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THE EFFECT OF REINFORCEMENT SURFACE TREATMENT WITH CITRIC ACID ON THE FLEXURAL AND HARDNESS PROPERTIES OF EPOXY/WOOD SAWDUST COMPOSITES

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Abstract. This study investigates the effect of citric acid surface treatment on wood sawdust used as reinforcement in epoxy-based composites by evaluating both bending strength and Brinell hardness. Wood sawdust was subjected to surface modification using citric acid and sodium hydroxide (NaOH) under equivalent molar concentrations. Bending strength tests showed that both citric acid and NaOH treatments significantly improved mechanical performance compared to untreated samples, with average strengths of 33.83 MPa and 32.82 MPa, respectively, versus 21.83 MPa for the untreated group. Statistical analysis was conducted to compare the two treatments. After confirming normal distribution but unequal variances, a Welch two-sample t-test was performed, yielding a p-value of 0.742 showed no significant difference between the two treatments. Brinell hardness measurements produced mean values of 9.56 HB, 9.01 HB, and 9.77 HB for citric acid-treated, NaOH-treated, and untreated composites, respectively. One-way ANOVA indicated no significant variation in hardness among these groups. These results demonstrate that citric acid treatment enhances flexural performance to the same extent as NaOH without altering surface hardness.

Keywords : ANOVA, Bending strength, Citric Acid, Composite, Hardness, Wood Sawdust.

1. INTRODUCTION

The increasing demand for sustainable materials has driven the development of composite materials derived from renewable natural resources. One such material with promising potential is wood sawdust waste, a byproduct generated from wood processing industries [1], [2]. The use of wood sawdust as a composite constituent enables the production of materials that can be tailored to meet specific performance criteria. Moreover, wood-based composites offer an opportunity to enhance the utility of low-quality wood by converting it into higher-value products [3].

However, one of the primary challenges in utilizing wood sawdust in polymer composites lies in the inherent incompatibility between the hydrophobic epoxy resin and the hydrophilic nature of wood particles. This mismatch leads to poor interfacial adhesion, which negatively affects the mechanical performance of the composite. To address this issue, surface treatment of the wood sawdust is essential for improving the compatibility and adhesion between the filler and the matrix [4].

Alkaline treatment using sodium hydroxide (NaOH) has been widely adopted to modify the surface of lignocellulosic materials, aiming to enhance the adhesion between wood fibers and polymer matrices in wood-plastic composites [5], [6], [7]. During this treatment, hydrogen bonds within the lignocellulosic matrix are disrupted, increasing the proportion of amorphous cellulose and surface roughness. While effective, the use of NaOH poses significant environmental hazards, creating a need for more eco-friendly and cost-effective surface treatment alternatives [8].

Citric acid, a natural organic poly(carboxylic acid) with three carboxyl groups, has demonstrated potential as a cross-linking agent through esterification with hydroxyl groups, forming stable cross-linked networks [9]. Beyond its applications in the food, beverage, and pharmaceutical industries, citric acid has shown strong cross-linking capabilities with plant fibers, paper, wood, starch, and natural elastomers [8], [10], [11]. Although citric acid offers notable advantages in improving fiber adhesion, research on its use for surface treatment of wood sawdust in composite applications remains limited.

This study aims to investigate the effect of citric acid surface treatment on wood sawdust used in epoxy-based composites. The influence of this treatment will be evaluated through mechanical characterization, specifically flexural (bending) strength and Brinell hardness, and benchmarked against both untreated and NaOH-treated counterparts. Emphasis is placed on assessing whether citric acid can serve as a viable and environmentally friendly alternative to conventional alkali treatments.

2. METHODS

This research employed a laboratory-based experimental approach to evaluate the effect of citric acid surface treatment on the mechanical performance of epoxy composites reinforced with wood sawdust. The primary focus was to investigate bending strength using a three-point bending test and Brinell Hardness, as these properties are essential for evaluating the rigidity and surface resistance of composite materials. The composite matrix was composed of a commercially available clear epoxy resin and its corresponding hardener, mixed in a 2:1 weight ratio, ensuring a consistent and reliable polymer crosslinking process.

Wood sawdust, obtained as a byproduct from a local woodcraft production facility, served as the reinforcing filler. The raw sawdust was used without prior chemical treatment unless otherwise specified for comparison. Food-grade citric acid purchased from a local supermarket was used as the primary surface treatment agent without further purification due to its known ability to modify lignocellulosic surfaces. Sodium hydroxide (NaOH), a widely used alkaline modifier, was also used as a comparative treatment, prepared at the same molarity as the citric acid solution to ensure consistency in treatment conditions. Before treatment, wood sawdust was manually sieved using a 40-mesh screen to obtain uniform particle size distribution. The particles retained on the sieve were subjected to surface modification. These sawdust particles were immersed in either NaOH or citric acid solution, stirred continuously for 2 hours to enhance contact between the chemical agents and the sawdust surface, then rinsed thoroughly with distilled water and oven-dried at 60 °C to remove residual moisture. After drying, the treated sawdust was mixed into the epoxy resin at a weight ratio of 19%. The mixture was stirred manually until a homogeneous blend was achieved and then poured into pre-fabricated molds. The molds were left at room temperature for curing under ambient laboratory conditions. After curing, the composite plates were demolded and cut into test specimens with standardized dimensions of 100 mm × 16 mm × 5 mm (length × width × thickness), suitable for mechanical testing.

Three-point bending tests were conducted using a universal testing machine with an 80 mm support span in accordance with ASTM D790 standards. This test provided data on flexural strength and modulus. Brinell hardness was measured by applying a predetermined load to a 2.5 mm diameter steel ball indenter pressed into each specimen's surface. The resulting indentation was used to calculate hardness values based on ASTM E10 guidelines. The combination of these two tests enabled a comprehensive evaluation of the mechanical behavior of the developed composites under different surface treatment conditions.

3. RESULTS AND DISCUSSION

3.1 Effect of Surface Treatment on Flexural Bending Strength

The epoxy composites reinforced with NaOH and citric acid-treated wood sawdust exhibited substantially higher bending strength than the untreated control. Untreated specimens averaged is 21.83 MPa, whereas the NaOH-treated group averaged 32.82 MPa and the citric-acid-treated group 33.83 MPa. These values correspond to roughly 50–55% improvement over the untreated case as shown in Figure 1.

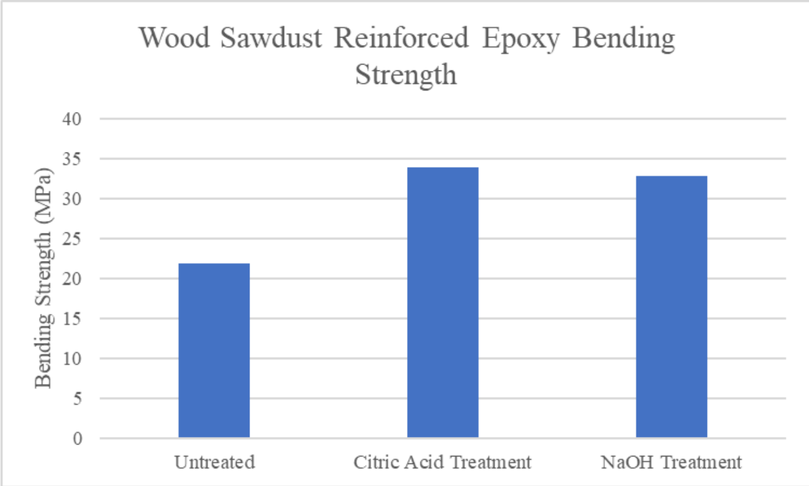

Figure 1. Wood sawdust reinforced epoxy bending strength comparison from different treatment

This level of enhancement is consistent with prior studies showing that chemical surface treatments of lignocellulosic fillers lead to stronger fiber–matrix bonding. For example, alkali (NaOH) treatment is known to remove lignin and other amorphous constituents from wood fibers, exposing cellulose and roughening the surface; this typically increases interfacial adhesion and flexural strength [12], [13]. Likewise, citric acid treatment promotes esterification between its carboxyl groups and fiber hydroxyls, improving fiber hydrophobicity and bonding with the polymer matrix [14]. In short, both treatments likely improved stress transfer across the interface, which accounts for the marked rise in bending strength [12], [14].

Due to high difference between treated sample and untreated sample, the statistical analysis was conducted only for the citric acid and NaOH treated group. First, the bending strength data for each group were assessed for normality using hypotheses H_0: data are normally distributed and H_1: data are not normally distributed. The test yielded p-values greater than the significance threshold for both citric acid and NaOH groups, indicating no significant departure from normality and thus failing to reject H_0 for either group as shown in Figure 2.

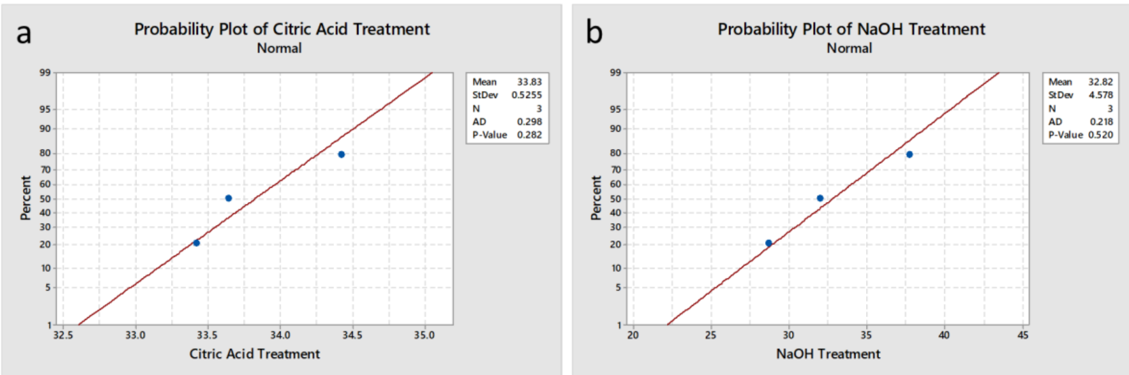

Figure 2. Normality test result for (a) Citric acid treatment and (b) NaOH treatment

Afterward, equal variances were evaluated with hypotheses H_0: group variances are equal; H_1: there is difference in variances. The equal variance test result in a statistically significant result which P value = 0.011 greater than significance level α = 0.05 as shown in Figure 3, leading to rejection of H_0 and indicating unequal variance.

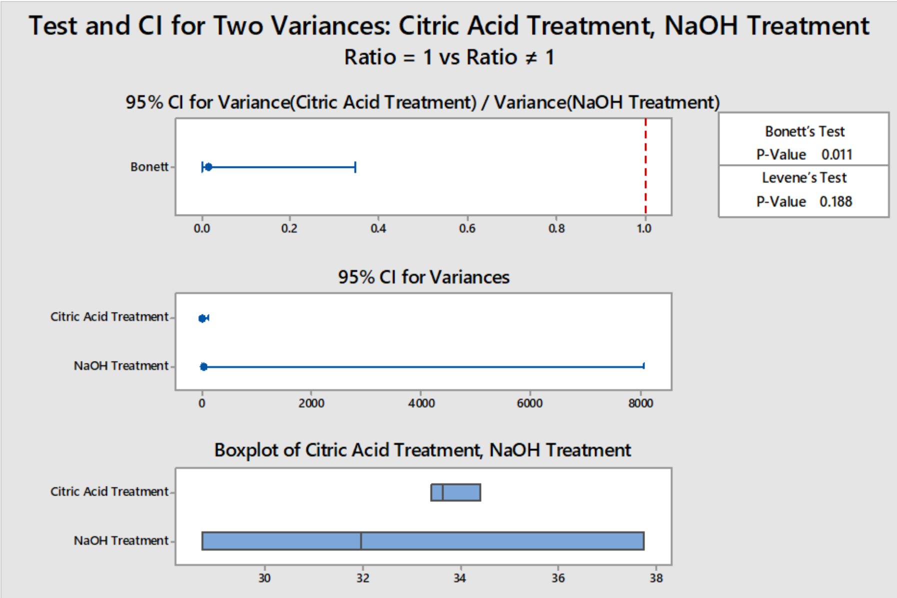

Figure 3. Test for equal variance of citric acid treatment and NaOH treatment

Due to the unequal variance, a Welch two-sample t-test is used to compare the group means. The hypotheses for the t-test are H_0: the mean bending strength is the same for both treatments, and H_1: the means is different. The result of the test is shown as Figure 4. The P value of 0.742 is greater than the significance level of $\alpha = 0.05$ and H_0 is not rejected, this indicates there is no significant difference in mean bending strength.

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Two-sample T for Citric Acid Treatment vs NaOH Treatment

                       N    Mean  StDev  SE Mean
Citric Acid Treatment  3  33.827  0.525     0.30
NaOH Treatment         3   32.82   4.58      2.6


Difference = μ (Citric Acid Treatment) - μ (NaOH Treatment)
Estimate for difference:  1.01
95% CI for difference:  (-10.44, 12.45)
T-Test of difference = 0 (vs ≠): T-Value = 0.38  P-Value = 0.742  DF = 2
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Figure 4. Welch two-sample t-test result

3.2. Effect of Surface Treatment on Hardness

The hardness value also evaluated through Brinell's hardness test with result shown as Figure 5. The mean hardness value for the epoxy, untreated, citric acid-treated, and NaOH-treated composites are 8.70 HB, 9.77 HB, 9.56 HB, and 9.01 HB respectively. These result indicate that incorporation of wood sawdust increase the surface hardness relative to epoxy.

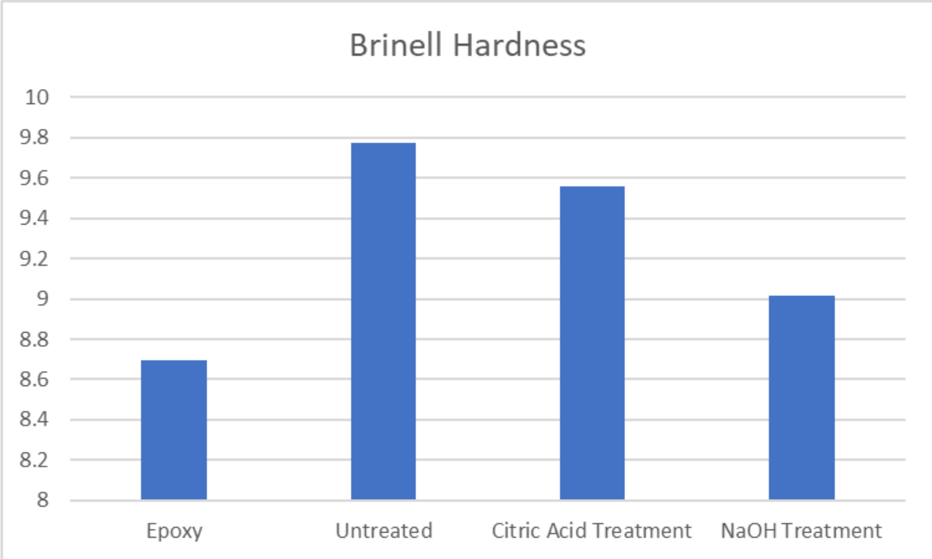

Figure 5. Brinell hardness

To determine wether these observed differences in hardness were statistically different, a one-way analysis of variance (ANOVA) was performed. The hypotheses are H_0: the mean Brinell hardness is the same for epoxy and all other treatments, and H_1: at least 1 mean is different resulting in a P value of 0.017 which is less than the significance level of $\alpha = 0.05$ and H_0 is rejected, indicating there is at least 1 mean is different as shown Figure 6.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Treatment	3	2.1891	0.7297	6.33	0.017
Error	8	0.9221	0.1153		
Total	11	3.1112			

Figure 6. Analysis of variance of hardness value

To determine wether the difference between a pair of groups is statistically significant, the Tukey Pairwise Comparison is deployed. As shown in Figure 7., the grouping from each treatment including epoxy resulting in there is significant difference between epoxy and untreated wood sawdust composite's Brinell Hardness. The range of hardness value for each treatment and the confidence interval for the difference between the means of hardness are shown in Figure 8.a and b respectively.

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

Treatment	N	Mean	Grouping
Untreated	3	9.773	A
Citric Acid	3	9.557	A B
NaOH	3	9.013	A B
Epoxy	3	8.697	B

Means that do not share a letter are significantly different.

Figure 7. Tukey pairwise comparisons result

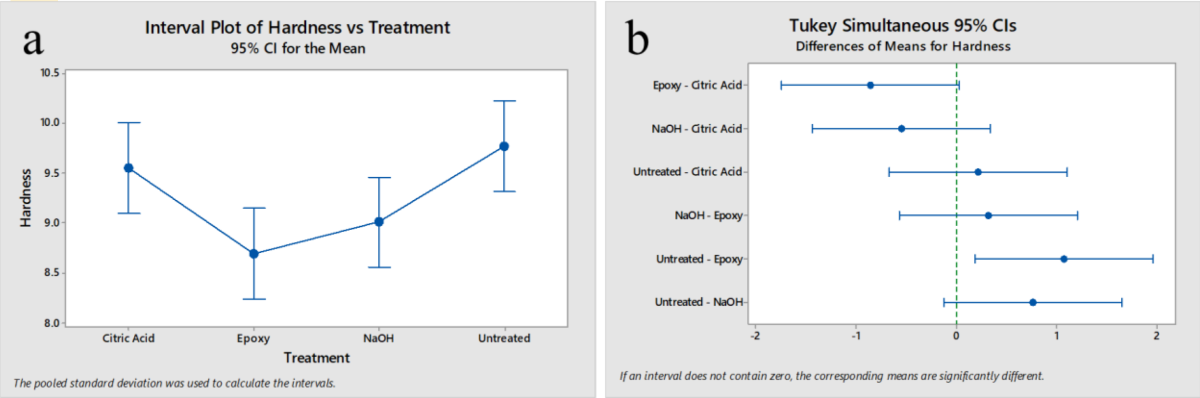

Figure 8. Interval plot of a) Hardness vs treatment; b) Difference of means for hardness

Figure 8.b show that the confidence interval for the difference between the means of untreated wood sawdust composite and epoxy does not include zero, which indicates that the difference between these means is significant. However, as shown in Figure 7. and 8.b, the hardness value for each composite of untreated wood sawdust, citric acid treated, and NaOH treated do not show any significant difference of means for hardness. These results indicate that both treatments enhanced fiber–matrix adhesion, leading to increased flexural strength without significantly affecting hardness. This aligns with prior studies showing that higher wood content generally increases hardness in wood–polymer composites [15]. However, since the wood sawdust content was constant across all specimens in this study, hardness remained unchanged. The elevated hardness values compared to pure epoxy further confirm the reinforcing effect of wood sawdust.

In summary, citric acid achieves the mechanical benefits of alkali treatment while being safer, renewable, and easy to implement. This makes citric acid surface treatment a practical alternative for producing high-strength, sustainable epoxy–wood composites, in line with trends in bio-composite research.

4. CONCLUSION

The citric acid surface treatment significantly enhanced the bending strength of the epoxy–sawdust composites compared with untreated sawdust. Statistical analysis using Welch's two-sample t-test showed no significant difference ($p = 0.742$) between the bending strengths of citric acid–treated and NaOH–treated composites, indicating that the two treatments are equivalently effective. In contrast, Brinell hardness testing revealed that wood sawdust reinforced composite achieved similar surface hardness regardless to the surface treatments, with one-way ANOVA indicating no significant difference among treatments. These results demonstrate that citric acid treatment enhances flexural performance to the same extent as NaOH without altering surface hardness. Due to its environmentally friendly characteristics, citric acid offers a viable and sustainable alternative to traditional alkaline treatments in natural fiber-reinforced composites.

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