Influence of Alkali Treatment on Bamboo/Coconut Husk Hybrid Fibre Polyester Composite

*Nurul Haiza Sapiee**, *Nurul Sharifah Aina Mohammad Nizar*
School of Chemical Engineering, College of Engineering,
Universiti Teknologi MARA, Selangor, MALAYSIA
**haizasapiee@uitm.edu.my*

Chee Yun Fatt
Polycure (M) Sdn. Bhd. 2, Jalan Eco Perindustrian 1/3c,
Eco Perindustrian 5 Bandar Puncak Alam,
42300 Kuala Selangor, Selangor, MALAYSIA

ABSTRACT

This research addresses the importance of hybrid composites in material science, specifically, demonstrating the effective reinforcement of unsaturated polyester with bamboo and coconut husk fibre through alkali treatment. The primary problem this work aims to solve is the improvement of the mechanical properties of unsaturated polyester by combining it with bamboo and coconut husk fibres which contain both lignocellulosic and cellulose fibres. Alkali treatment, particularly, sodium hydroxide (NaOH) solution, was used in this study due to its cost-effective nature and its ability to enhance the strength of both individual fibres and resulting composite. The effect of NaOH treatment with concentrations ranging from 2 – 10 wt.% on dried hybrid fibres was investigated. Chemically treated fibre composites were discovered to have improved mechanical characteristics as both tensile tests and thermogravimetric analysis (TGA) showed higher strength and thermal stability after being treated. Notably, findings identified an optimal content of 8 wt.% NaOH solution for surface modification, showcasing the potential for using NaOH-treated waste fibres to reinforce polyester resin. The implications of this study extend to a promising avenue for enhancing the performance and application of natural fibre-reinforced composites. The insights provided here offer a foundation for advancing the utilization of hybrid composites.

Keywords: *Hybrid Fibre Polyester Composite; Alkali Treatment; Natural Fibres; Unsaturated Polyester; Mechanical Properties; Waste Materials*

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Introduction

Researchers are using natural fibres instead of synthetic fibres to make polymer composites due to environmental concerns. Natural fibres are regarded as being environmentally beneficial due to their low energy consumption during combustion, abundant availability, and their ability for biodegradation [1]. Natural fibre-reinforced polymeric composites (NFPCs) have received greater attention than synthetic fibre-reinforced polymeric composites. In fact, natural fibres use only 17% of the energy during production as compared to synthetic fibres [2].

Nowadays, a lot of companies across various industries are interested in natural fibres and are looking into developing novel composites to replace polymers or composites based on glass fibre [3]. Natural fibres are used in a variety of sectors, including civil construction, aerospace, and automobile industries [4]. Additionally, natural fibres have a much higher water absorption capacity than synthetic fibres. Despite the long list of benefits of natural fibres, there are still several disadvantages, such as poor mechanical qualities when compared with composites made with glass or carbon fibre [5]. To address this issue, hybrid composites are introduced as a solution for these drawbacks and to enhance the qualities of natural fibre-reinforced polymer composites. Therefore, in this research work, bamboo and coconut husk were chosen to be the natural fibre-reinforced hybrid polyester composites.

One of the reasons why bamboo and coconut husk were chosen to be hybridised together is due to the lack of sufficient number of studies being conducted specifically using bamboo and coconut husk. Hybridisation was chosen to be executed in this research work as the outcome resulted in a unique product. When a natural fibre is combined with another natural fibre or synthetic fibre in a single matrix, the composite created appears to be different from others. Specifically, in this study, bamboo and coconut husk were combined with polyester to produce a hybrid fibre polyester composite. Previous studies on other types of hybrid composites have shown to have a superior mechanical and thermal properties compared to individual fibre-reinforced polymer composites [4], [6]-[8]. These hybrid composites offer a way to combine qualities like stiffness, ductility, and strength that single fibre reinforced composites are unable to. Compared to single fibre reinforced composites, hybrid composites also have a longer fatigue life, higher fracture toughness, and lower notch sensitivity [7].

Bamboo is categorised as a non-timber forest product plant, however, due to its yield in a short amount of time, high biomass, fast growth and high efficiency in a few years, it has been recognised as a superior herb. Bamboo has a significant potential for use in building due to its nodes, which increase bending and tensile strengths, comparable to those of steel and cement [9]. The primary reason why bamboo was chosen in this research work is due to its high strength. This can be seen from the strong physical appearance of bamboo. The high strength context here can be explained by its stronger fibres which results in more robust culm structures. Bamboo culm strength is derived from multiple bamboo fibres oriented lengthwise along the bamboo's length [10]. On the other hand, a high lignin concentration in bamboo fibres prevents fibre separation, resulting in reduced matrix absorption between fibres [10]. When compared to other natural fibres, bamboo fibres have a significantly higher amount of lignin. Both lignin and cellulose contribute a lot to the improvement of the mechanical properties of a composite, where lignin improves the ductility and cellulose increases the strength [11]. This is why it is essential to utilise natural fibres with high lignin and cellulose content. Table 1 shows the composition of several natural fibres [10].

Table 1: Composition of several natural fibres in terms of their lignin and cellulose content [12]-[13]

Types of fibre	Lignin (%)	Cellulose (%)	Others (%)
Bamboo	32.2	60.8	7
Coir	41-45	36-43	13-24
Banana	5	63-68	19
Sisal	8-11	67-78	22-26
Jute	12-13	61-72	14-21
Hemp	4-6	70-74	19-24
Kenaf	15-19	31-29	21.5

Unfortunately, bamboo waste has not been utilised properly, particularly in Asian nations, due to a lack of knowledge about its strength and industrial potential [14]. Therefore, using bamboo fibres in this research could help increase bamboo's utilisation potential and reduce wastage.

Next, coconut fibre or coir, which is normally obtained from coconut husk, is also being used in this research work. Coconut husk was chosen because it is one of the highest lignin-containing natural fibres. A ligno-cellulose natural fibre is coir. It is a fruit fibre made from a coconut's skin or outer shell. Many different types of flooring materials, furniture, yarn, rope, and other products are made with this fibre. As a result, efforts have been made in research and development to find new applications for husk/coir, such as using it as a reinforcement in polymer composites [15].

Coconut husk/coir has been extensively studied as reinforcement and filler in the composite. This shows that the coconut husk has the potential as a strong fibre composite to be used in many applications. In past works, Hoang et al. [1], studied the mechanical properties of coconut trunk particle/polyester composite based on alkali treatment. They carried out experiments to study the tensile, flexural and impact properties of coconut trunk particle-reinforced polyester composites. Coconut trunk particles were taken as reinforcement and unsaturated polyester as matrix, they observed that the tensile properties were increased as sodium hydroxide (NaOH) concentration increased from 2 wt.%, 4 wt.% followed by a decrease of the increasing sodium hydroxide (NaOH) concentration. Based on the study, they discovered that the relationship between the mechanical properties and the concentration of the surface treatment solution (NaOH) was nonmonotonic. The highest findings for all mechanical parameters show that NaOH at 4 wt.% is the optimal solution concentration for coconut trunk particles and polyester resin composites.

Meanwhile, Mulinari et al. [16] studied the mechanical properties of coconut fibres reinforced polyester composites. The composites were prepared by a compression moulding technique. According to this study, it was examined that untreated and treated fibre showed difference conditions, where the untreated fibre showed a large number of debris adhering to the surface of the fibres bundles due to it being coated with cellulosic material. Meanwhile, the treated coconut fibre will have the removal of wax, pectin, lignin and hemicellulose on fibre surface.

In addition to that, there have been several research reviews of natural fibre-reinforced hybrid composites. The importance of these studies may be related to the essential properties of natural fibre-based hybrid composites, which have been discovered to be predominantly impacted by variables such as variation in fibre volume/weight percentage, change in stacking sequence of fibre layers, fibre treatment, and environmental conditions. For instance, it was discovered that natural fibre composites have better strengths than wood composites and some plastics, which increases the likelihood that they will produce better and more reliable useful goods in the future [7].

Despite the several advantages of using natural fibres as the solution to reduce the reliance on limited natural resources, there are still several disadvantages to using natural fibres. Due to their unexpected chemical compositions and inadequately prepared surface quality, this can result in reduced interfacial strength between the fibre and matrix, thereby causing a decline in the mechanical properties of the composite materials. To work optimally, composite materials must have good interfacial adhesion between the fibre and matrix [7]. Thus, in conjunction with fulfilling the companies' needs for the most reliable substitution for synthetic fibre, the authors have chosen the chemical treatment of fibres as one of the strategies to establish a good bond strength between fibre and matrix, leading to the enhancement of mechanical properties. This is in line with the main purpose of this study, which is to enhance the surface quality of natural fibres.

Therefore, this study presents a study of mechanical properties (with respect to tensile strength) by using alkali treatment on the combination of bamboo and coconut husk in unsaturated polyester resins. Unsaturated polyester resin was used in this study as it is one of the most common polymers used as a matrix in composites for application in various industries. Meanwhile, alkali treatment was chosen as it is cost-effective method to obtain high performance natural fibre as it can increase the strength of both fibres and composite [17].

Several attempts have been made to enhance the performance of bamboo/coconut husk hybrid fibre reinforced composites on mechanical properties. Several of them, however, are based on research into the mechanical properties of composites reinforced with synthetic polymers. A few examples of synthetic polymers include polyethylene (PE), polystyrene (PS), polyester, poly (vinyl chloride) (PVC), epoxy, Teflon and many more [18]. An attempt was made to study the mechanical properties of bamboo fibre and particulate coconut shell incorporated into polyvinyl chloride (PVC) matrix, which highlights varying the proportions of bamboo fibre and coconut shell particulate [19].

Another study on the same treated hybrid fibre with different synthetic matrices was done by [20], where the author aimed to improve material qualities in terms of NaOH concentration for treating coconut and bamboo fibre in order to improve the mechanical properties of natural fibre epoxy hybrid composites. Since there are no reports on the mechanical properties of alkali treated bamboo/ coconut husk hybrid fibre compounded with polyester resin, the results are undetermined. A study conducted by Akash et al. [21], studied the mechanical properties of treated and untreated sisal fibre reinforced polyester composites, whereby the results obtained from the study revealed that treated sisal fibre showed good mechanical properties compared to untreated sisal fibre. Similarly, a review by Deshmukh and Singh [22] also concluded that pre-treatment has a good impact towards the mechanical properties of natural fibre reinforced polymer composites.

In this study, polyester was chosen as a matrix due to its low cost, availability of high mechanical qualities, and use in a variety of applications, such as transportation, maritime, and sports. Although the effect of alkali treatment on individual fibre has been investigated and found to improve after chemical modification, very limited efforts have been made to determine the effects of NaOH concentration (%) on the polyester composite and show great mechanical properties to the hybrid fibre. Specifically, experimental investigations were performed to determine the effect of alkali treatment on the mechanical properties of the bamboo/coconut husk hybrid fibre polyester composite.

Materials and Methodology

The experimental steps were categorized into three main parts, the fibre treatment, the sample fabrication and finally the mechanical testing which will be further described below. A rough flowchart of the methodology is shown in Figure 1.

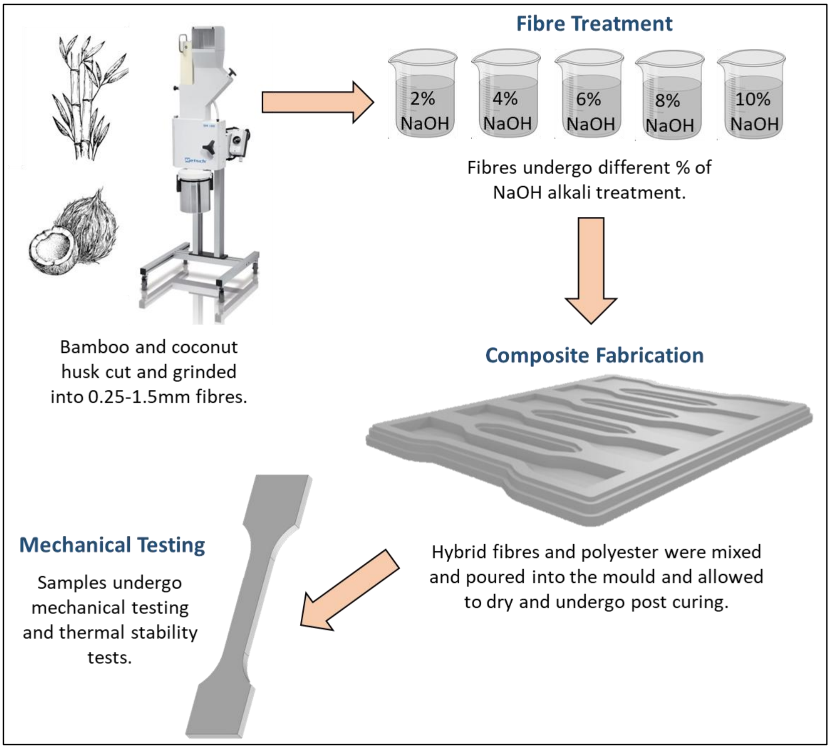

Figure 1: General schematic of the experimental steps performed to produce and test the alkaline treated bamboo and coconut husk hybrid polyester composite

Materials

Bamboo residues and coconut husks were collected from a woodland in Puchong, Selangor. Raw materials were cut into small sizes and ground into powder form using a cutting mill (Retsch SM 100). Figure 2 shows the coconut husk and bamboo prior to being ground. After grinding, the average size of the ground fibres ranged from 0.25 - 1.5 mm.

Figure 2: Image of the coconut husk and bamboo residue after being cut and before being ground into fine particles

Unsaturated polyester resin (R-230) in the form of resin pellets was obtained from Polycure Industries (M) Sdn. Bhd. located in Puncak Alam, Selangor, and used as the matrix as a weaker material being imbedded by natural fibres (known as reinforcement). Unsaturated polyester was selected as it is a common thermoset that is used in industrial applications due to its strong mechanical qualities, particularly when reinforced with fibres or fillers. A rough representation of the functions of the bamboo and coconut husk as the reinforcement in the unsaturated polyester resin is shown in Figure 3.

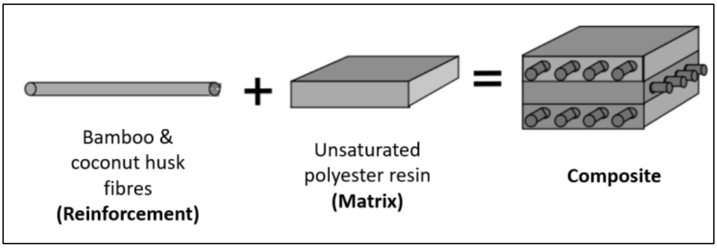

Figure 3: Rough representation of the composite material where in this study, the unsaturated polyester resin is reinforced with bamboo and coconut husk fibres which have been treated with NaOH

Sodium hydroxide pellet, NaOH from R&M Chemicals, and hardener Methyl Ethyl Peroxide (MEKP) (D-5003) from Polycure (M) Sdn. Bhd. was used as an alkali treatment and accelerator. The function of sodium hydroxide is as the alkali treatment for the hybrid fibre, meanwhile, hardener MEKP is used in room temperature curing for 100 g of resin to solidify the polyester resin.

Fibre treatment

Distilled water was used to dilute the solid state sodium hydroxide (NaOH). Meanwhile, dried hybrid fibres of both bamboo and coconut husk were fully immersed in the NaOH solution for 2 days. Afterwards, the fibres were taken out and rinsed with distilled water. Finally, the treated hybrid fibres were dried at 40 ℃ for 2 hours in an oven. Five different samples of hybrid fibres immersed in different concentrations of NaOH solution (2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%, and 10 wt.%) were prepared for the composite fabrication and testing purposes.

Composite fabrication

In order to fabricate the composite, 1.5 ml of the hardener, MEKP, was mixed with 45 g of unsaturated polyester resin matrix, in accordance with the manufacturer's recommendations. The treated hybrid fibre was mixed with polyester resin with a weight ratio of 1:15. This ratio was chosen after several trial-and-error attempts were made on the mixing ratio due to the instability of the sample solution. The ratio for both treated hybrid fibre and polyester resin for each of the 5 samples of NaOH concentration (2%, 4%, 6%, 8%, and 10%) are fixed at 3 g and 45 g, respectively. A total of 18 samples were made, with 3 samples for each treated concentration and an additional 3 samples for which hybrid fibres were untreated. The sample compositions are further illustrated in Table 2.

Table 2: Composition for each composite sample

Treatment of fibres	Fibres		Resin		Total samples
NaOH solution (wt %)	Bamboo fibre (g)	Coconut husk fibre (g)	Polyester (g)	MEKP (g)	
Untreated	1.5	1.5	45	1.5	3
2	1.5	1.5	45	1.5	3
4	1.5	1.5	45	1.5	3
6	1.5	1.5	45	1.5	3
8	1.5	1.5	45	1.5	3
10	1.5	1.5	45	1.5	3

Next, the mixture was poured into the mould where it will be pressed to ensure uniformity. The mould used was based on the dumbbell-shaped tensile testing standards (ASTM: D-638-10) with dimensions of 165 x 13 x 4 mm, as shown in Figure 4. Afterwards, the composite plate was cured inside the compressed mould at room temperature. In order to remove the sample, the mould was opened uniformly and postcuring was done in an oven at a temperature of 80 °C for 2 hours by holding the sample inside the oven [1]. Postcuring was done to increase strength and decrease residual stress and outgassing in thermosets. Experiments have shown that high postcuring temperatures cause ether crosslinking and polymer backbone dehydration [23].

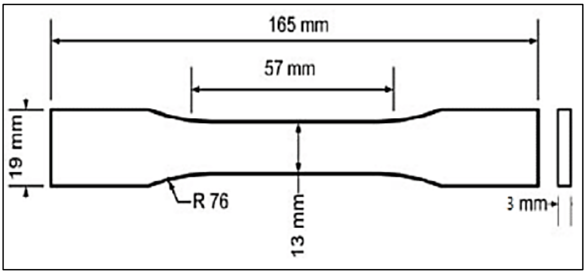

Figure 4: Dimensions of the specimen for tensile testing in accordance with the ASTM: D-638-10

Mechanical testing

After the successful fabrication of the samples, tensile tests were performed and measured using a computerised SHIMADZU Servopulser (SEL-100Kn) universal testing machine from Strength of Material Laboratory II, School of Mechanical Engineering at UiTM Shah Alam. This test was done to examine the ability of the hybrid fibre to resist breaking under tensile load. The crosshead speed used was 5 mm/min. The results of mechanical strength properties and correlation of NaOH concentration were recorded for all the bamboo and coconut husk fibre reinforced polyester composite. The average value was reported for each different concentration of NaOH. Tensile strength (σ_T) and elastic modulus (E) are as in Equations (1) and (2).

$$\sigma_T = \frac{P}{bt} \tag{1}$$

$$E = \frac{\Delta\sigma_T}{\Delta\varepsilon_T} \tag{2}$$

From the equations above, P indicates the load, b is the width of the specimen, (mm), t indicates the thickness of the specimen, (mm), $\Delta\sigma_T$ is the gradient stress that took place in the elastic region, (MPa) and lastly, $\Delta\varepsilon_T$ is the gradient strain in the elastic area.

Thermal stability of hybrid fibre

The thermal degradation of materials was investigated using the NETZSCH STA 449 F3 Jupiter. Thermal analysis was performed on an 8 mg sample from

30 ℃ to 600 ℃ at a heating rate of 10 ℃/min in a nitrogen atmosphere with a flow rate of 50 ml/min. The sample was analysed by putting it in an Alumina Al_2O_3 crucible. Nitrogen was utilized to improve heat transmission and remove volatiles from the sample. The results obtained were plots of the weight loss vs temperature.

Results and Discussions

All 18 samples were successfully fabricated and subjected to both mechanical and thermal tests. Figure 5 shows an example of the treated bamboo-coconut husk hybrid composite being tested in the universal testing machine and the samples before and after tensile testing, respectively.

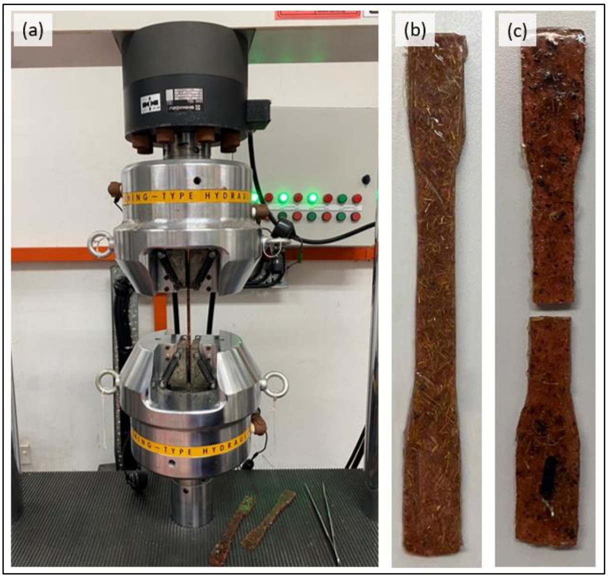

Figure 5: (a) Sample being tested in the universal testing machine, (b) example of the bamboo-coconut husk hybrid fabricated before testing, and (c) example of the bamboo-coconut husk fibre composite after tensile testing and failure

Mechanical properties

Tensile strength, or a material's ability to resist breaking under tensile strain, is one of the most important and thoroughly researched properties of composite materials utilised in structural applications. A total of eighteen tensile specimens were tested, with each NaOH concentration ranging from untreated hybrid fibre, 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.% that were represented have three specimens each. Figure 6 depicts a non-monotonic relationship between NaOH concentration and the average tensile strength and elastic modulus of bamboo and coconut husk particle/unsaturated polyester resin composites.

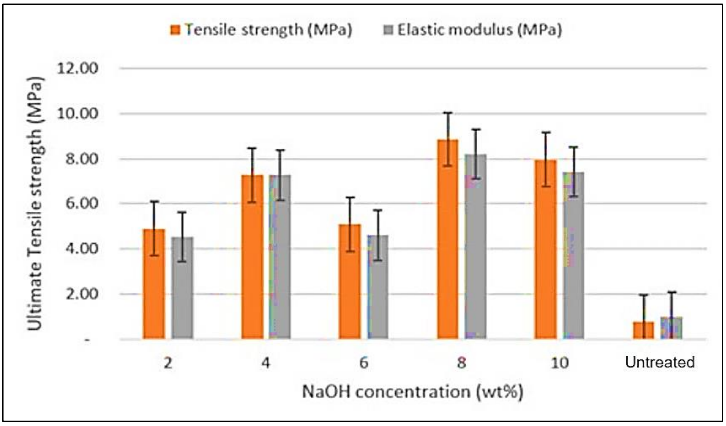

Figure 6: Tensile strength (MPa) and elastic modulus for both untreated and treated hybrid fibre at different concentrations of NaOH used for treatment.

The tensile properties showed a notable increase as the concentration of alkali solution treatment increased from 2 wt.% to 4 wt.%. After reaching its peak, there was a slight decrease in the tensile properties at 6 wt.%, followed by an increment at higher NaOH concentrations until 10 wt.%. Similar trends can be observed for the elastic modulus. According to the graph, the hybrid composite has a significant improvement in tensile strength over the untreated hybrid fibre composites. One possible reason for this is due to the insufficient elimination of impurities from the fibres when it has not been treated. As a consequence, it leads to weak interfaces within the fibre matrix and a higher level of delignification, ultimately causing fibre weakening and damage [24].

Another reason to support why untreated hybrid fibre was found to be the weakest is because the alkali treatment used on the natural fibre showed improvements in tensile strength. Another important factor contributing to higher tensile strength in treated hybrid fibre could also be the curing process that was done on the composites where, theoretically, it can strengthen the durability of the composite. The curing process allows for the creation of strong chemical connections between the fibres and the matrix, boosting load transfer capabilities and strengthening the composite's overall strength and stiffness [20], [25].

Compared to an untreated hybrid fibre that underwent postcuring, the tensile strength would certainly improve, however, it may have compatibility between the hybrid fibres and the polyester matrix. When this happens, it usually indicates a weak adhesion, also referred to as the molecular interaction between the fibre and the reinforcement. Thus, to enhance this, a surface treatment method can be employed for the fibres to modify their surface characteristics and promote better adhesion [24]. Therefore, many factors have influenced the hybrid fibre to have higher tensile strength over the untreated hybrid bamboo and coconut husk reinforced unsaturated polyester composites.

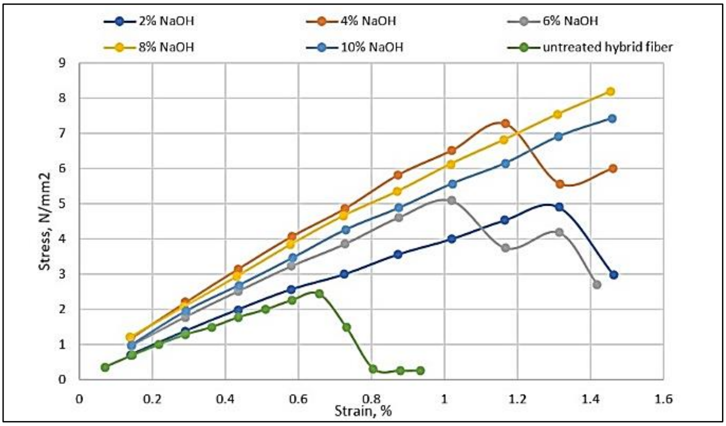

Figure 7: Stress-strain curve for both untreated and treated hybrid fibres at different NaOH concentrations

The findings of this study have led to the development of a polyester hybrid composite with excellent tensile properties. The tensile strength of the material is greatly improved by removing hemicellulose, wax, lignin, and other impurities. Additionally, it facilitates efficient stress transfer within the composite, which is shown by the improved yield strength shown by the stress-strain curve illustrated in Figure 7. The application of an alkaline treatment also leads to an expansion in the surface area of the fibril fibres, enabling them to effectively bond with the resin when moistened [26]. Based on these findings, it can be inferred that the hybrid composite contributes to the formation of a composite material with enhanced value and improved tensile strength characteristics.

Thermal stability

The thermal stability of the composite is important to ensure that the composite can endure heat during the manufacturing process while maintaining its properties. To ensure the thermal stability of the hybrid composite polyester, Thermo-Gravimetric Analysis (TGA) was performed. TGA measures the weight of a material sample as a function of temperature and/or time in a precisely controlled atmosphere, which in this study, specifically uses nitrogen. Typically, the thermal analysis of a material is conducted to assess the chemical, physical, and structural alterations that took place within the material when subjected to a temperature variation [27]. The TGA analysis was conducted to investigate the extent of weight loss observed in polyester at different temperature levels.

The hybrid composites' TGA results describe their thermal stability at high temperatures. Thermal stability is considerably influenced by chemical composition, thermal history, and environmental conditions such as temperature, pressure and many more. The weight loss at the onset of thermal degradation is explained by the TGA curves given in Figure 8.

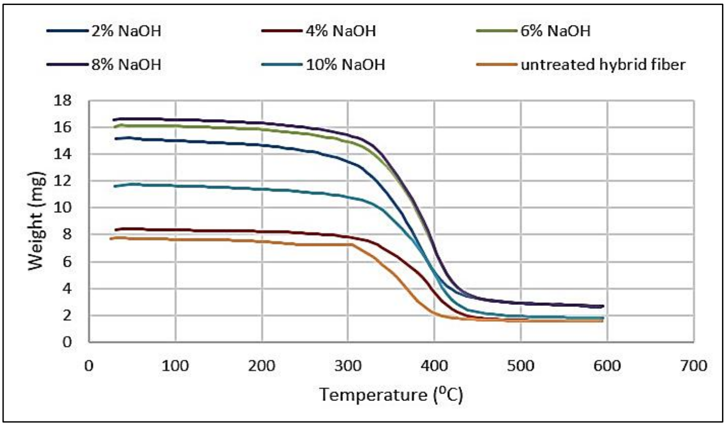

Figure 8: TGA curves of untreated and treated hybrid composites at different NaOH concentrations

The TGA analysis showed that composite samples for NaOH concentrations of 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%, 10 wt.% and untreated hybrid fibre presented weight loss at 70 ℃ due to vaporisation and removal of bound water in the samples in the polyester matrix. Major weight loss as shown in Figure 5 occurs at 310 ℃ (13.05 mg), 330 ℃ (7.80 mg), 315 ℃ (15.2 mg), 335 ℃ (15.6 mg) and 325.10 ℃ (10.8 mg), and 305 ℃ (7.10 mg), respectively. However, the 2 wt.% and 4 wt.% treated hybrid fibre showed an early weight loss at 75 ℃ owing to solvent release from the matrix, and a major weight loss at 310 ℃ (13.05 mg) and 330 ℃ (7.8 mg) due to polyester and fibre volatilisation and degradation.

There are three stages involved in the thermal degradation where the next weight loss of the treated hybrid fibre is attributed to thermal degradation of the decomposition of hemicellulose [28]-[29]. This will usually take place as the second stage in the TGA curve after the water evaporating stage [30]. Based on Figure 8, during the weight loss phase between 200 ℃ and 400 ℃, the molecular instability inside the composites weakens the contact, allowing the degradation process to proceed more quickly. However, at higher temperatures, the interfacial bonding characteristics of the composites, as well as the presence of chemical elements such as a-cellulose and lignin, may contribute to their capacity to endure heat [31].

According to the TGA curve, it can be stated that all of the compositions of sodium hydroxide (NaOH) concentration (2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.%) have shown thermal decomposition weight loss at higher temperature during the degradation compared to untreated hybrid fibre polyester composites. Overall, this shows that treated hybrid fibre has good thermal stability. The findings show that the thermal stability of the treated hybrid composites is enhanced as predicted and corresponds well to that previously reported by Asim et al. [32]. As a result, the treated hybrid fibre composites created in this work have capability in engineering applications.

Conclusions

The concentration of NaOH employed in the treatment has a significant impact on the mechanical properties of the hybrid bamboo/coconut husk unsaturated polyester resin composite material as shown by the improved mechanical and thermal properties. The optimal alkali treatment composition for coconut husk particles was discovered to be 8 wt.% NaOH, which produced the best results in tensile, and TGA tests. According to the thermal analysis, the weight loss in composites was influenced by certain factors such as chemical treatment. Therefore, it is shown that chemically treated hybrid fibre has shown better thermal stability compared to untreated hybrid fibre as discussed.

This study has also highlighted the potential use of waste material, both bamboo and coconut husk to help reinforce polyester resin so that it can be used in various engineering applications. The enhanced mechanical properties give the ability to endure the requisite heat during the manufacturing process and maintain their inherent properties following heat exposure. For instance, employing natural fibres characterised by low lignin content yields superior thermal performance in composites. Additionally, the incorporation of synthetic fillers or fibres can be utilized to enhance the thermal stability of natural fibre reinforced composites.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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Conflict of Interests

All authors declare that they have no conflicts of interest.

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