tensile testing.
4. Mix YUKALAC BQTN-157 polyester resin with the catalyst to accelerate the curing process.

5. Pour the resin matrix into the mold according to the required amount. Use a stick to smooth out and evenly distribute the fibers within the matrix.
6. Cover the mold with acrylic sheets and press down with bricks to ensure the matrix spreads uniformly into the fibers.
7. Allow the specimen to dry for 15-30 minutes. Once the resin has cured, carefully remove the specimen from the mold.
8. The composite is now ready for tensile testing.

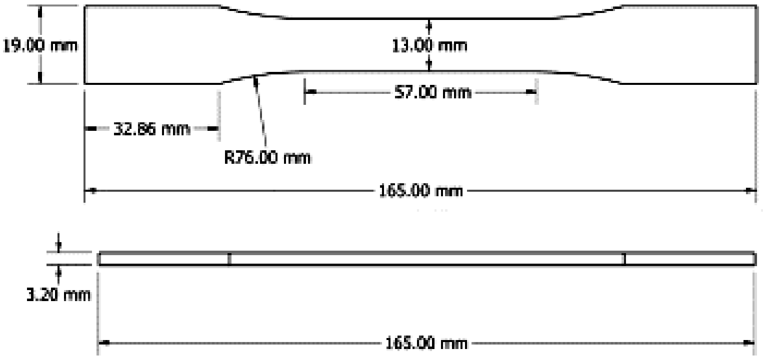


Figure 2. Tensile test dimension (ASTM D638 TYPE 1)

- Sample test

In this study, tensile testing of the composites was conducted using the Universal Testing Machine, Zwick Roell Model Z20 Xforce K. The testing procedure is as follows:

1 Prepare all specimens and equipment required for the tensile testing process.
2 Calibrate the tensile testing machine according to the manufacturer's instructions to ensure accurate measurements.
3 Place the specimens securely on the tensile testing machine.
4 Ensure that the testing machine properly clamps and holds the specimens in place.
5 Adjust the control panel to set the desired testing speed.
6 Monitor the results displayed on the control panel's monitor throughout the test.

- Factorial 2^k Design Method

The 2^k factorial design is a basic design that includes k factors, each with two levels [16]. This study used a 2^2 factorial design because each factor has two levels. In this factorial design, the levels are categorized as "low," represented by the symbol (-), and "high," represented by the symbol (+) [17].

Table 1. The 2^k factorial design

Treatment	A	B
(1)	-	-
A	+	-
B	-	+
Ab	+	+

The design involves combinations of treatments between the levels of the two factors, with each combination being tested a total of n times. The details of these treatment combinations and the experimental setup are shown in Table 1.

- Analisis of varian (ANOVA)

Analysis of Variance (ANOVA) is an advanced form of two-way ANOVA. The primary difference is the interaction effect between the two independent variables. This ANOVA method calculates the total sum of squares to assess the variability within and between groups. The general formula for the sum of squares in ANOVA can be expressed as follows [15]. The sums of squares for factors A, B, the interaction between A and B, total, and error can be calculated using the following equations:

$$SS_A = \frac{\left(ab+a-b-(1)\right)^2}{n.4} \quad (1)$$

$$SS_B = \frac{\left(ab+b-a-(1)\right)^2}{n.4} \tag{2}$$

$$SS_{AB} = \frac{\left([ab+(1)-a-b]^2\right)}{n.4} \tag{3}$$

$$SS_T = \sum_{i=1}^{2} \sum_{j=1}^{2} \sum_{k=1}^{n} y^2 ijk - \frac{y^2}{4n} \tag{4}$$

$$SS_E = SS_T - SS_A - SS_B - SS_{AB} \tag{5}$$

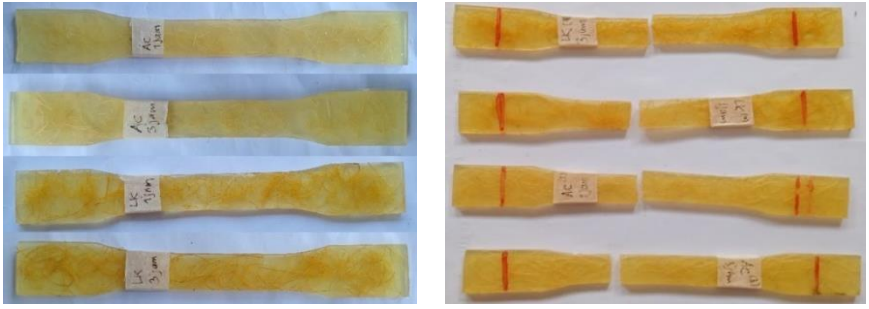
The ANOVA table for a factorial design summarizes the sources of variation, degrees of freedom (DF), sum of squares (SS), mean squares (MS), and F-values. For a 2^2 factorial design, the ANOVA table is structured as follows:

Table 2. ANOVA 2^2 factorial design

Source of Variation	SS	DF	MS	F_{value}
A	SS_A	1	$MS_A = \frac{SS_A}{1}$	$\frac{MS_A}{MS_E}$
B	SS_B	1	$MS_B = \frac{SS_B}{1}$	$\frac{MS_B}{MS_E}$
AB	SS_{AB}	1	$MS_{AB} = \frac{SS_{AB}}{1}$	$\frac{MS_{AB}}{MS_E}$
Error	SS_E	$ab(n-1)$	$MS_E = \frac{SS_E}{ab(n-1)}$	
Total	SS_T	$N-1$		

3. Result and Discussion

The tensile test sample, molded according to ASTM D638 Type 1 standards, has specific shapes and dimensions designated for testing the mechanical properties of materials. This standard ensures the accuracy and consistency of tensile test results. After the molding process is complete, the sample is tested to determine the material's tensile strength, and the test results can be seen in Figure 3, showing the sample's condition after testing.



(a) (b)

Figure 3. Tensile test sample (a) Before testing, (b) Sample's condition after testing

- Tensile test data

After conducting a tensile test using the Universal Testing Machine, Zwick Roell Model Z20 Xforce K, the tensile strength results were obtained in Megapascals (MPa). This machine operates by gradually applying a load until the sample reaches its breaking point. The test results provide information about the maximum strength a material can withstand before failure. The number of samples (or replications) for each treatment depends on the desired accuracy and the variation present in the system being studied. While 3 samples per treatment may be sufficient in some cases, it may not always provide the most reliable results, especially if there is significant variability. Table 2 shows the tensile strength test results, which are used for further analysis of the material's mechanical properties.

Based on the tensile test data, it is evident that the immersion treatment in liquid smoke solution significantly affects the material's tensile strength. The highest value, 10.93 MPa, was achieved in the sample soaked for 1 hour, indicating that a shorter treatment time results in more optimal strength. Conversely, a longer immersion time of 3 hours produced the lowest tensile strength value of 8.96 MPa. This decrease in strength may be due to prolonged exposure to the solution, which could lead to changes in the material's structure, such as reduced intermolecular strength or material degradation. These results provide important insights into determining the optimal immersion duration to achieve maximum material strength.

Table 2. Tensile strength test results

No	Time (hours)	Fiber Treatment	Tensile Strength (Mpa)			Average (Mpa)
			1	2	3	
1	1	Soaking with Liquid Smoke	12.90	11.50	8.40	10.93
2	1	Boiling with turmeric solution	9.37	10.30	10.10	9.92
3	3	Soaking with Liquid Smoke	6.44	11.00	9.44	8.96
4	3	Boiling with turmeric solution	9.80	10.40	8.95	9.72

- Normality test

The normality test was conducted after obtaining the tensile strength response data to assess the normality of the generated data. This process was performed using Minitab software. The results of the normality test for the tensile strength response data can be seen in Figure 5. Conducting this test is essential to ensure that the data follows a normal distribution, which is a fundamental assumption for further statistical analysis and validation of the experimental results. The outcome of this test provides insights into whether the tensile strength data can be analyzed using parametric statistical methods.

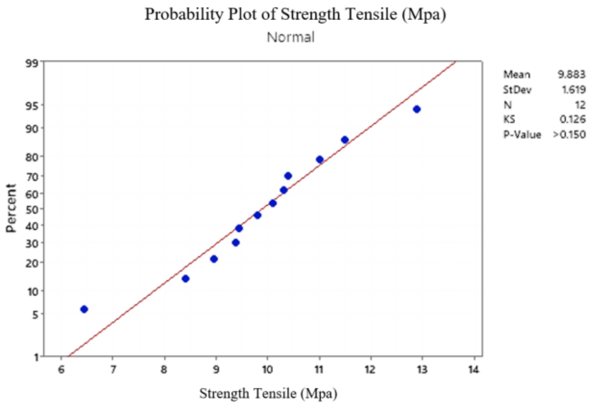


Figure 4. *Probability Plot of* Strength Tensile (MPa)

Based on Figure 4, the data shows no significant difference from the standard normal distribution. This distribution is indicated by the Kolmogorov-Smirnov (KS) value of 12.6%, higher than the significance level α = 5%. Since the KS value is greater than the significance level, the data meets the criteria for being normally distributed. This normality test is essential because the validity of statistical analysis depends on the assumption that the data follows a normal distribution. With this result, further analysis, such as parametric statistical analysis, can be conducted based on the assumption that the tensile strength response data complies with normality rules. In conclusion, the data is eligible for further analysis.

- Analysis of Variance

The analysis of variance (ANOVA) is conducted by calculating the sum of squares from the experimental data based on Table 2. The sum of squares calculation includes several important components using equations (1) through (5). The calculated components include the sum of squares for factor A, the sum of squares for factor B, the sum of squares for the interaction between factors A and B, the sum for error, and the total sum of squares. After all components of the sum of squares are calculated, the results are entered into the ANOVA table. This ANOVA table is used to identify the significance of the effects of each factor and their interactions on the response variable, as well as to determine the proportion of data variability explained by the model.

$$\mathrm{SS_A} = \frac{(29.15 + 26.88 - 29.77 - 32.8)^2}{3.4} = 3.5643$$

$$\mathrm{SS_B} = \frac{(29.15 + 29.77 - 26.88 - 32.8)^2}{3.4} = 0.04813$$

$$\mathrm{SS_{AB}} = \frac{(29.15 + 32.8 - 26.88 - 29.77)^2}{3.4} = 2.34083$$

$$\mathrm{SS_T} = (12.9^2 + 11.5^2 + 8.4^2 + \cdots + 8.95^2) - \frac{(32.8 + \cdots + 29.15)^2}{4(3)} = 28.8433$$

$$\mathrm{SS_E} = 28.8433 - 3.5643 - 0.04813 - 2.34083 = 22.89$$

Based on the sum of squares calculations, the resulting values are entered into the ANOVA table for further analysis. This ANOVA table summarizes the components of the sum of squares, including the sum of squares for factors A and B, the interaction between factors A and B, and the error. Each sum of squares value is then used to calculate the degrees of freedom, mean square, and F-value. Using the F-value, we can determine whether the effects of each factor and their interaction are significant on the response variable. This process helps in understanding the extent to which the variability in the data can be explained by the model used.

Table 4. Analysis of Variance

Source of Variation	SS	Df	MS	F_{value}	F_{table}
A	3.5643	1	3.5643	1.24571	5.317655
B	0.04813	1	0.04813	0.01682	5.317655
AB	2.34083	1	2.34083	0.81812	5.317655
Error	22.89	8	2.86125		
Total	28.8433	11			

Based on Table 4, the decision and conclusions from the analysis of variance (ANOVA) are as follows:

Hypotheses:

1. For the variable treatment duration:
   - H_0: Treatment duration does not affect the tensile strength value.
   - H_1: Treatment duration affects the tensile strength value.

2. For the variable fiber treatment:
   - H_0: Fiber treatment does not affect the tensile strength value.
   - H_1: Fiber treatment affects the tensile strength value.

Decision:

- If F_{value} > Ft$_{able}$, then the null hypothesis (H_0) is rejected, meaning the variable has a significant effect on the tensile strength.
- If F_{value} < F_{table}, then the null hypothesis (H_0) fails to be rejected, meaning the variable does not have a significant effect on the tensile strength.Significance Level: The significance level used is 5% or 0.05.

Based on Table 4, the factorial design ANOVA results show that the calculated F-value for treatment duration is smaller than the F-table value. The calculated F-value for treatment duration is 1.24571, while the F-table value is 5.317655. Thus, the decision is that H0 fails to be rejected, meaning that treatment duration does not significantly affect the composite's tensile strength value. Next, the calculated F-value for the fiber treatment is also smaller than the F-table value. The calculated F-value for fiber treatment is 0.01682, with the same F-table value of 5.317655. This result also indicates that the decision H0 fails to be rejected, meaning that fiber treatment does not significantly affect the tensile strength. Furthermore, the interaction between treatment duration and fiber treatment also shows a calculated F-value smaller than the F-table value. The calculated F-value for the interaction between these two factors is 0.81812, with an F-table value of 5.317655. Based on this result, the decision is again that H0 fails to be rejected, meaning the interaction between treatment duration and fiber treatment does not significantly affect the tensile strength of the composite. The conclusion from this ANOVA is that neither treatment duration nor fiber treatment nor their interaction significantly influences the tensile strength of the tested composite.

4. **Conclusion**

In this study, the tensile strength response data of the citronella fiber-reinforced composite showed a normal distribution. The highest tensile strength was 10.93 MPa, achieved with fiber treatment soaked in liquid smoke for 1 hour. On the other hand, the lowest tensile strength value of 8.96 MPa was recorded for fiber treatment soaked in liquid smoke for 3 hours. Based on the results of the Analysis of Variance (ANOVA) on the tensile strength data, it was found that both factors studied, namely the duration of the treatment and the fiber treatment itself, did not significantly affect the tensile strength of the resulting composite. This result indicates that variations in soaking time and fiber treatment with liquid smoke do not significantly change the tensile strength of the citronella fiber-reinforced composite. Thus, this treatment may not be effective in significantly improving the tensile strength of the composite, and other factors may need to be considered in optimizing this natural fiber-reinforced composite.

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