RESEARCH ARTICLE

Enhanced Mechanical and Thermal Properties of Alkali-Treated Coir Fibers Incorporated with Murraya Koenigii: A Comparative Study of Mono-Layer and Tri-Layer Biocomposites

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Abstract: This study examined the surface morphology and thermal behavior of the coir fibers incorporated with Murraya koenigii before and after alkaline treatment. The mechanical properties of biocomposites reinforced by the untreated fibers, treated fibers, mono-layer and tri-layer treated fibers incorporated with Murraya koenigii were systematically investigated. The experimental results showed significant enhancements in tensile strength, flexural strength, hardness, and impact resistance of the biocomposites reinforced by treated fibers. Notably, the alkaline treatment has produced a rougher surface on coir fibers, promoting improved mechanical interlocking between the fibers and the polyethene matrix. These effects become more significant when Murraya koenigii particles are incorporated into the fibers. It was found that the tri-layer treated fibers incorporated with Murraya koenigii biocomposites showed higher tensile and flexural strength, superior hardness, and impact strength when compared to the mono-layer reinforcement biocomposites. At higher temperatures, the treated fibers showed better thermal stability than the untreated fibers and polyethene. Incorporating Murraya koenigii particles increased the thermal resistance of the treated fibers, revealing the potential of this green synthesis approach for the development of thermally stable natural fiber composites.

Keywords: coconut coir fibers, Murraya koenigii, surface morphology, thermal behavior, mechanical properties

1. Introduction

Since the last decade, sustainable green wastes have been of increasing interest due to their beneficial applications in various end uses such as agriculture, biofuels, and engineering. These green wastes include wood chips, straw, and plant-based fibers. In comparison with other green waste materials, plant-based fibers have been reported to exhibit more significant energy efficiency during production. Wang et al. [\[1](#page-7-0)] compared various types of plant-based fibers with man-made fibers and found that the coir fibers show higher toughness in tension, larger elongation at failure, and the least global warming potential (the potential to release $CO₂$ to the environment during production is insignificant). Coconut coir fiber is well-known as an essential crop species in tropical countries such as Malaysia. Many researchers have reported the benefits of using coconut coir fibers as reinforcement material due to their rich cellulose and lignin in contents, making the pre-treated fibers more water-resistant with enhanced stiffness [[2](#page-7-0), [3\]](#page-7-0). It is also worth noting

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that when the coir fibers are treated with alkaline, the hydroxyl groups (O-H) could be removed, resulting in increased strength [\[4](#page-7-0)]. Although coir fibers exhibit various beneficial effects than other plant-based fibers, the major drawback of coir fibers is their porous structures that are susceptible to moisture, little resistance to microbial attacks, and weak adherence to the matrix surface [\[5](#page-7-0)]. To synthesize these fibers by green route, this study proposes to incorporate the coconut coir fibers with Murraya koenigii in the composite fabrication. Murraya koenigii has been used as the additive in the development of synthesizing metal oxide nanoparticles [[6](#page-7-0)–[9\]](#page-7-0) and as the thin layer in an indirect solar layer [\[10](#page-7-0)]. However, literature regarding the potential of Murraya koenigii as a composite reinforcement is limited and needs further investigation.

To date, the use of agricultural waste materials in composites has piqued industries' interest. In particular, plant-based fibers have been found to outperform other natural fibers as the reinforcement material in the composite. Plant-based fibers are comparatively cheap, abundantly available, renewable, and biodegradable. The lignocellulosic fibers include coir, flax, hemp, kenaf, jute, rice husk, and softwood materials. Coir fibers collected from coconut husks *Corresponding author: Hooi Peng Lim, Department of Mechanical and softwood materials. Coir fibers collected from coconut husks
Engineering, Politeknik Ibrahim Sultan, Malaysia. Email: limhooipeng@ show superior properties

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high hardness and stiffness, and hard-wearing capability [[2](#page-7-0)]. In addition, the usage of coir fibers reinforced in composite was found to enhance thermal conductivity. Despite being acknowledged to have advantageous properties, coir fibers are also associated with drawbacks due to their porous structures. In the past, several efforts have been made to overcome the aforementioned weakness through physical or chemical surface modification. Therefore, this study aims to produce hard yet tough coconut coir fibers incorporated with Murraya koenigii particles reinforced biocomposites using alkaline treatment. In this study, coconut coir fibers are proposed to be treated chemically using 6% sodium hydroxide (NaOH). Following alkaline treatment, a hard yet tough biocomposites consisting of polyethene resin matrix was reinforced by coconut coir fibers incorporating Murraya koenigii particles with various architectures, i.e., mono-layer and tri-layer reinforcement.

2. Coconut Coir Fibers and Murraya Koenigii

2.1. Morphology and physical properties of coconut coir fibers

A composite is created by combining two or more components, the resultant substance having different properties than the original materials. Usually, the composite consists of two materials: a matrix or resin and a reinforcement for the matrix. These reinforcements are utilized to enhance the material's sustainability, reduce weight, and increase mechanical properties. This study aims to produce alkalinetreated coconut coir fiber incorporated with Murraya koenigii biocomposites in the polyethene resin matrix. The reason for utilizing natural coir fibers and plant-based particles as reinforcement materials is that they are generally biodegradable and non-toxic. The polyethene resin matrix is one of the thermoplastics which can be reused and reformed.

Coconut, also known as Cocos nucifera, is abundantly available in most tropical countries, especially Malaysia. It is commonly used to extract coconut oil for food and cosmetics. The by-product of coconut is the coir fibers extracted from the coconut's mesocarp. It is usually done by storing the coconut husk in water for a water-retting process to separate the fibers by biologically degrading the pectin that holds the fibers together. The fibers are then washed and dried after the retting process.

In general, coconut coir fiber has a cylindrical shape with a diameter of roughly 10−460 μm [[11](#page-7-0)]. It contains a hollow central section with 200−300 elementary fibers implanted, as shown in Figure 1 [\[12](#page-7-0)]. An elementary fiber is a hollow cylindrical structure composed of individual fiber cells with a diameter of 10−20 μm and a length of around 1 mm [[13](#page-7-0)]. Coconut coir fibers have a cellulose content (approximately 15−40%) in the primary cell wall and are arranged in disorderly scaffold fibrils [[14](#page-7-0)]. The cellulose content in the secondary cell wall rises to 90% of the dry weight [\[15\]](#page-7-0). These cellulose fibrils are tightly packed and organized helically. The cellulose fibrils supply most of a fiber's tensile load-bearing ability and, consequently, high tensile strength. Therefore, the higher the cellulose content in a fiber, the higher the tensile strength and elastic modulus. Coir fibers outperform other plant-based fibers with superior elongation at failure due to their high microfibril angle and relatively low cellulose content, as shown in Table [1](#page-2-0). Apart from cellulose, coconut coir fibers also consist of hemicellulose and lignin. Coir fibers contain a high lignin content of about 40–45%, higher than cellulose (see Table [1](#page-2-0)). High lignin content in coir fibers results in better water resistance, subsequently increasing coir fibers' durability. Although the coir fibers show various promising properties due to the rich cellulose, hemicellulose, and lignin, the

Figure 1 Scanning electron microscope image of cross-sections of a typical coir fiber with lacuna and elementary fibers [\[12\]](#page-7-0)

shortcoming of the coir fibers is inevitable. Results carried out by scanning electron microscope analysis revealed that coconut coir fibers had porous surface structures [\[16\]](#page-8-0). Total porosity was measured to be about 30−50%, containing pit and tylosis, as shown in Figure 2. High porosity is not favorable and detrimental to the properties of coir fibers, resulting in higher water absorption than the other plant-based fibers [\[17](#page-8-0)].

Figure 2 Scanning electron microscope micrograph of the coir fiber surface showed pits and tyloses [\[16](#page-8-0)]

2.2. Mechanical properties of coconut coir fibers

Tensile strength has long been recognized as one of the most important properties of coir fibers. For instance, a fiber-reinforced polymer must be employed when coir fibers are utilized as reinforcement in composite structures. Coir fibers were observed to have a relatively high percentage elongation in the tensile test; hence, coir fibers are useful as reinforcement for composites that suffer large elastic deformations [\[18\]](#page-8-0). Several studies have stated the effect of alkaline treatment on the properties of coir fibers. Rout

et al. [\[19\]](#page-8-0) analyzed treated and untreated coir's mechanical and microscopic behavior and reported that the tensile strength of 10% NaOH-treated fiber was 259.7 N/mm², substantially greater than that of untreated fiber (i.e., 108 N/mm²). They also found that the impact strength of the untreated fibers increased dramatically from 433 J/m to 634 J/m after treatment with 5% alkali [\[19\]](#page-8-0). Chemical treatment of fibers can increase these composite qualities even further. Silva et al. [\[20\]](#page-8-0) investigated the untreated and alkaline-treated coir fibers for mechanical and thermal properties. The coir fibers were found to be oval-shaped after alkaline treatment, as shown in Figure 3, and to have increased mechanical characteristics, i.e., tensile strength from 76 ± 15 MPa to 94 \pm 12 MPa. A study by [[21](#page-8-0)] examined the effects of chemical treatment on the surface of coir fibers. They revealed that the NaOH treatment caused a rough surface. According to their findings, thinner coir fibers offer stronger tensile qualities than bigger ones. However, the fibers' diameter does not influence the percentage strain. Gu [\[22\]](#page-8-0) examined the tensile strength of heatpressed polypropylene and coir net composites after NaOH treatment of coir. It was found that the NaOH content must be less than 8% since a higher concentration might significantly reduce tensile strength.

Figure 3 Scanning electron microscope micrograph of coir fiber showed oval-shaped after being treated for 72 h in 5wt% NaOH [[20\]](#page-8-0)

Karthikeyan and Balamurugan [[23\]](#page-8-0) prepared the coir and epoxy composite. They examined the effect of NaOH treatment on composite impact strength and observed a 15% improvement. Duan et al. [[24\]](#page-8-0) investigated the untreated and 10% NaOH-treated fiber loading of coir, sisal, and polylactide hybrid composites. Tensile and flexural strengths were observed to initially decline and then slightly rise as the quantity of treated coir fiber in the polylactide matrix was increased. In comparison, Figure 4 shows that the tensile and flexural modulus rose as the proportion of sisal fiber increased. Measurements were made using 30% treated coir and 70% polylactide to determine the tensile strength (44 MPa), flexural strength (90 MPa), Young's modulus (1650 MPa), and flexural modulus (4400 MPa). Kumar et al. [\[3\]](#page-7-0) found that composites produced with 6% NaOH-treated coir fibers showed higher fracture toughness values and improved mechanical properties than sole polypropylene. The aforementioned works of literature show that the chemical treatment of the fibers improved the mechanical properties of the composite fabricated.

The length of the coir fibers also plays a crucial role in enhancing the properties. Long coir fibers may be a good choice for increasing the stiffness and strength of tropical soil. The fiber length considerably impacts the stiffness and strength of tropical

Figure 4

(a) Tensile modulus of PLA/USF (untreated sisal fibers) and PLA/ASF (alkali-treated sisal fibers) composites with different sisal fibers content and (b) flexural modulus of PLA/ PLA/USF (untreated sisal fibers) and PLA/ASF (alkali-treated sisal fibers) composites with different sisal fibers content [\[24](#page-8-0)]

soil, and long fibers were recommended [\[25](#page-8-0)]. Glycerol was used as a thermoplasticizer to prepare cassava starch-based coir fiber composite [\[26](#page-8-0)]. The mechanical characteristics of the composite were found to be greater than those of the matrix. Without treatment, the composite (with 30% fiber content) has a tensile strength of 1.56 MPa and Young's modulus of 14.56 MPa, whereas the annealed composite has a tensile strength of 3.24 MPa and Young's modulus of 59.81 MPa. A coir-based composite with a matrix of starch and ethylene vinyl alcohol was successfully fabricated by [\[27](#page-8-0)]. They found that adding NaOHtreated fibers greatly improved the composite's tensile strength and modulus. Some authors suggested that the architecture of coir fibers as reinforcement materials could change the properties of composites. Li et al. [[28\]](#page-8-0) looked into the various flexural characteristics of cement composites based on coir mesh. Flexural strength of the composites is increased by 44% with the addition of three layers of coir mesh, which makes up 1.8% of the overall weight. The results show that the inclusion of a starch mix and architectural layout can improve the characteristics of coir fibers. It also reported an increase of about 20% in flexural and tensile strength of the aligned coir fibers compared to the randomly

orientated fibers [[29](#page-8-0)]. The increased tensile and flexural strength in the aligned fibers compared to randomly orientated fibers could be attributed to the orientation of the aligned fibers, which eases the transfer of load between the matrix and fibers. The effect of fiber orientation on the mechanical properties was also reported by [[30\]](#page-8-0), in which the maximum flexural strength was found in the aligned fibers parallel to the loading direction (i.e., 0° angle). The finding was in accord with $[31]$ $[31]$ where the fibers cut at 0° angle showed the highest tensile and flexural strength compared to the fibers cut at 90° and chopped fibers. Geethamma et al. [[32\]](#page-8-0) found that an interlayer of rubber on the surface of the alkali-treated coir fibers increased the tensile strength of coir/rubber composites due to the good interfacial adhesion between the rubber and the increased surface area of the alkali-treated fibers. Table [2](#page-5-0) compares the architectures of fibers on the tensile and flexural strength of previous works. It is worth noting that incorporating particles into the porous surface of coir fibers and alteration of the composite's architecture could significantly enhance the properties.

2.3. Morphology and physical properties of Murraya koenigii

Plant extracts have gained a lot of interest lately due to their many benefits, including their wide availability, affordability, safety, lack of corrosiveness, and capacity to treat a variety of human ailments thanks to their different metabolites. Murraya koenigii originated from the Rutaceae family and is commonly known as curry leaves. They are employed as flavoring ingredients in culinary products and have a little spicy taste. Murraya koenigii is well-known for its antioxidant properties and range of therapeutic uses. Given that Murraya koenigii is among the most often produced solid wastes on a daily basis, it is essential to utilize it as a value-added substance. In this study, Murraya koenigii is suggested to be incorporated into coconut coir fibers as a filler for the porous surface of coir fibers. Results carried out by transmission electron microscopy characterization revealed that the formation of nanocomposites synthesized using Murraya koenigii had an average particle size of 18−22 nm [\[33\]](#page-8-0). These particles showed high absorption ability on the surface of nanomaterials, which is beneficial to smoothen the porous surface morphology of the materials. The average particle size of synthesized Murraya koenigii reported by previous studies lay between 10 and 20 nm [[7](#page-7-0), [9\]](#page-7-0). The small size of the nanoparticles is beneficial in imparting a high surface area, which increases the absorption resistance. Bhatt et al. [\[34\]](#page-8-0) found that Murraya koenigii exhibited substantial resistance to hydrolysis and the efficient ability to enhance the growth of almost all strains. Previous studies have stated that Murraya koenigii indicates excellent interaction energy for adsorption. Kumar and Yadav [\[35](#page-8-0)] revealed that Murraya koenigii had strong adsorption, as shown by the high electronegativity, binding, and adsorption energies. The kinetic studies revealed that Murraya koenigii-mediated magnetite nanoparticles had an increased binding affinity for the Murraya koenigii substances [\[36\]](#page-8-0). However, works of literature regarding the effect of incorporating Murraya koenigii as filler are limited and need to be explored.

3. Materials and Methods

3.1. Materials

Extraction of coconut coir fibers and Murraya koenigii was carried out after drying at a constant temperature of 200 °C in a furnace. The coconut coir fibers and Murraya koenigii were subjected to alkaline treatment in 6% NaOH solution at room

temperature (between 27 and 30 °C) for up to 24 h. Alkaline treatment using a 6% NaOH solution is chosen because various studies have reported the excellent mechanical properties of treated fibers using this percentage [[3](#page-7-0), [22,](#page-8-0) [23](#page-8-0), [37](#page-8-0)]. After the alkaline treatment, the coconut coir fibers and Murraya koenigii were rinsed in distilled water for 1 h to clean and remove the residual NaOH.

3.2. Surface morphology characterization of the fibers by field emission scanning electron microscopy (FESEM)

FESEM (model Jeol JSM-7900F) was used to produce high magnification surface morphology of the untreated, treated coir fibers, and treated fibers incorporated with Murraya koenigii. The accelerating voltage used during the scanning was 5 kV. FESEM is operated by producing electron beams (within an energy range of 0.01−30 kV) discharged from an electron source.

3.3. Thermogravimetric/differential scanning calorimetry (TGA/DSC) analysis

A TGA/DSC (Model of TGA/DSC 3+, METTLER TOLEDO) analysis was performed on the samples of the untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii particles to study the thermal behavior of the reinforcement. TGA/ DSC was performed at a heat rate of $0.2 \degree$ C/s, raising the temperature from room temperature to 600 $^{\circ}$ C in steps of 5 $^{\circ}$ C.

3.4. Fabrication of biocomposite samples

Following that, coconut coir fibers/Murraya koenigii particles reinforced polyethene resin matrix biocomposite were fabricated using compression molding. The mono-layer and tri-layer coir fibers incorporated with Murraya koenigii particle reinforcements were reinforced in the polyethene resin matrix, respectively. Various layers of reinforcement used in this study examine the effect of the reinforcement architecture on the properties of the biocomposites.

3.5. Tensile test (ASTM D638)

A tensile test referred to by the American Society for Testing and Materials (ASTM D638) was performed using a Computerized Universal Testing Machine (Model MT-02) to investigate the tensile strength of the samples at room temperature. The determination of characteristic values includes peak load, peak stress, strain peak stress, and tensile stress.

3.6. Flexural test (ASTM D790)

The 3-point flexural test (ASTM D790) is used to evaluate stiff and semi-rigid polymers and fiber composites, including long-fiber reinforcements. In this study, the flexural test was carried out using a Computerized Universal Testing Machine (Model MT-02) to examine force at break and maximum stress at sample break.

3.7. Rockwell hardness (ASTM D785)

ASTM D785 specifies many Rockwell hardness grades for plastic testing. Unlike ball indention hardness, which measures the indentation depth under load, the Rockwell method measures the indention depth at a predetermined preload. The biocomposite

sample was placed on the hardness tester's surface. After applying a minor load, the gauge was reset to zero. A lever was tripped to apply the major load. Within 15 s, the major load was removed. The hardness was determined using the dial with the minor load applied, allowing the sample to recover for 15 s.

3.8. Impact test (ASTM D256)

The impact test (ASTM D256) is a standard Izod impact test used to assess the resistance of material samples using a swinging hammer to strike and shatter a notched sample. Until the sample breaks, the standard test uses the dynamic energy required to cause a fracture. In this study, the samples were cut from fabricated biocomposites and fixed in the sample stage, while impact load was applied by releasing the pendulum. The load required to break the samples was recorded, and the procedure was repeated five times to ensure the consistency of the results.

4. Results and Discussion

4.1. Surface morphology characterization of the fibers

Before alkaline treatment, an uneven fiber surface consisting of globular particles was observed in the untreated fibers (see Figure 5). These globular particles could be the tyloses that cover the pits on the

surface of the fibers [[16\]](#page-8-0). Some impurities, such as debris, were also observed on the surface. After alkaline treatment, the impurities and lignin were removed, resulting in a rough surface, as shown in Figure 6. This could be attributed to the alkaline treatment that removed all the protrusions, leaving voids and pits on the surface of the coir fibers [[19,](#page-8-0) [38](#page-8-0)]. The treated fibers incorporated with Murraya koenigii showed rougher surfaces where impurities and debris were not seen (Figure 7). It has been known that a rough surface facilitates mechanical interlocking between fiber, matrix, and particles, which helps to create mechanical bonding between fiber, matrix, and particles.

Figure 6 Field emission scanning electron microscopy image of treated fiber surface at 500 magnification

4.2. Thermal stability

TGA measures the weight loss of the samples with increasing temperature. An increase in the weight loss of the samples means the material would have less resistance to heat. Figure [8](#page-6-0) shows the TGA graph of untreated fibers, treated fibers, and treated fiber incorporated with Murraya koenigii and polyethene. At higher temperatures, the treated fibers incorporated with Murraya koenigii had the least percentage of weight loss among the samples, indicating excellent thermal stability. Figure [9](#page-6-0) shows the DTG graph of untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii and polyethene. The degradation peak of treated fibers incorporated with Murraya koenigii is at 455 °C, the highest compared to the untreated fibers,

Figure 8 Thermogravimetric of untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii

Figure 9 DTG of untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii

treated fibers, and polyethene. This shows better thermal stability and excellent resistance to weight loss when heating. This is because of the improved interfacial bonding after the alkaline treatment and greater dispersion with the incorporation of Murraya koenigii particles.

4.3. Tensile and flexural strength

The tensile and flexural tests were repeated five times on each sample. Figure 10 shows the tensile and flexural strength of untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii and polyethene. The tri-layer treated fibers incorporated with Murraya koenigii biocomposites showed a higher tensile strength of 38.5 N/mm² than the mono-layer reinforcement (36.2) N/mm²). The treated and untreated fibers had a tensile strength of 32.8 N/mm² and 28.7 N/mm2 , respectively. The lowest tensile strength was found in polyethene at 27.0 N/mm².

The maximum flexural strength of 51.4 N/mm2 was found in the tri-layer treated fibers incorporated with Murraya koenigii biocomposites, followed by the mono-layer reinforcement, treated fibers, and the untreated fibers of 48.7 N/mm^2 , 41.3 N/mm^2 , and 40.1 N/mm2, respectively. Polyethene had the lowest flexural strength of 38.0 N/mm2 among the other biocomposites. The biocomposites incorporated with untreated and treated fibers showed higher tensile and flexural strength than polyethene. The tri-layer treated fibers incorporated with Murraya koenigii biocomposites exhibited the highest tensile and flexural strength among these samples.

4.4. Hardness and impact strength

Figure 11 shows the average hardness and impact strength of untreated fibers, treated fibers, and treated fibers incorporated with Murraya koenigii and polyethene. The tri-layer treated fibers incorporated with Murraya koenigii biocomposites showed the

Figure 11 Hardness and impact strength of untreated fibers, treated fibers, treated fibers incorporated with Murraya koenigii and polyethene

highest hardness value of 83.2 N/mm². The incorporation of fibers into polyethene was found to increase the hardness. It was also observed that the samples incorporated with treated fibers have higher impact strength, followed by the untreated fibers and polyethene. The impact strength of the tri-layer treated fibers incorporated with Murraya koenigii biocomposites is 36.8 J/m, higher than the mono-layer reinforcement biocomposites. The superior hardness and impact strength of the tri-layer treated fibers incorporated with Murraya koenigii biocomposites could be attributed to the synergistic effect of coir fibers/ Murraya koenigii reinforcement and the layering architecture. The dispersed coir fibers were reported to create more interfaces, inhibiting the crack propagation at the fiber interface to the matrix [[39\]](#page-8-0).

5. Conclusion

Treated coir fibers demonstrate improved thermal stability at elevated temperatures, surpassing untreated fibers and polyethene. Incorporating Murraya koenigii particles into the fibers further elevates the thermal resistance, offering a robust response to thermal stress. The tri-layer reinforcement design of the treated fibers, incorporating Murraya koenigii, distinctly outperforms the mono-layer reinforcement. This architecture yields superior tensile and flexural strength, hardness, and impact strength, significantly enhancing the mechanical properties of the biocomposites.

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflicts of Interest

The authors declare that they have no conflicts of interest to this work.

Data Availability Statement

Data sharing does not apply to this article as no new data were created or analyzed in this study.

Author Contribution Statement

Hooi Peng Lim: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Resources, Writing – original draft, Writing – review $\&$ editing, Visualization, Project administration. Salim Bachok: Methodology, Writing – review $\&$ editing, Visualization. Halimatul Sa'diah Talib: Methodology, Writing – review & editing, Visualization.

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