






Influence of Treatments on the Mechanical Properties of Epoxy Resin Hybrid Composites Reinforced with Pineapple Fiber and Snail Shell Particulates

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ABSTRACT

The growing environmental concern regarding synthetic materials in various engineering applications is driving increased research into the production of green composites. In this study, pineapple leaf fiber (PLF) and snail shell powder amended with sodium hydroxide (NaOH) solution, at concentration levels of 0, 2, 4, 6 and 8% for 30, 60 and 90 minutes, respectively, were used to produce various composite samples; and their mechanical properties tested in agreement with American Society for Testing and Materials (ASTM) International approved procedures. The laboratory test results revealed that both the NaOH concentration and treatment period considerably influenced the tensile and flexural strengths of the composite samples. It was observed that the composite samples, made with reinforcement materials modified with NaOH concentrations of 0%, 2%, 4%, 6%, and 8% for durations of 30, 60, and 90 minutes, exhibited tensile strengths of 8.12, 9.88, 11.04, 14.11, and 16.74 MPa; 10.93, 14.22, 17.04, and 15.71 MPa; and 12.27, 15.19, 14.06, and 13.84 MPa, respectively. Similarly, the results portrayed that the composite samples produced with reinforcement materials treated with 2%, 4%, 6% and 8% sodium hydroxide concentrations for durations of 30, 60 and 90 minutes, developed flexural strength of 31.98, 38.82, 43.97 and 49.03 MPa; 36.55, 44.17, 53.38 and 47.93 MPa; and 39.62, 46.08, 48.17 and 43.66 MPa, respectively. It was also interesting to observe that 6% NaOH treatment for 60 minutes yields the optimum tensile and bending strengths of 17.04 and 53.38 MPa respectively. This finding revealed the potential of using bio-composites for engineering applications, mostly where moderate tensile and flexural strengths characteristics are sought after.

Keywords: Biodegradability, Composites, Hybridization, Organic materials, Strength optimization



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INTRODUCTION

There is a growing recognition of the environmental impact of traditional inorganic materials, especially those derived from petroleum sources. The production and disposal of these synthetic (inorganic) materials contribute significantly to pollution, resource depletion, and climate change. [David and Niculescu \(2021\)](#) reported that the production of synthetic materials often leads to the emission of harmful pollutants, including volatile organic compounds into the environment. Similarly, [Rádis-Baptista \(2023\)](#) expresses great concern about using synthetic materials in making household items, as most of these materials contain toxic chemicals that adversely affect human beings. Neurotoxicity and other hypersensitive reactions which can result in the downregulation of gene expression in human beings are some of the negative health effects of synthetic compounds ([Pinkas *et al.*, 2017](#)). The toxicity of harmful compounds depends on the duration, volume and concentration of exposure ([Uguru *et al.*, 2022](#)). Therefore, there is a global push toward developing and adopting sustainable organic alternatives for the producing and utilizing materials for both domestic and industrial applications.

Intensive research into plant-based organic materials, known for their renewability, biodegradability, and lower environmental impact, is underway for the production of composites in diverse engineering applications ([Liu *et al.*, 2017](#); [Asim *et al.*, 2018](#)). Composites manufactured from natural fibers and fillers are being rigorously researched as substantial alternatives to synthetic composite reinforcement materials. Bio-based composites are gaining industrial attention, as these materials are widely used in a variety of industrial applications ([Sivakumar *et al.*, 2017](#); [Asim *et al.*, 2018](#); [Edafiadhe *et al.*, 2019](#); [Obukoeroro and Uguru, 2021](#); [Cionita *et al.*, 2022](#)). One of the significant advantages of natural materials is their biodegradable nature; which contrasts with synthetic materials, as they persist in the environment for much longer durations. Natural materials used for composites production-particulates, fibers and resin-offer numerous advantages such as renewability, low environmental impact, energy efficiency, local availability and biodegradability, compared to synthetic materials hence, making them sustainable composite for numerous applications in the industries ([Koronis *et al.*, 2013](#); [Fiore *et al.*, 2015](#); [Edafeadhe *et al.*, 2020](#)).

The design and production of green composite with natural fillers, fibers or hybridization of both primary materials to produce highly efficient engineering materials have garnered significant attention from numerous researchers ([Usman *et al.*, 2016](#); [Adeyanju *et al.*, 2017](#); [Tepsila and Suksri, 2018](#); [Obukoeroro and Uguru, 2021](#)). The interfacial adhesion between organic reinforcement materials and the matrices is essential in determining the overall mechanical properties, of natural fiber-reinforced composite produced. A strong

interfacial adhesion correlation between the fibers/fillers and matrix help to improve the tensile strength of the composite, due to the effective transfer of tensile loads from the filler to the matrix. ([Edafiadhe *et al.*, 2019](#)). A strong interface between the reinforcement and matrix also encourages the primary elements (fibers and matrix) to work together, resulting in the composite absorbing more tensile and flexural forces/energies. This will lead to a more gradual failure rate of the composite and prevent sudden and catastrophic failure during field applications ([Edafeadhe *et al.*, 2020](#)).

Some inherent components-hemicelluloses, cellulose, pectin, lignin, waxes and water-soluble substances-of natural materials tend to limit their utilization level in the composite industry, as they cause strength reduction in the composite produced ([Ahmad *et al.*, 2019](#); [Hamidon *et al.*, 2019](#)). These limitations caused high hydrophilic behavior and smooth surface topography. The poor surface roughness of most natural fibers and particulates tends to hinder the mechanical interlocking between the fibers/fillers and the matrix; therefore leading to a decline in the overall mechanical strength and performance of the composite ([Abiola, 2017](#); [Ekwueme *et al.*, 2019](#)). An adequate understanding of overcoming these limitations is essential for producing composite materials with better engineering properties. Some solutions to the problems associated with poor bonding of natural fibers are: surface/mechanical treatment, chemical modifications, and applying hydrophobic agents to the composite during production.

The engineering properties of agricultural materials depend on the crop variety, cropping system, processing and storage methods. Different crop varieties have distinct physical and mechanical behaviors that affect the engineering properties of any material produced from them, mainly in the construction industry. Additionally, improper drying, seasoning and other processing techniques can result in severe issues related to mechanical defects-cracks, brittleness or lowered strength-of the agricultural material ([Ijabo *et al.*, 2019](#); [Nwanze and Uguru, 2020](#)). Several chemicals are being used to modify the engineering properties of organic materials and improve the mechanical attributes ([Owen *et al.*, 2018](#); [Baarimah *et al.*, 2021](#); [Mohd Ghaztar *et al.*, 2022](#)). These amendments are frequently embarked upon to improve the bio-materials engineering characteristics-strength, durability, flame resistance, and dimensional stability. A major goal achieved through alkaline treatment/modification of agricultural materials is the alteration in their hydrogen bonding structure. Hydrogen bonding plays an essential part in the structure and properties of materials; therefore, its alteration can increase the roughness of the material's effective surface area, and enhance its mechanical properties in the process ([Mohanty *et al.*, 2000](#); [Ray *et al.*, 2001](#); [Farahani *et al.*, 2012](#)).

Recently, there have been several researches on the production of hybridized pineapple leaf fibre PLF and other organic fillers to produce a sustainable material ([Siregar *et al.*, 2010](#); [Tepsila and Suksri, 2018](#); [Nnodu *et al.*, 2020](#)). Findings from related literature search revealed very little information on the production of high quality composite from the hybridization of alkaline modified pineapple leaf fiber and snail shell fillers ([Igwe, 2007](#); [Onuegbu and Igwe, 2011](#)). Therefore, the aim of this research was to achieve an optimum alkaline (sodium hydroxide) treatment for snail shell fibers and pineapple leaf fiber, and its effect on the mechanical properties of the bio-composite produced.

MATERIALS and METHODS

Materials

The epoxy resin (LY556), hardener (HY951) and sodium hydroxide used for this study were all purchased from a chemical shop at Onitsha, Anambra State of Nigeria. Likewise, the pulverized snail shell and pineapple fibers were obtained from the farm structure and materials laboratory of the Delta State University of Science and Technology, Ozoro, Nigeria.

Methods

Preparation of the snail shell

The milled snail shells were sized with a 150 mm sieve to obtain fine snail shell particulate.

Alkali modification of the organic materials

The snail shell fillers and pineapple leaf fiber (PALF) were treated with sodium hydroxide (NaOH) with varying concentrations of 0%, 2%, 4%, 6% and 8%, for 30, 60 and 90 minutes at ambient room temperature ($29\pm 2^{\circ}\text{C}$). The NaOH concentration was expressed in w/v%.

This wide range of chemical concentrations was adopted in order to optimize the treatment conditions, therefore achieving the best desired behaviors of the natural reinforcement materials. Reaction duration and temperature conditions are vital factors that influence the chemical reactions, suitability and compatibility of the product produced.

Composite samples mix ratio design

The non-metallic matrix used for the composite production was prepared by mixing the epoxy resin and hardener in a ratio of 8:2. Then the green composite was prepared by reinforcing 80 wt% of the epoxy matrix with 15 wt% PLF and 5 wt % snail shell fillers. In summary, the bio-composite was prepared by mixing the matrix and reinforcement materials at a ratio of 80:15:5. The entire weight percentage was calculated based on the weight of the matrix. For easy identification of the samples, the sample codes are presented in Table 1.

Table 1. Treatment codes.

Sample code	Treatment concentration	Treatment duration
Control	0%	0 minutes
S1	2%	30 minutes
S2	4%	30 minutes
S3	6%	30minutes
S4	8%	30 minutes
W1	2%	60 minutes
W2	4%	60 minutes
W3	6%	60 minutes
W4	8%	60 minutes
T1	2%	90 minutes
T2	4%	90 minutes
T3	6%	90 minutes
T4	8%	90 minutes

Composite preparation

The hand lay-up method was used to fabricate the composite material, using the ASTM International approved mold size of 200×150×5 mm³. Adopting the ASTM standards will ensure that the composite is in accordance with internationally established procedures and dimensions.

During the production, the filler (snail shell particulates) was mixed thoroughly with the resin for 20 minutes to obtain a near consistent mixture. After that, the hardener was added to the mixture and stirred vigorously and carefully for 5 minutes, before it was poured into the already prepared (oiled) mold. But before the mixture of the matrix and filler was poured into the mold, the required amount of the PLF was manually placed into the already prepared mold, as described by [Edafeadhe *et al.* \(2020\)](#).

Each composite sample was subjected to a dead load of 10 kg at ambient environmental conditions (temperature 30±5°C, 80±5% relative humidity) for 24 hours to expel any entrapped air from it, before it was demolded and taken to the material laboratory for mechanical properties analysis. Expelling air from the materials (reducing the porosity) helps to produce a composite with better consolidation, leading to materials with improved mechanical properties ([Edafiadhe *et al.*, 2019](#)).

Mechanical properties

Tensile strength test

The tensile strength test of the composite was conducted with the Universal Testing Machine (UTM) with 1000 kg loading capacity (Testometric model, series 500-532), in harmony with the [ASTM D3039/D3039M \(2008\)](#) procedures. The sample was tensile by the machine at 1 mm/min speed until rupture occurred (as shown in Figure 1), as explained by [Edafeadhe *et al.* \(2020\)](#). At the end of each testing, the tensile strength of each sample was calculated through Equation 1.

$$\text{Tensile strength, } \sigma = \frac{\text{Force}_{\text{Max}}}{\text{Area}} \quad (1)$$



Figure 1. A composite sample undergoing tensile strength test.

Flexural strength test

The flexural strength test of the samples was performed using the UTM in accordance with [ASTM D790 \(2017\)](#) guidelines, and employing the 3-points bending fixture method. At the end of the test, the flexural/bending strength was calculated using the expression shown in Equation 2.

$$\text{Bending Strength, } \sigma = \frac{3FL}{2bd^2} \quad (2)$$

Where; F= load (force) at the fracture point (N), L= length of the support span, b= sample width, and d= sample thickness.

Statistical Analysis

The results obtained from this work were thoroughly analyzed through the use of appropriate charts with the aid of MS-Excel for Windows. All the tests were carried out in four replicates, and the mean value was recorded for the plotting of the charts.

RESULTS AND DISCUSSION

Tensile strength

The plot presented in Figure 2 shows the tensile strength of all the composite samples prepared in this research. The results revealed that the composite samples prepared with 0%, 2%, 4%, 6% and 8% reinforcement materials treated for 30 minutes, developed tensile strength of 8.12, 9.88, 11.04, 14.11 and 16.74 MPa respectively. Also, the composite made with reinforcement materials treated with the various NaOH concentrations for 60 minutes (1 hour) had tensile strengths of 10.93, 14.22, 17.04 and 15.71 MPa, respectively. Furthermore, the findings indicated that composite samples, created from green materials modified with four different NaOH concentration levels (2%, 4%, 6% and 8%) for 90 minutes, exhibited tensile strengths

of 12.27, 15.19, 14.06, and 13.84 MPa, respectively. These findings clearly showed that the composite tensile strength increased unevenly with increasing NaOH concentration used to modify the fillers and fibers, and the duration of treatment. Among the treatment options, the composite produced with organic materials treated with 4% NaOH for one hour showed the maximum tensile strength (17.04 MPa).

These results comply with the findings of [Siregar *et al.* \(2010\)](#), when the tensile strength of polystyrene composite strengthened with pineapple leaf fiber increased from 22.64 to 29.95 MPa, as the NaOH treatment concentration increased from 0 to 4%. Furthermore, the results of this study are similar to those reported by [Wijianto *et al.* \(2019\)](#) and [Meon *et al.* \(2012\)](#). In [Wijianto *et al.* \(2019\)](#) investigation into the alkaline modification of organic fibers, they noted that the optimal tensile strength was recorded in 5% NaOH treatment for 60 minutes. Similarly, [Meon *et al.* \(2012\)](#) reported that 6% NaOH treatment of kenaf fibers for 24 hours gave the optimum mechanical properties values. The superior tensile strength observed in alkaline-treated organic materials can be linked to the improved adhesive characteristics of the materials surface ([Mohanty *et al.*, 2000](#)). Alkaline treatment often removes impurities from the materials. It modifies the surface of the materials hence, improving the adhesion capacity and compatibility of the bio-materials with matrices during composite production. This will increase the mechanical properties of the composite produced in most cases ([Meon *et al.*, 2012](#)).

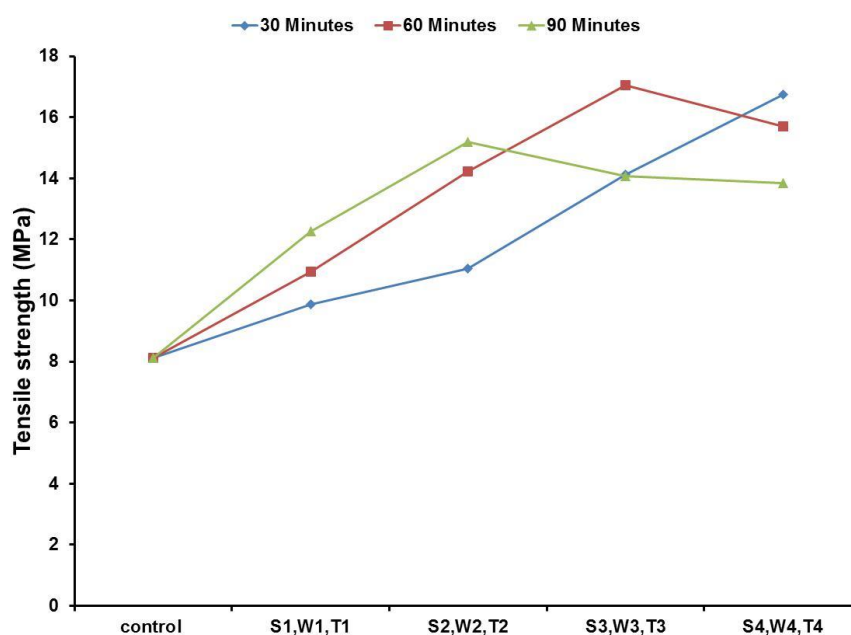


Figure 2. The tensile strength of the composite materials.

Flexural strength

The results of the flexural strength of the composite samples are presented in Figure 3. It was revealed that the composites, made with reinforcement items treated with 2%, 4%, 6% and 8% sodium hydroxide concentrations for 30, 60 and 90 minutes, developed flexural strength of 31.98, 38.82, 43.97 and 49.03 MPa; 36.55, 44.17, 53.38 and 47.93 MPa; and 39.62, 46.08, 48.17 and 43.66 MPa, respectively. Similar to what was obtained under the tensile strength, the flexural strength of the composites was highly influenced by the treatment concentration and duration. It is also interesting

to highlight those 60 minutes treatment duration yields the optimum flexural strength, irrespective of the alkaline concentration level. The impact of treatment options on the flexural strength of the materials obtained in this work is similar to those obtained by [Siregar *et al.* \(2010\)](#), where the bending strength of PLF-fortified polystyrene composite increased from 31.66 to 40.79 MPa as the alkali concentration increased from 0 to 4%. Similarly, [Cao *et al.* \(2006\)](#) reported flexural strength and modulus of biodegradable composite tend to increase non-linearly after chemical treatment of the bio-fibers, which they accredited to the deletion of impurities from the surfaces of the fibers, leading to the creation of higher bonding strength.

The findings of this study emphasize the importance of treatment options (concentration and duration) in optimizing the tensile and flexural parameters of composite materials. [Mukhlis *et al.* \(2021\)](#) in their research into the mechanical properties of natural fibers reinforced composite observed that treatment duration had noteworthy impacts on the mechanical properties of all composite specimens produced. Remarkably, these results revealed that concentration and treatment time influenced the composite materials' mechanical parameters. Therefore, by carefully manipulating the treatment concentration and duration, composite materials with enhanced mechanical properties can be produced. Exposure of bio-materials to extreme alkaline or acid environment could lead to more aggressive chemical reactions with the fibers or fillers. This might weaken the strength of the fibers/fillers; lowering the composite's mechanical properties. [Zin *et al.* \(2018\)](#) stated that exposure of organic fibers to high alkali concentrations cause extreme delignification and deterioration of the fiber, reducing its strength properties.

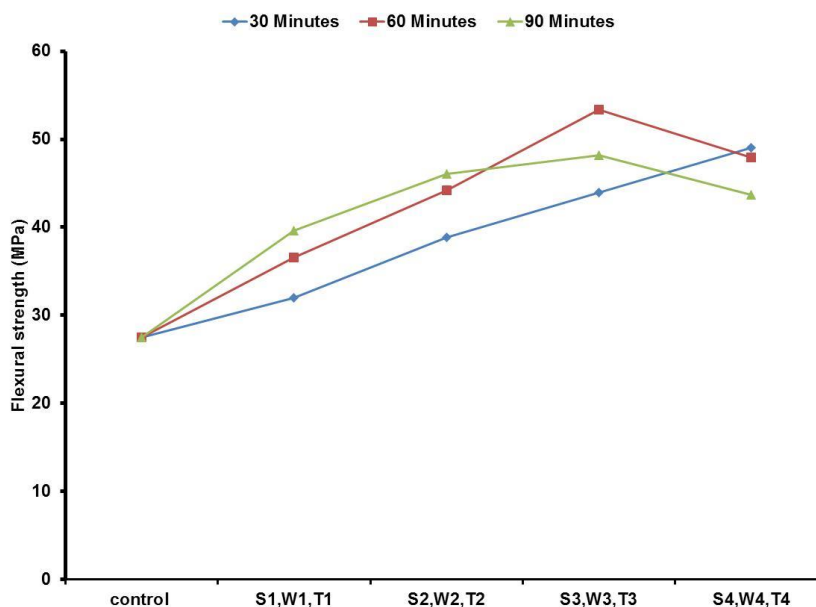


Figure 3. The composite samples flexural strength.

CONCLUSION

There is an increase in use of bio-composite, as light-weight biodegradable material for industrial and domestic applications. This research was conducted to optimize

the strength behavior of green composite, through chemical modification of the organic fillers and fibers. The pineapple leaf fiber and snail shell fillers underwent treatment with sodium hydroxide (NaOH) at concentrations of 0, 2, 4, 6, and 8%, each for durations of 30, 60, and 90 minutes. In each treatment scenario, a green composite was formulated by reinforcing 80 wt% of the epoxy matrix with 15 wt% pineapple leaf fiber (PLF) and 5 wt% snail shell fillers. The mechanical and strength behaviors of these composites were subsequently assessed in accordance with ASTM guidelines. Results of the mechanical test depicted that the treatment concentration and duration, significantly affected mechanical parameters of the composite samples. The ductile and bending behaviors of the prepared composite increased non-linearly at the alkaline concentration, and the treatment period increased linearly. This study's findings indicated that 6% NaOH treatment for one hour duration was the optimum condition to achieve the maximum tensile and flexural strengths of 17.04 and 53.38 MPa, respectively. High alkali concentration and treatment duration tends to weaken the strength of the fibers/fillers; hence lowering the strength properties of the composite produced. This study's findings revealed the potential of using alkaline-modified bio-composites for engineering applications, where moderate tensile and flexural strength characteristics are desirable.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no conflict of interest

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The authors declared that the following contributions are correct.

Onyekachukwu Nicklette Akpenyi-Aboh: Designed the research Methodology and writing of the original draft.

Moses Akwenuke: Edited the manuscript.

Donatus Edafiadhe: Designed the research and writing the original draft.

ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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