

Effect of Mercerisation Soaking Time and Concentration on Tensile Properties of *Pentaclethra macrophylla* Fibre Reinforced Composite for Automotive Application

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Abstract

Lately, there has been a growing global awareness about the negative impact of synthetic fibres and the need for replacement with eco-friendly, plant-based natural fibres. However, the hydrophilic nature of these natural fibres can create compatibility issues with various matrices, which limits their use. This has sparked an increased research interest in finding an effective mercerization process to modify the surface of these plant-based fibres, ultimately enhancing their mechanical properties. In this study, fruit fibres from the *Pentaclethra macrophylla* plant was processed, experimented with NaOH soaking times of 1 hour, 3 hours, and 5 hours, along with treatment concentrations of 2%, 5%, and 8% by weight. The tensile properties of the composite made from these processed fibres were examined, and statistical analysis using one-way analysis of variance (ANOVA) and regression in Minitab software was conducted. The results showed a 21% decrease in tensile strength and a 32% increase in elastic modulus as the soaking time increased from 1 hour to 5 hours. Additionally, increasing the NaOH treatment concentration from 2% to 8% led to enhancements in both tensile strength and elastic modulus. This study shows that the tensile properties of the fiber composite are greatly impacted by both the soaking time and NaOH concentration. Therefore, optimising the parameters of the mercerisation process can enhance these properties, making the *Pentaclethra M.* fibre-reinforced composite a viable option for automotive applications.

Keywords: Mercerisation Treatment, Natural Fibre Composite, *Pentaclethra Macrophylla*, Sustainable Materials, Tensile Properties.

1. Introduction

Recently, there's been a noticeable shift towards using environmentally friendly and sustainable materials, particularly natural fibres for reinforcing polymers in industrial composite products. This change is largely driven by the environmental issues linked to synthetic fibres like glass fibres, which are not biodegradable and can pose health risks to humans (Andrew & Dhakal, 2022; Khalid et al., 2021). The advantages of utilizing natural fibers for composite reinforcement are quite compelling as they are lightweight, eco-friendly, strong, cost-effective, and readily available (Sanjay et al., 2016).



Because of these benefits, there has been increase research focused on renewable biocomposites that utilise natural fibres sourced from plants as alternatives to synthetic polymer composites (Sanjay et al., 2019).

Various plant-based fibres, including flax, hemp, coir, plantain, rice husk, jute, and kenaf, have been explored and are increasingly being used to replace synthetic fibres in many industrial composite applications (Amir et al., 2017; Hosseini et al., 2023). However, it's worth noting that natural fibres do come with some challenges, such as their tendency to absorb moisture and their poor compatibility with hydrophobic polymer materials. These fibres are made up of cellulose, hemicellulose, and lignin, which contribute to their hydrophilic properties and can lead to weak adhesion with hydrophobic polymers when they absorb moisture. This can ultimately result in a decline in the mechanical and thermal properties of the biocomposite products (Neto et al., 2022)

The compatibility between natural fibres and polymer matrices can be enhanced through surface modification using an appropriate chemical treatment process. By applying chemicals like NaOH alkaline solution (mercerisation), we can reduce the hydrophilic characteristics, boost the hydrophobic traits, and increase surface roughness, leading to better mechanical properties in the composite materials (Bezazi et al., 2014; Elsheikh et al., 2022; Sanjay et al., 2016). For optimal mechanical performance, factors such as soaking time and concentration percentage are crucial, as highlighted by various researchers.

For instance, research conducted on oil palm empty fruit bunches revealed notable enhancements in both tensile and flexural strengths of the natural composite material. A peak tensile strength of 20.100 MPa was attained by using a 5% weight alkaline concentration and soaking the material for 36 hours. Similarly, the highest flexural strength of 30.216 MPa was observed with the same alkaline concentration but with a shorter soaking time of 12 hours (Fatra et al., 2016). Additionally, a study on how changing the amount of NaOH and duration of soaking affects the strength of individual bamboo fibers discovered that by removing hemicellulose and other impurities from the surface, the tensile strength of the fibers increased (Salih et al., 2020). Another study on the effects of NaOH treatment on the tensile properties and thermal stability of *Dichrostachys cinerea* bark fibres (DCFS) reported optimal tensile strength and thermal stability at a 5% NaOH concentration and a soaking period of 90 minutes (Baskaran et al., 2022).

Furthermore, research on the surface modification of kenaf fibres with NaOH indicated an increase in tensile strength, peaking at a 10% weight NaOH treatment (Ismojo et al., 2019). "The study involving sugarcane-epoxy fiber demonstrated a significant improvement in the composite's mechanical characteristics after undergoing a 2-hour treatment with 5% NaOH. The composite exhibited a peak impact strength of 0.12 J/mm² and a tensile strength of 10.80 MPa (Laksono et al., 2021). In the same way, a research study on how treating sugar palm reinforced polylactic acid (PLA) affected its mechanical properties showed enhancements in tensile, flexural, and impact characteristics following a 6% alkaline treatment lasting 20 minutes (Sherwani et al., 2022). Furthermore, a study on the strength of *Cyrtostachys renda* (CR) leaf stalk fibers revealed improved mechanical characteristics after being exposed to a 3% NaOH solution for one hour (Loganathan et al., 2022).

On the other hand, the fruit fibres from *Pentaclethra macrophylla* plants haven't been thoroughly explored for composite manufacturing. Commonly known as the 'African oil bean,' this plant is prevalent in Nigeria's North Central and Southern regions, as well as along the coastal areas of West and Central Africa (Onwuzuruike et al., 2024). The fruit valve fibres, a byproduct of this plant, is produced in large amounts but often discarded indiscriminately as waste after the edible seeds are extracted. This highlights the need to find a way to turn this

waste into something valuable. In this study, the tensile properties of *Pentaclethra M.* fruit valve fibres reinforced composite, which are essentially waste from the fruit husk (pods) were examined, after treating them with an alkaline solution for various soaking times and concentrations. The research aimed to investigate the impact of varying soaking times and alkali solution concentrations on the tensile properties of this composite material. Additionally, it sought to assess the viability of using this material as a reinforcement in composites, specifically for structural and automotive purposes.

2. Methods

2.1. Processing of Materials

The fibres which were obtained from the fruit husk were sourced from *Pentaclethra macrophylla* plant in Enugu state of Nigeria. Sodium hydroxide (NaOH) pellets, methyl ethyl ketone peroxide (MEKP) catalyst, cobalt naphthenate accelerator, release agent and polyester resin of 1.114 g/cm³ density for the composite fabrication were all procured from Enugu State. The fruit was harvested, and the seeds were removed. The empty fruit husk (valve) was washed in water and dried at 40°C for 2 weeks. It was then milled and sieved with a 250µm particle size sieve (See Figure 1).



Figure 1. (a) Washing of empty fruit husk (b) Milled and dried fruit husk fibres

2.2. Sample Treatment Based on Soaking Time

The fibre particles were then separated into three portions and each treated with 2%wt of NaOH for 1, 3 and 5 hours soaking time duration. The samples were neutralised with water and dried at 40°C for 72 h in an oven.

2.2.1. Composite fabrication based on soaking time

The hand lay-up method and a resin-fibre ratio of 70:30 was used for this fabrication. First, appropriate quantity (30% weight fraction) of the 1 h treated fibres samples was measured, mixed with the corresponding amount of the polyester resins and stirred 1400rpm using a mixer. The accelerator and catalyst were added, stirred before the mixture was applied to a standard wooden mould to cure for 72 h under room temperature. The cured composite was demolded and trimmed to sample size of length, width and thickness of 250 x 25 x 4mm as shown in Figure 2. The same procedure was repeated for the 3 and 5 h treated fibre particles.

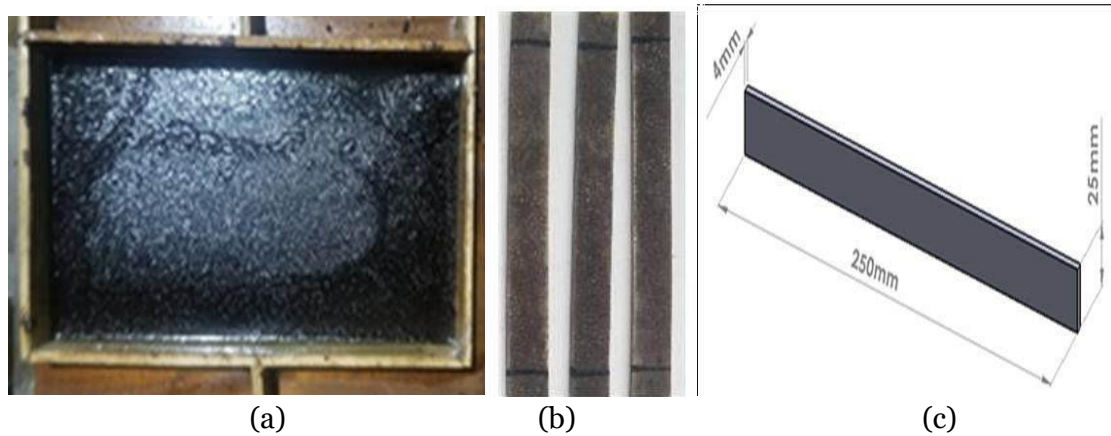


Figure 2. (a) Fabricated composite; (b) Composite test samples; (c) Composite sample dimensions

2.2.2. Tensile Test

The composite samples underwent tensile testing with a universal machine of 30-50kN at a test speed of 5mm/minute, and maintaining a gauge length of 170mm at the start of the tensioning. Each composite sample was clamped on the jaws of the machine, and as tensioning began, the values of the load and extension were recorded. The tensile strength and elastic modulus of the composite were determined from these values using Eqs. (1) and (2) (Beer et al., 1999; Hoang et al., 2020).

$$\sigma = \frac{F}{w \times t} \quad (1)$$

$$E = \frac{\Delta \sigma}{\Delta \epsilon} \quad (2)$$

where σ is the tensile stress, F is the maximum load (N), w is the width, t is the thickness, E is the elastic modulus, $\Delta \sigma$ is the gradient stress in the elastic region (MPa), and $\Delta \epsilon$ is the gradient strain in the elastic region.

From this test, the composite with the maximum tensile strength was determined, and the soaking time for this composite was utilised for further composite fabrication based on the concentration of the NaOH treatment of the fibre.

2.3. Fibre treatment based on percentage concentration

The sieved fibre particles were separated into portions, and each treated was treated with 5% and 8% NaOH solution for 1 h. The samples were neutralised with water and dried at 40°C for 72 h in an oven.

2.3.1. Composite fabrication based on percentage concentration

The hand lay-up method and a resin-fibre ratio of 70:30 were also used for this fabrication. First, an appropriate quantity (30% weight fraction) of the 5% NaOH treated fibre samples was measured, mixed with the corresponding amount of the polyester resins and stirred. The accelerator and catalyst were added, stirred, before the mixture was applied to a standard wooden mould to cure for 72 h at room temperature. The cured composite was demolded and trimmed to a sample size of length, width and thickness of 250 x 25 x 4mm. The same procedure was repeated for the 8% NaOH-treated seed husk fibre particles.

2.3.2. Tensile Test

These composite samples were also subjected to tensile tests at a test speed of 5mm/minute, and a gauge length of 170mm at the start of the tensioning was used. The values of the load and extension were recorded. The figures were utilized in establishing the tensile resistance and flexibility coefficient of the composite material.

2.4. Statistical Analysis

The accuracy of the data obtained from the tensile test was statistically analysed and plotted on graphs. One-way analysis of variance (ANOVA) and regression analysis were utilized with Minitab 16 software to examine the differences in statistical significance between the mean values of tensile strength and elastic modulus in the composite samples (Aryadoust, 2022; Shamsuri & Darus, 2020).

3. Results and Discussion

3.1. Tensile test

3.1.1. Soaking time

The tensile test result for the composite sample from fibre particle soaked for 1 h in NaOH revealed an average tensile strength and elastic modulus of 18.572 N/mm² and 942.708 N/mm², respectively. For the fibre soaked for 3 h, the average tensile strength and elastic modulus are 11.529 N/mm² and 448.510 N/mm², respectively. For the fibres soaked for 5 h, the average tensile strength and elastic modulus are 14.707 N/mm² and 1244.638 N/mm², respectively. From the ANOVA result for tensile strength (Appendix 1), the P-value for soaking time is 0.002, and this value is less than the significance level of 0.05, hence, the alternate hypothesis, which states that there is an effect of soaking time on the tensile strength of the composite, is selected. Moreover, the R-squared coefficient suggests that a significant portion of the variability in tensile strength can be attributed to the duration of fiber immersion in NaOH solution. The coefficient of determination (R-Squared) value was validated using regression analysis, which gave the same result for R-Squared and the quadratic regression model presented in Eq (3).

$$Ts = 25.93 - 8.632 * St + 1.278 * (St)^2 \quad (3)$$

where Ts is the tensile strength and St is the soaking time.

The grouping information using the Turkey method reveals that the fibres soaked for 1h in NaOH solution have a significantly higher mean than the fibres soaked in NaOH solution for 3 h.

From the individual value plot as shown in Figure 3, the tensile strength has the highest mean at the 1h NaOH soaking time and the lowest mean at 3h NaOH soaking time.

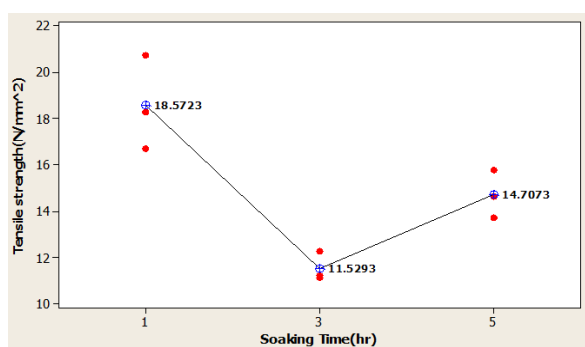


Figure 3. Individual Value Plot of Tensile Strength Versus Soaking Time

The residual plot results for the tensile strength revealed that the model meets the model assumptions since the normal probability plot (Figure 4a) showed that the residuals followed a straight line. Also, the data points are distributed randomly as shown in the versus fits plot (Figure 4b), and this indicates a constant variance. In addition, the versus order plot (Figure 4c) reveals the independence of the residuals, as there was no pattern in the time order of the residuals.

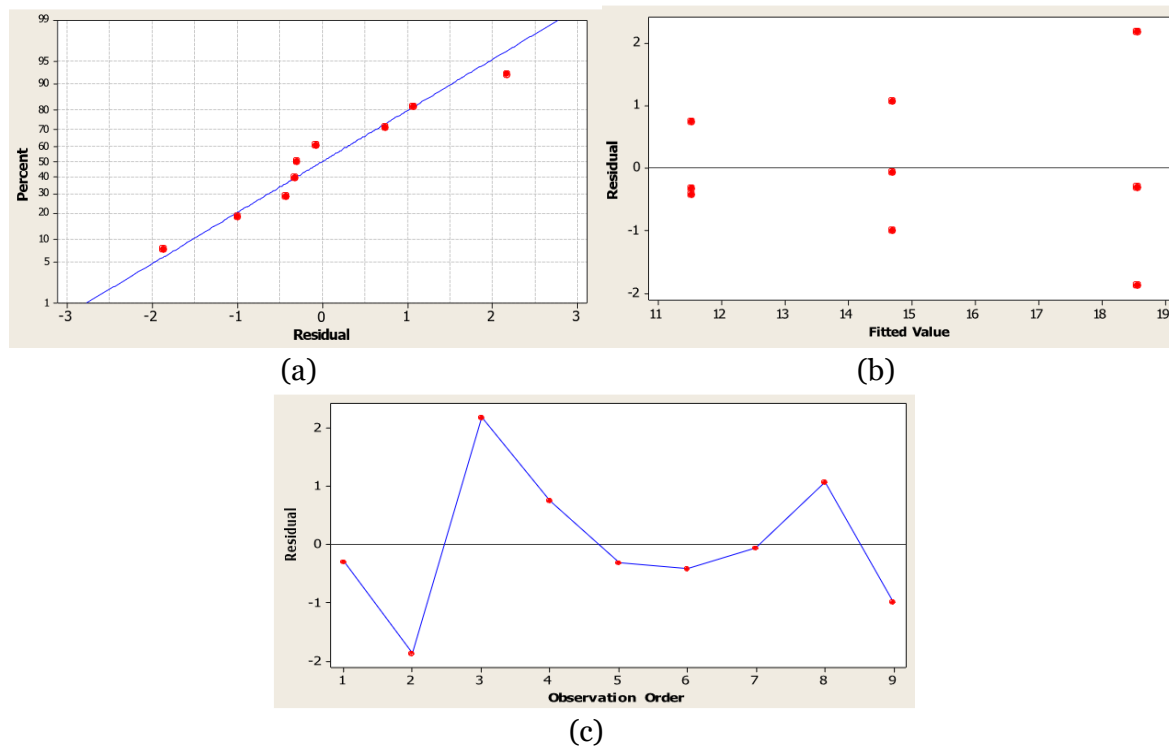


Figure 4. Residual plots for tensile strength based on soaking time in NaOH solution: (a) Normal probability plot; (b) Versus fits plot; (c) Versus order plot

Similarly, the ANOVA result for elastic modulus (Appendix 2), revealed a P-value of 0.017 for soaking time, and the value falls below the significance level of 0.05, leading us to accept the alternate hypothesis that the duration of soaking impacts the elastic modulus of the composite material. In addition, the R-Squared value revealed that 74.21% of the variation in the elastic modulus of this composite is attributed to the soaking time of the fibre in the NaOH solution. The coefficient of determination (R-Squared) value was validated using regression analysis, which gave a similar R-Squared value and the quadratic regression model presented in Eq (4).

$$E_M = 1674 - 892.3 * S_t + 161.3 * (S_t)^2 \quad (4)$$

where EM is the elastic modulus.

The grouping information using the Turkey method revealed that fibres soaked for 1h in NaOH solution have a significantly higher mean than the fibres soaked for 3 h. From the individual value plot as shown in Figure 5, the elastic modulus has the highest mean at the 5 h NaOH soaking time and the lowest mean at 3h NaOH soaking time.

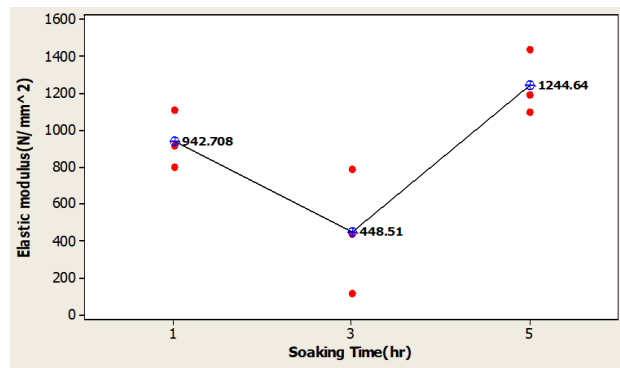


Figure 5. Individual value plot of elastic modulus versus soaking time

The residual plot results for the elastic modulus also revealed that the model meets the model assumptions since the normal probability plot (Figure 6a) showed that the residuals followed a straight line. Also, the data points are distributed randomly as shown in the versus fits plot (Figure 6b), and this indicates a constant variance. In addition, the versus order plot (Figure 6c) reveals the independence of the residuals as there was no pattern in time order of the residuals.

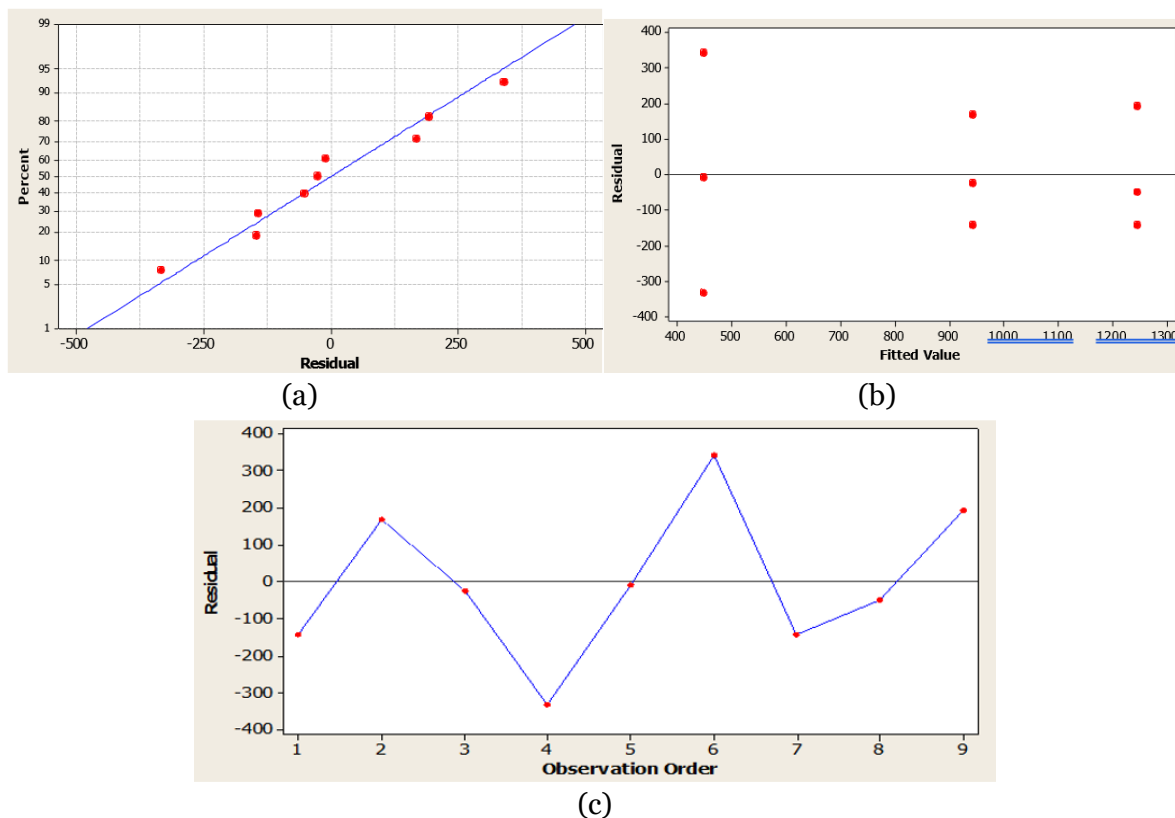


Figure 6. Residual plots for elastic modulus based on soaking time in NaOH solution: (a) Normal probability plot; (b) Versus fits plot; (c) Versus order plot

Generally, the results from the ANOVA revealed that the soaking time duration in NaOH solution significantly influences the tensile and elastic modulus of the *Pentaclethra macrophylla* fibre reinforced composite. The analysis of the result shows that increasing the soaking time duration from 1 h to 5 h decreases the tensile strength by 21%, but the elastic modulus increases by 32%. This result agrees with the reports that a 1 h NaOH soaking time duration increased the tensile strength of natural fibres (Loganathan et al., 2022). It also

agrees with the research findings on kenaf fibre treated with NaOH that the elastic modulus increased as the soaking time was increased to 5 h (Hassan et al., 2018).

3.2. Concentration of treatment

The result from the tensile test of the composite from the fibre treated with 2% NaOH concentration revealed an average tensile strength and elastic modulus of 18.572 N/mm² and 942.708 N/mm², respectively. The average tensile strength and elastic modulus for the 5% NaOH-treated fibre composite were determined as 17.78 N/mm² and 2043.079 N/mm², respectively. The average tensile strength and elastic modulus for the 8% NaOH treated fibre composite were determined as 23.555 N/mm² and 1936.904 N/mm², respectively.

The ANOVA result for the tensile strength (Appendix 3) revealed a P-value of 0.041 for concentration, which is less than the 0.05 significance level; thus, the null hypothesis, which states that there is no effect of concentration of NaOH treatment of fibre on the tensile strength of the composite, was rejected. The coefficient of determination (R-squared) revealed that 65.53% of the variation in tensile strength is attributed to the percentage concentration of NaOH treatment. The statistical significance was validated using regression analysis, which gave a similar coefficient of determination and the quadratic regression model presented in Eq (5):

$$TS = 22.75 - 2.818 * C - 0.3649 * C^2 \quad (5)$$

From the Turkey method, the grouping information reveals that the 8% NaOH treated fibre composite has a significantly higher mean than the 5% NaOH treated fibre composite. The individual value plot (Figure 7) showed that the tensile strength has the highest mean at the 8% NaOH treatment and the lowest mean at 5% NaOH treatment.

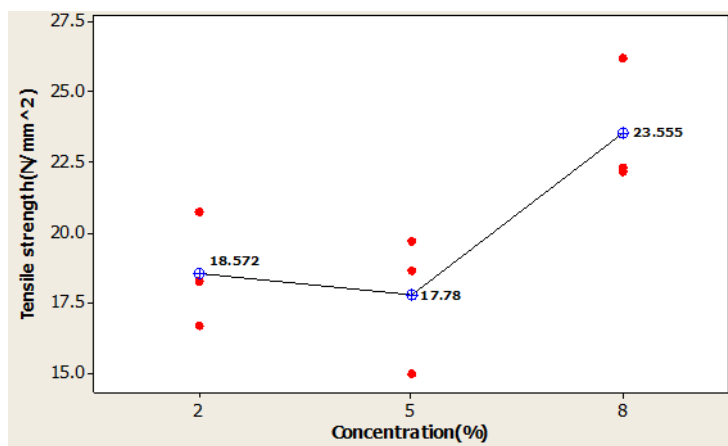


Figure 7. Individual Value Plot of Tensile Strength Versus Concentration

The result from the residual plots for tensile strength shows that the model meets the model assumptions since the normal probability plot (Figure 8a) revealed that the residuals followed a straight line. Also, versus fits plots (Figure 8b) show that the data points are distributed randomly, and this indicates a constant variance. The independence of the residuals was revealed from the versus order plot (Figure 8c) as there was no pattern in time order of the residuals.

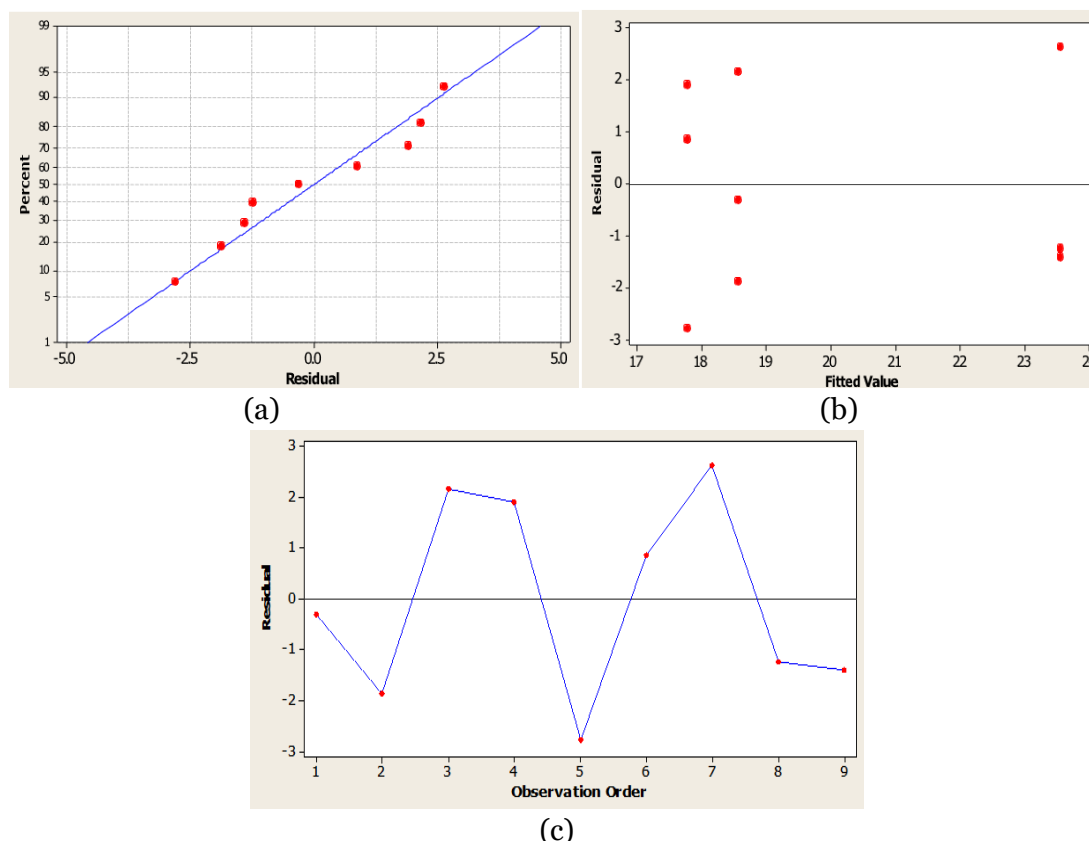


Figure 8. Residual plots for tensile strength based on concentration of NaOH treatment: (a) Normal probability plot; (b) Versus fits plot; (c) Versus order plot

The ANOVA result for the elastic modulus (Appendix 4) revealed a P-value of 0.005 for concentration which is less than 0.05 significance level, thus, the null hypothesis was rejected. The coefficient of determination (R-Sq. value) revealed that 83.28% of the variation in elastic modulus is attributed to the concentration of NaOH treatment. The statistical significance was validated using regression analysis, which gave a R-sq. value and the quadratic regression model presented in Eq (6):

$$EM = 461.2 + 836 * C - 67.03 * C^2 \quad (6)$$

The grouping information from the Turkey method revealed that the 5% NaOH treated *Pentaclethra macrophylla* fibre reinforced composite has a significantly higher mean than the 2% NaOH treated *Pentaclethra macrophylla* fibre composite. The individual value plot (Figure 9) showed that the elastic modulus has the highest mean at the 5% NaOH treatment and the lowest mean at the 2% NaOH treatment.

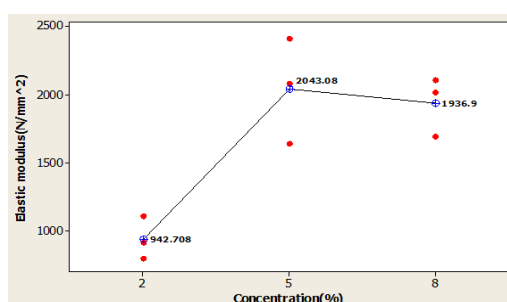


Figure 9. Individual value plot of elastic modulus versus concentration

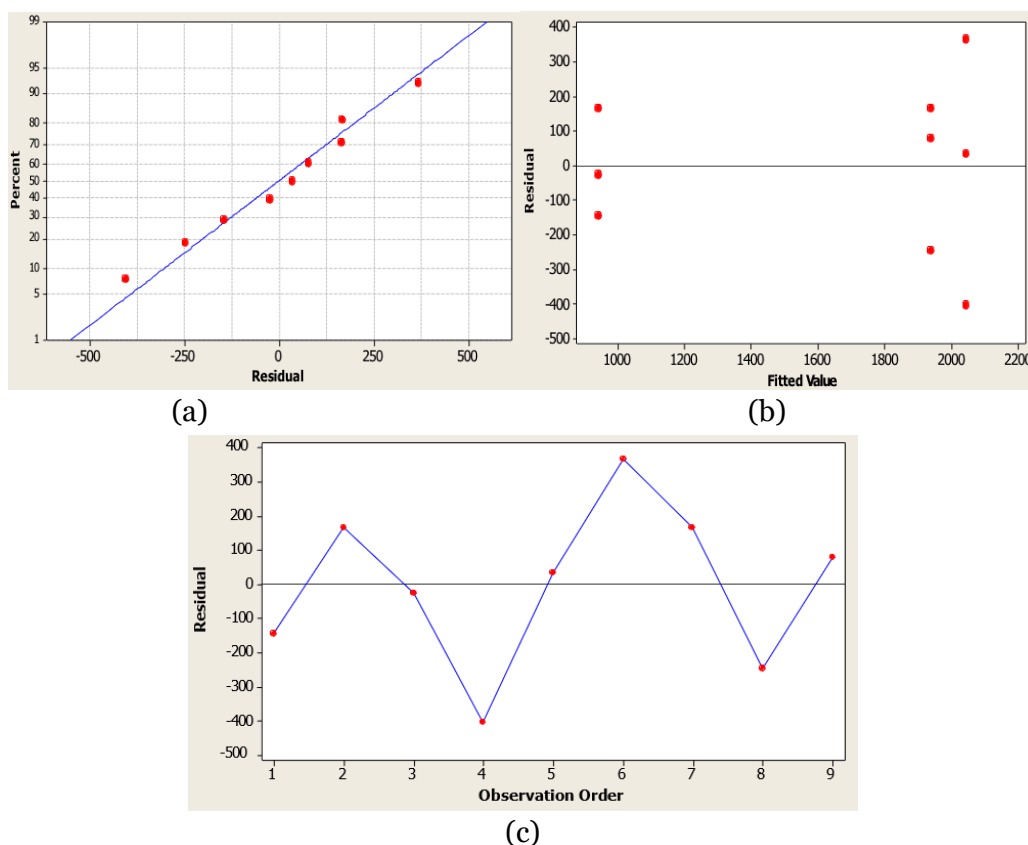


Figure 10. Residual plots for elastic modulus based on concentration of NaOH treatment: (a) Normal probability plot; (b) Versus fits plot; (c) Versus order plot

Generally, the results from the ANOVA revealed that the concentration of NaOH treatment significantly influences the tensile and elastic modulus of the *Pentaclethra macrophylla* fibre reinforced composite. The outcome of the study showed that increasing the concentration of NaOH treatment from 2% to 8% resulted in a 27% increase in tensile strength and a 105% increase in the elastic modulus of the composite. This aligns with previous research on banana bunch fiber composites, which also found that higher concentrations of NaOH treatment led to significant improvements in tensile strength and elastic modulus (Pratama et al., 2024). It also agrees with the research findings on ramie fibre treated NaOH that the maximum tensile strength and elastic modulus were obtained at 8% NaOH treatment, which is due to increase cellulose and lignin level and the lowering of hemicellulose content by the treatment; above 8%, the fibre structure is damaged (Jamilah & Sujito, 2021).

The results regarding soaking time and concentration indicate that the optimum tensile strength for *Pentaclethra Macrophylla* fibre-reinforced composites can be achieved by adjusting treatment factors like soaking duration and the concentration of the alkaline solution. This is consistent with results found in other research studies (Adeyi et al., 2021; Baccouch et al., 2020; Bhagat et al., 2024; Raharjo et al., 2024; Yudsonio et al., 2024)

4. Conclusion

Natural fibres are hydrophilic, which creates some challenges when they're mixed with polymer matrices in composite production. This often results in weak bonding between the fibre and the polymer, ultimately affecting the mechanical strength of the final product. A great way to address these issues is through surface modification, especially by applying an appropriate alkaline treatment process.

The research focused on how the treatment process of fruit fibres from the *Pentaclethra macrophylla* plant affects the tensile properties of its reinforced composite. Using one-way Analysis of Variance (ANOVA) and regression, the study found that both the soaking time and the concentration of NaOH treatment significantly influenced the tensile strength and elastic modulus of the fibre composite. The results indicated a quadratic relationship between the factors and the responses in all scenarios. Notably, increasing the soaking duration from 1 hour to 5 hours resulted in a 21% drop in tensile strength, while the elastic modulus of the composite increased by 32%. Additionally, raising the NaOH concentration from 2% to 8% led to improvements in both tensile strength and elastic modulus. These enhancements in tensile properties from the NaOH treatment were attributed to higher levels of cellulose and lignin, a reduction in hemicellulose content, and increased surface roughness, which all contributed to better bonding between the fibre and matrix. Overall, this analysis highlights that carefully choosing the chemical treatment parameters for *Pentaclethra macrophylla* fibre can significantly enhance its tensile properties, making this fibre composite suitable for automotive applications.

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4.2. Competing interests

No potential conflicts of interest were reported by the author(s).

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