The development of technology has increased the need for composite materials, where the technology of composite materials with natural fiber reinforcement is growing. The existence of natural fiber is very abundant, and it has not been fully utilized. Until now, the use of coconut fiber was still limited to the furniture and household handicraft industries. Coconut coir fiber has the potential as a raw material for composite materials. The lack of strength of the bonds is due to the hydrophobic fiber, and the fiber surface is less rough, and dirty. This study evaluates the coir surface characteristic of the fiber and its bounding with the polyester matrix after being treated by limestone water. The scanning electron microscope was used for observing fiber surfaces and surface matrix. The wettability test to observe fiber surface energy was performed. Interface shear strength to evaluate the bonds between fibers and matrix was determined. Coconut coir fibers were immersed in limestone water, with a 5 % percentage of limestone and time variations of 0, 4, 8, 12, 16, and 20 hours. The scanning microscope electron observations of fibers show that the fiber surface tends to be clean, rough, and grooved. The highest surface energy was obtained at 40.74 mN/m during the limestone water immersion for 8 hours. The highest value of the interface shear strength between the fiber and the matrix is 3.80 MPa during 8-hour immersion, 0, 4, 12, 16, and 20-hour immersion, respectively, 3.02, 3.09, 3.52, 3.47, and 4.40 MPa. The results showed that coir fiber with limestone water immersion for 8 hours had a clean, rough, and grooved surface so that the bond between the fiber and matrix was better. This research shows that limestone water can be used as a fiber treatment medium which was natural

Keywords: limestone, coconut fiber, immersion, surface fiber, wettability, interfacial shear strength

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EFFECT OF COCONUT FIBER TREATMENT WITH LIMESTONE WATER MEDIA ON THE FIBER SURFACE, WETTABILITY, AND INTERFACE SHEAR STRENGTH

Sutrisno
Doctoral Student in Mechanical Engineering*
E-mail: sutrisno@unmer-madiun.ac.id
Rudy Soenoko
Professor in Mechanical Engineering*
E-mail: rudysoen@ub.ac.id
Yudy Surya Irawan
Doctor of Engineering, Assistant Professor*
E-mail: yudysir@ub.ac.id
Teguh Dwi Widodo
Doctor of Engineering, Assistant Professor*
E-mail: widodoteguhdwi@ub.ac.id
*Department of Mechanical Engineering
Brawijaya University
Jl. Mayjend Haryono, 167, Malang, Indonesia, 65145

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1. Introduction

Currently, the technical materials used in the industry are still very dependent on metals, but other engineering materials are starting to be considered substitutes for metal. The engineering materials that will be developed are composite materials. Composite materials consist of two types, namely particle composite materials, and fiber composite materials. Fiber composite material consists of fibers bound by a matrix; there are two kinds of forms, namely long fibers and short fibers. Fiber composites in the industrial world began to be developed instead of using particle materials. Fiber composite materials have the main advantages of being healthy, challenging, and resistant to heat inside the matrix [1]. Fiber composite materials have many advantages, including light weight, higher strength, corrosion resistance, and lower assembly costs due to a reduced number of components and connecting bolts [2].

Some of the composite parts are reinforcement that serves as the primary load bearer on the composite. Fiber generally consists of two types, namely natural fibers, and synthetic fibers. Natural fibers are fibers that can be obtained directly from nature, usually fibers that can be obtained directly from plants and animals. These fibers have been widely used, including cotton, wool, silk, banana fronds, coconut husk, palm fiber, bamboo, pineapple and, kenaf, or burlap. Natural fibers have a weakness; namely, the size of the fiber is not uniform; the strength of the fiber is greatly influenced by age [3]. The existence of coconut trees in Indonesia is very abundant [4].

Coconut fruit consists of fruit and skin, whereas coconut skin consists of fiber and flesh. Coconut coir fiber contains three main elements, they are cellulose, hemicellulose, and lignin. Lignin is a fiber impurity element, which causes it to be difficult to bond automatically. The coconut coir fibers need to be treated to reduce the lignin content of the fibers and have good quality [5]. Chemical treatment can include central degradation of the fiber, which can result in surface damage, thereby decreasing the mechanical properties of the thread. As a result, there is a need for a treatment that can clean the thread without destructing the cellulose.

The characteristics of natural fibers are pretentious by the processing and treatment of textures. Some of the treatments used can comprise chemical treatment or natural treatment with natural substances, and some chemical treatments contain alkali treatment [2, 5, 6].

Therefore, studies devoted to determining the effect of limestone water immersion on the properties of coconut fiber are of scientific relevance.

2. Literature review and problem statement

The mechanical properties of the composite material are influenced by several things, including bonding with the matrix. The bond between the fibers and the matrix is influenced by several factors, including the type of matrix, the type of fiber (fiber content and fiber surface). Several studies have used natural ingredients (sea water) and chemical sodium hydroxide (NaOH) and aminoethyl aminopropyl tri-methoxy silane (AAMS) as fiber processing media.

The main content of coconut fibers is cellulose, hemicellulose, and lignin [6, 7]. The strength or ductility of the composite can be obtained by increasing the bond between the fiber surface and the matrix. Composite properties are determined by the compatibility of natural fibers with the matrix. Natural fibers have hydrophilic properties, the surface of natural fibers also has impurities and other substances that can affect the strength of the fiber bond with the matrix. Treatment of natural fibers with chemicals with NaOH for 3 hours with a concentration of 5, 10, 15, and 20 % resulted in a rough fiber surface, decreased crystallinity index and increased bonding between fibers and matrices [8].

Increasing the mechanical properties of natural fibers in the manufacture of composites, alkaline treatment and heat treatment of kenaf fiber were carried out. Compared with other heat treatment temperatures, the tensile strength of kenaf fiber at 140 °C reaches the maximum value, which can be attributed to the increase in the crystallinity index of the fiber after heat treatment. The results showed that the fracture strain treated with alkaline was better. The treatment then causes a decrease in the crystallinity and loosely bound structure of the fibers. This treatment results in a higher elongation at break in the treated fiber [9].

Treatment of elephant fiber with NaOH solution (weight fraction composition of 2 % and 5 %). The elephant grass fibers were analyzed by chemical methods, FTIR, and 13 C NMR solid-state. NaOH solution and the effect of alkaline treatment on the composition and structure of the fibers were studied. The alkaline treatment removes the amorphous hemicellulose component from the fiber to a greater extent. Fiber morphology before and after alkaline treatment was observed by a scanning electron microscope. The fibers become thin with a rough surface, and the cell structure collapses after the alkaline treatment and shows higher tensile properties [10].

Cellulose fibers have significant importance and potential for polymer reinforcement. It was very important to modify the fiber surface to get an excellent fiber-matrix interface. Surface treatment can increase the surface roughness of the fibers, change their chemical composition, and introduce new parts that can effectively interlock with the matrix, resulting in good mechanical properties of the composites. This was mainly due to the increased adhesion of the fiber matrix. The treatment can also reduce the water absorption rate by converting the hydroxyl groups on the fiber surface into other functional groups. Chemical modification of the surface of the regenerated cellulosic fibers of the lyocell type was carried out by alkaline and silane treatment, which significantly changed the properties of the lyocell fibers. Three parameters were considered when fiber surface treatment was performed: concentration (2–15 wt %), temperature (25 °C and 50 °C) and time (30 min–72 hours). Fourier transform infrared spectroscopy, and spectroscopy were used for chemical analysis and qualitative analysis of cellulose crystallinity due to surface treatment. The mechanical strength of the fiber is tested by a tensile test. Mass reduction, moisture recovery and fiber measurements were carried out before and after treatment, which showed significant changes in fiber properties during treatment. The fiber heat capacity is measured for untreated and treated fibers, and the thermal degradation of the fibers was checked for fiber stability at high temperatures. The increased wetness and surface energy were measured by the dynamic contact angle method on three wetting media. Scanning electron microscopy (SEM) was used to study the morphological properties of fibers [11].

The fiber from the Cissus quadrangular plant and all parts of this plant can be used in many applications. The textile industry uses a lot of plant fibers for various applications obtained from many resources. The advantages of natural fibers were their sustainable supply, easy and safe handling, and their biodegradability. The use of enzymes in the textile industry approves the development of environmentally friendly technologies in fiber processing and tactics to improve the quality of the final product. Natural cellulose fibers were extracted from the Cissus quadrangular plant using an environmentally friendly method (amylase enzyme). Physico-chemical, thermal and mechanical properties of Cissus quadrangular fibers have been reported in this paper. Furthermore, the property of CQSF ensures that CQSF can play a significant role in the material manufacturing industry [12].

Cornhusk fiber has been extracted by the alkalization process at different alkaline concentrations and treatment times. The effect of extraction process parameters on the physical properties, mechanical properties and thermal resistance characteristics of the corn husk fiber was investigated. The chemical structure of the fibers was studied by measuring infrared spectroscopy. The average length, linear density and moisture content of the extracted fibers decreased with increasing concentration and duration of bases. The breaking force decreases with increasing base concentration. Increasing the treatment duration for all concentration levels increases the breaking force, ductility, and initial modulus up to a point and then decreases it for a longer duration. The highest tensile performance can be obtained from 5–10 g/L of NaOH treatment for 60–90 minutes. Alkalization under harsher conditions results in higher thermal resistance up to 320 °C with a higher cellulose fraction, but lower resistance above this temperature. FTIR spectrum analysis proved higher cellulose but lower hemicellulose and lignin content under harsher treatment conditions. The marginal effect of the concentration decreases for higher concentrations, which indicates that the sites available for the chemical reaction were occupied at a moderate level of concentration [13].

The effect of chemical immersion treatment on the properties of the cantal fiber and the quality of the bonding interface of the cantal fiber and recycled high-density polyethene. The cantal fiber was treated by alkaline, silane and a combination of the two. The results showed that the loss of hemicellulose and lignin after alkaline treatment, and the presence of a silane layer on the fiber surface after silane or alkali-silane treatment, increased thermal stability, surface energy, and shear stress. The highest surface energy

of 45.37 mN/m was obtained during the alkaline treatment with a content of 2 % NaOH. Alkali-silane treatment with 0.75 wt % fraction of silane [14].

NaOH treatment on corn husk fiber with (0.5 %, 1 %, 2 %, 5 %, and 8 %) for 2 hours resulted in reduced hemicellulose and lignin and decreased water content. SEM analysis showed the presence of a rough surface and a number of lumens in the fiber bundle. Processed corn fiber shows better mechanical properties than fiberglass [15].

Mendong fiber research to determine morphology, structure, and chemistry. Physical properties of mendong fiber extracted from mendong grass (Fimbristylis globulosa) in the form of raw fibers and processed by chemical compounds including alkalis and functional groups and to evaluate the strength and properties of mendong fibers compared to other natural fibers. Research on mendong fiber shows that the chemical content of mendong fiber is 72.14 % cellulose, 20.2 % hemicellulose, 3.44 % lignin, 4.2 % extractive, and 4.2 %–5.2 % moisture. The mechanical properties of the fiber are of a strong character with a tensile strength of 452 MPa, and a modulus of 17 GPa. The structural properties of mendong fibers include crystallinity, crystal index, microfiber angle, and crystal size, which are 70.17 % and 58.6 %, 22.9º, and 14.3 nm. This fiber has a competitive advantage compared to other fibrous natural fibers and can be further developed as a potential strengthening of polymer matrix composites [16, 17].

The bond between the fiber and the matrix will affect its mechanical properties, where its characteristics involve fiber wetness (wettability). The wettability parameter, among others, is determined by the contact angle formed between the matrix and the fiber surface and the interfacial bonding. The adhesion between the fiber as reinforcement and the matrix greatly affects the mechanical properties of the resulting composite material [18–20]. Shear stress is obtained by modifying the chemical properties of the fiber surface to optimize the adhesion properties between the fiber and matrix [17, 21].

Characterization of pineapple leaf and coconut coir fibers for possible applications in composites. In this study, a shear stress test was carried out to compare the interface adhesion with the epoxy resin of these two fibers which have very different characteristics. The results showed a critical length of 70 % higher for coir fiber compared to pineapple leaf fiber and 3.5 times less interface strength, which indicates stronger adhesion of pineapple leaf fiber with epoxy resin. This can be confirmed by the different morphological aspects, in particular the rougher surface of the pineapple leaf fibers. Mechanical tests were carried out on a composite of coconut fiber and pineapple leaf fibers. The results show a relatively low penetration depth (18.2 mm) for coconut coir with pineapple leaf fiber composite and penetration depth (31.6 mm), both of which are considered efficient according to body protection standards [22].

Some of these papers present the results of research on fiber treatment, and the results show that treatment with chemicals produces fiber surfaces that experience excessive degradation and cause environmental pollution. Several previous studies used natural media for processing natural fibers; these media include seawater, fumigation, and vacuum processes. From several fiber processing media, it is necessary to obtain a fiber processing medium that is readily available and the availability of lots of materials. As a result, there is a need for a treatment that can fresh the line without damaging the cellulose. Natural treatment is needed to maintain the fiber surface and cellulose content.

3. The aim and objectives of the study

The aim of the study is to determine the effect of fiber immersion in limestone water on the fiber surface, wettability, and interface shear stress.

To achieve the aim, the following objectives were set:

– to determine the surface morphology of the fiber by using SEM testing;

– to determine the level of fiber wetness by testing the wettability on ASTM D7334 – 08 (2013);

– to determine the shear stress by performing a pull-out with the ASTM D5321/D5321M – 20.

4. Materials and methods to study the effect of coconut fiber treatment with limestone water media on the fiber surface, wettability, and interface shear strength

Materials:

1. Coconut Fiber.

Coconut coir was obtained from coconut farmers in Wonogiri Regency, Central Java, Indonesia. Coconut coir fiber was obtained by separating the fiber from the cork; the fiber was taken in the middle. The chemical solution used was limestone dissolved in distilled water. The limestone composition is 5 %, with variations in immersion time of 0, 4, 8, 12, 16, and 20 hours.

The variation of coconut coir treatment with variations in soaking time (0, 4, 8, 12, 16, and 20 hours), and constant variation: the mass of limestone 5 % of the fixed fraction is shown in Table 1.

Table 1

Coconut fiber treatments

No.	Notation	Immersion time (hours)	Limestone (% wt)
1	UT	0	5
2	IT4H	4	5
3	IT8H	8	5
4	IT12H	12	5
5	IT16H	16	5
6	IT20H	20	5

Notation:

– UT: Untreated specimen;

– IT4H: Immersion time of 4 hours;

– IT8H: Immersion time of 8 hours;

– IT8H: Immersion time of 12 hours;

– IT16H: Immersion time of 16 hours;

– IT20H: Immersion time of 20 hours.

Coconut fiber should be cleaned of meat and other contaminants. Coconut coir fiber in clean conditions and the immersion process of coir fiber can be seen in Fig. 1.

The coir fibers that have been immersed were washed with distilled water and dried at room temperature. The fiber wettability test was carried out by arranging the fibers in a U profile (10 fibers each). Observations were made using a microscope. The shear strength test was carried out by planting the fibers in the BQTN polyester matrix and then placing the embedded fibers on the cardboard using glue. Observation of the fiber surface was carried out with an electron microscope photo.

a *b*

Fig. 1. The main signature: *a* — coconut coir fiber; *b* — coconut coir fibers soaked in a solution of limestone water

2. Limestone water.

Limestone water was obtained by dissolving limestone in distilled water. The existence of limestone is very abundant and has a very abundant supply in the Java region, Indonesia.

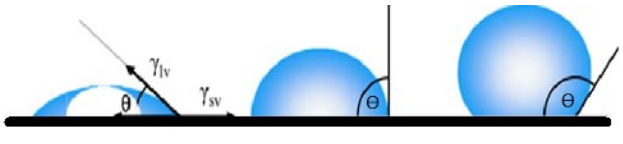
3. Polyester resin BQTN.

The polyester resin was a low viscosity liquid resin, hardens at room temperature by using a catalyst without generating gas during testing, as many other resins. The main additive is a catalyst, a type of motivation for polyester resin, namely Methyl Ethyl Ketone Peroxide (MEKPO). The trigger serves to speed up the process of resin liquid hardening.

Methodology:

– Wettability (Wettability of fiber).

Among others, the wettability parameter is determined by the contact angle formed between the matrix and the fiber surface and the interfacial bonding. A quantitative measure involving the contact angle (θ) between the fiber-matrix surfaces, namely giving a liquid that is placed on a solid surface, is shown in Fig. 2.



a *b* *c*

Fig. 2. The main signature illustration of the contact angle formed by liquid droplets on a surface: *a* — θ<90°; *b* — θ=90°; *c* — θ>90° [9]

The smaller the contact angle, the better the wettability, so that the matrix as a fiber adhesive medium must have the ability to cover the surface area of the fiber optimally. The contact angle for optimum wetting is not more than 30°. Wettability is indicated by the contact angle (θ) between the solid fiber and the liquid matrix in the form of droplets [23].

Shrink contact between matrix and fiber to observe the amount of surface energy. Surface energy occurs between the fibers and the matrix to measure their bond. Surface energy consists of polar and dispersive components [24]. The polar component occurs due to the interaction of dipolar and dispersive due to the Van Der Waals forces between the material molecules [25]. Surface energy is measured by calculating the contact angle of polar and non-polar fluids on the surface of the fiber. Polar and non-polar fluids are distilled water and ethylene glycol.

– Interfacial shear strength (pull out).

The fibers are embedded in a matrix and are pulled apart, as shown in the figure. The interfacial shear strength is highly dependent on the quality of the bonds between the fibers/matrix.

The schematic of the shear stress test for coconut coir grown in a matrix with a depth of 3 mm is shown in Fig. 3.

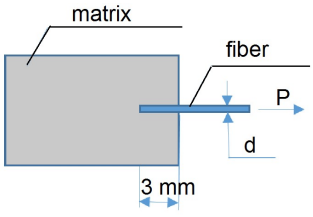


Fig. 3. Interfacial shear strength mechanism [26]

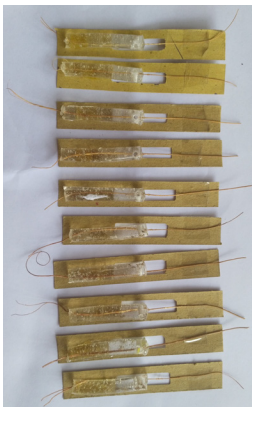


Fig. 4. Samples of interfacial shear strength

The longitudinal tensile force on the fibers will produce shear in the fiber/matrix interface. When the fibers are pulled apart from the matrix, the interfacial shear strength occurs between the fibers/matrix.

5. Results of the effect of surface modification of coconut fibers on wettability and interface shear strength with the matrix

5. 1. Results of the fiber surface morphology

The results of the fiber content test showed that the cellulose content in the process of immersing the fibers in the limestone water solution for 8 hours was 36.99 %. These results have a higher percentage value compared to the pre-treatment and other immersion processes.

The chemical composition of coconut coir before and after treatment is presented in Table 2.

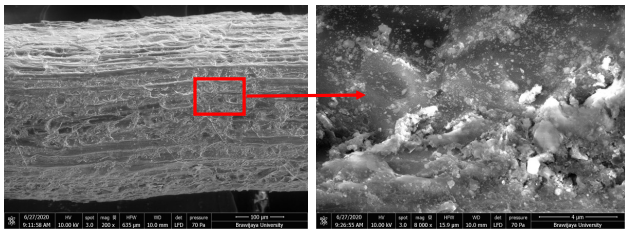
Table 2

The constituent content of fiber (wt %) present in the treated fibers

Notation	Immersion Time	Composition (weight)		
		Hemicellullose (%)	Alfa Cellullose (%)	Lignin (%)
UT	Untreated specimen	66.31	33.89	35.72
T4H	4 Hours	60.05	34.27	36.24
T8H	8 Hours	60.76	36.99	34.84
T12H	12 Hours	59.23	32.06	33.40
T16H	16 Hours	59.59	23.44	34.80
T20H	20 Hours	57.38	22.65	34.25

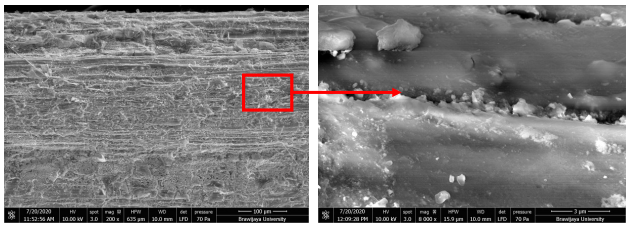
The SEM test results of the coconut coir fiber surface, the fiber by soaking for 8 hours showed the results of the fiber surface that was cleaner. The fiber surface is 8 hours cleaner than the untreated and other treated fibers.

The surface morphology of the fibers before and after the immersion treatment is shown in Fig. 5–10.



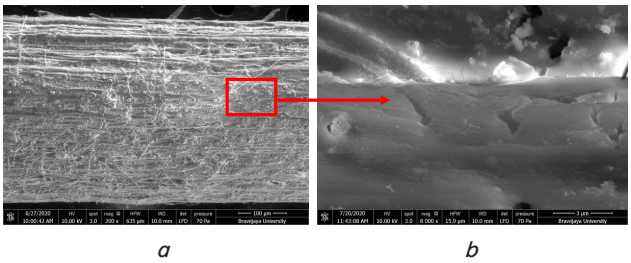
a *b*

Fig. 5. The main signature indicates UT: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections



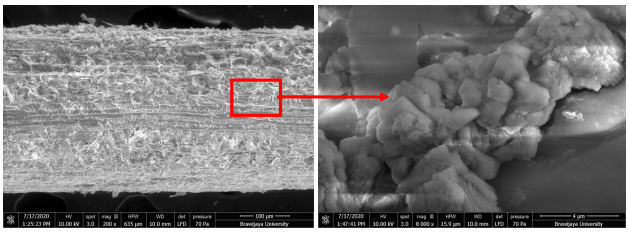
a *b*

Fig. 6. The main signature indicates T4H: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections



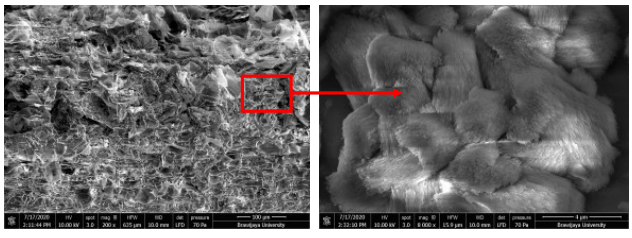
a *b*

Fig. 7. The main signature indicates T8H: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections



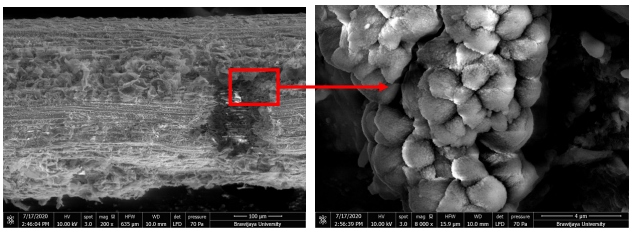
a *b*

Fig. 8. The main signature indicates T12H: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections



a *b*

Fig. 9. The main signature indicates T16H: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections



a *b*

Fig. 10. The main signature indicates T20H: *a* — coconut coir surfaces; *b* — SEM of the heterogeneities of the coconut fiber cross-sections

5. 2. Results of testing the wettability and surface energy of the fiber

The results of the wetness test showed that the fiber with the 8-hour treatment showed the smallest contact angle when compared to the untreated one and other treatments. In this treatment, it has a high surface energy, namely 40.74 mN/m, which is higher when compared to without treatment and other treatments. The results of the overall wetness test are shown in Table 3.

Table 3

Test results for contact angle and surface energy

Notation	Angle of contact		Dis-persion (mN/m)	Polarity (mN/m)	Surface Energy (mN/m)
	distilled water	ethylene glycol			
UT	71.89	70.76	1.21	35.56	36.77
T4H	69.37	68.23	1.27	37.90	39.17
T8H	68.54	68.23	1.02	39.72	40.74
T12H	69.67	69.46	0.96	38.78	39.74
T16H	70.32	69.76	1.05	37.73	38.78
T20H	70.43	70.21	0.95	38.05	38.99

5. 3. Results of the interfacial shear stress

Fig. 11 shows the results of the interfacial shear stress test. The results of the fiber shear stress test embedded in the matrix show that the fibers with an 8-hour immersion treatment have shear stress of 3.80 MPa. Fig. 14 shows the SEM of the fibers detached from the matrix. The image shows some of the fibers still attached to the matrix.

Fig. 12–17 show the Scanning Electron Microscopy (SEM) results of the fibers detached from the matrix.

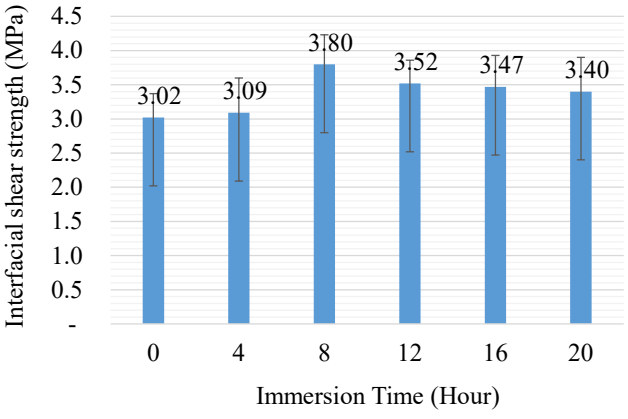
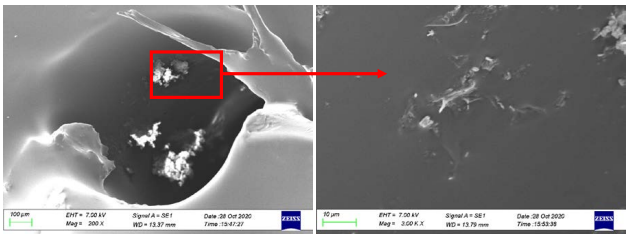
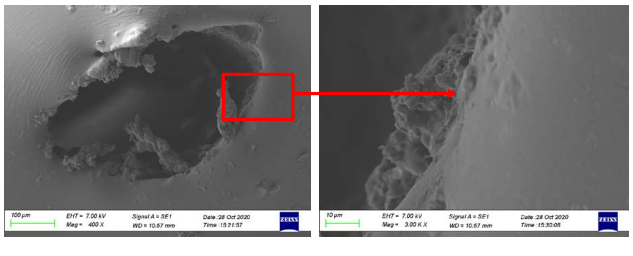


Fig. 11. The relationship between immersion time and interfacial shear strength



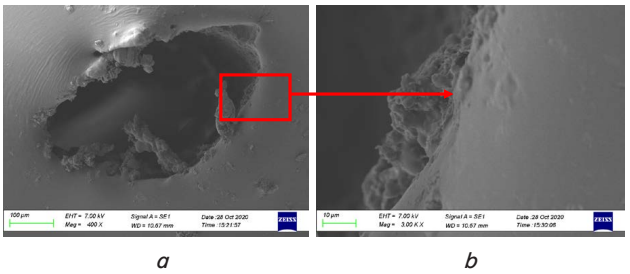
a *b*

Fig. 12. The main signature SEM coconut coir fibers detached from the matrix UT: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber



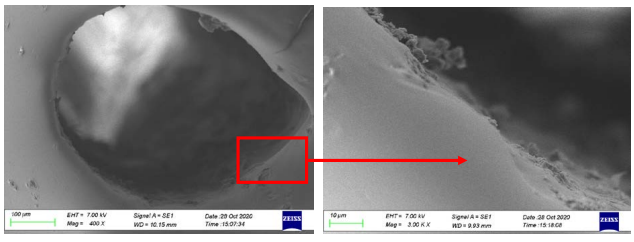
a *b*

Fig. 13. The main signature SEM coconut coir fibers detached from the matrix T4H: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber



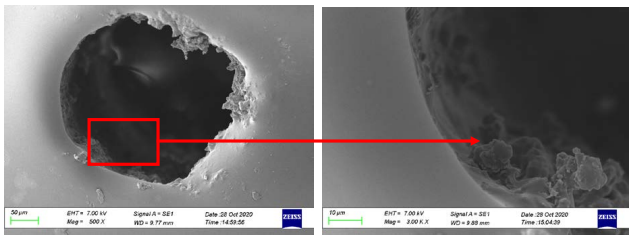
a *b*

Fig. 14. The main signature SEM coconut coir fibers detached from the matrix T8H: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber



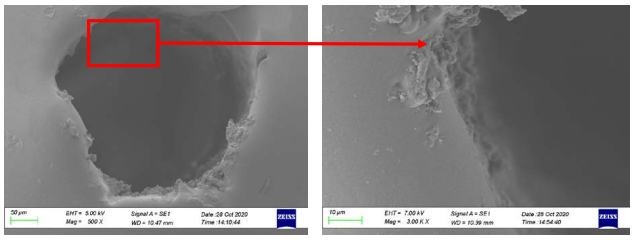
a *b*

Fig. 15. The main signature SEM coconut coir fibers detached from the matrix T12H: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber



a *b*

Fig. 16. The main signature SEM coconut coir fibers detached from the matrix T16H: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber



a *b*

Fig. 17. The main signature SEM coconut coir fibers detached from the matrix T20H: *a* — surface matrix of coconut fiber loose holes; *b* — surface of the heterogeneities cross-sections of the matrix and coconut fiber

Fig. 12 shows a clean-looking lint hole in the matrix; this indicates that the fiber is not well bound by the matrix. Fig. 13, 14 show a fibrous hole in a fibrous matrix; this shows that the fiber and matrix bonds occur well. Fig. 15–17 show the grain marks in the matrix which looks clean; this shows the fiber and matrix bonds are not good.

6. Discussion of the effect of surface modification of coconut fibers on wettability and interface shear strength with the matrix

The results showed that the effect of immersing coconut coir in limestone water could reduce the content of lignin, clean the surface/increase the roughness of the fibers, increase the level of wetness, and increase the interface shear stress. Limestone water as a fiber processing

medium has added value, which is more environmentally friendly and more economical when compared to other studies [11, 14–16, 19, 21, 27] that use chemicals. The significant result of this research is the use of limestone water which can be used as a natural (environmentally friendly) fiber treatment medium, so that it can be used as fiber treatment that often uses chemical media.

The compositions of coir fibers that have and have not been treated with limestone water immersion have different chemical contents, as shown in Table 1. Fibers that are treated with limestone water immersion for 8 hours have the highest alpha-cellulose value (36.99 %). The cellulose content in the 8-hour treatment increased, because the hemicellulose and lignin content was eroded by the limestone water solution, so that the percentage of hemicellulose and lignin decreased. This research was following the research that sodium hydroxide treatment can remove non-cellulose components [5, 28–30].

The surface of the treated and untreated fiber surface is shown in Fig. 5–10, whereas with chemical treatment the immersion times of 4, 8, 12, 16, and 20 hours. Fig. 5 shows the untreated fiber surface, the picture shows the presence of dirt, which is still attached to the fiber surface. The fiber surface indicates that the hemicellulose and lignin compositions are still attached to the fiber surface. In Fig. 7, fiber was immersed for 8 hours, the surface of the fiber looks clean, grooved regularly and rough. Fig. 7 shows that the lignin and hemicellulose composition has been eroded from the fiber surface [29, 30].

In Fig. 8–10, the fiber surface starts to look rough but grooved irregularly. The rough surface is caused by the cellulose component being eroded by the limestone water. The surface of the coconut fiber appears to be damaged, degraded by the time of immersion in limestone water.

The value of matrix and fiber adhesion can be seen from the wettability of the fibers. The wetting of the fibers can be determined by calculating the surface energy. Table 3 shows the contact angle and surface energy for untreated and treated cantile fiber. In water and ethylene glycol, untreated fibers have a higher contact angle with lower surface energy compared to treated fibers. This indicates that the UF is hydrophobic with low polarity and is difficult to wet. The presence of non-polar materials, such as wax, oil, pectin, and lignin, on the fiber surface causes UF to become hydrophobic.

The removal of the T8H lignin component caused an increase in polarity from 35.44 to 43.47 mN/m and at surface energies from 39.17 to 40.74 mN/m. Similar cases were found in hemp fibers [31]. Limestone water makes the surface of the fiber clean, and the surface energy increases. The increase was caused by the reaction between limestone water and coconut fiber. This phenomenon was under the results reported by [32]. The highest surface energy of 45.37 mN/m was obtained after the limestone water immersion treatment.

The results of the interfacial shear strength test of coconut coir fibers with a polyester matrix are shown in Fig. 11. The interfacial shear strength values of the fibers without treatment were 3.02 MPa, while those treated for 4 hours and 8 hours were 3.09 MPa and 3.80 MPa, respectively. The interfacial shear strength of the fibers decreased in the variation of immersion time for 12 hours, 16 hours, and 20 hours, namely 3.52 MPa, 3.47 MPa, and 3.40 MPa.

The 8-hour immersion variation has the maximum interfacial shear strength; this is due to the clean fiber surface, reduced lignin content. So that the bond between the fiber and matrix can be maximized, as shown in Fig. 11. Variations of 12 hours and so on experienced a decrease in interfacial shear strength, this was due to the sizeable degraded fiber surface. Degradation of fibers due to the immersion of limestone water for too long causes the fiber surface to be damaged and the cellulose content also decreases [33–35].

Fig. 12 results from the interfacial shear strength of the untreated fibers, showing a clean trace matrix. Fig. 12 shows that the fibers separated from the matrix leave no marks. Fig. 12 shows that the fibers and automatic bonding are not good enough. Fig. 13, 14 show the fibers that are released in the matrix better than in Fig. 12. The fibers that were released from the matrix appear as fibers left behind and the holes in the matrix look uneven, this indicates that the fibers and matrix bonding can occur properly. Other research have also shown that good adhesion between matrix and fiber occurs when the interface shear test occurs when the fiber surface is attached to the matrix [22].

Fig. 15–17 show the interface shear marks of the fiber and matrix. Fig. 15–17 show the surface of the fiber left on the polyester matrix, each showing the surface of the fibers left in the matrix is not as much as in Fig. 14. Fig. 15–17 show the ability of the matrix to bind to the fiber is not as good as in Fig. 14. Fiber surface has begun to break down and the cellulose content has decreased. Fiber that is too long soaked in a solution of lime water causes the fiber surface to become damaged.

Some of the problems in this study are the limitations of laboratory use in connection with the Covid 19 pandemic. We, as researchers, try hard to obtain data as a result of an investigation. The results of this research are the result of the hard work of the research team to improve knowledge, especially materials science.

Several things that make the disadvantages in this study are the selection of the starting material (coconut fiber) which has a variety of sizes and types. We hope that before carrying out the research process, the preparation of fiber types will approximately have the same diversity. This was to save laboratory costs which may be quite expensive. Research with a more thorough and systematic preparation will produce a better study.

Hopefully, in the future with this research, to produce useful data, it is necessary to add a simulation method or a mathematical method so that it can support this research using the experimental procedure. Some suggestions from the research team, before carrying out empirical research, it is better to do research using simulation methods. The data obtained from the simulation and experimental results can be compiled so as to produce good research.

The results of this research can be applied in the composite manufacturing industry. Coconut coir fiber, which has good properties, can be used as reinforcement for composite materials. Composite materials with natural fiber reinforcement have a huge opportunity. There is a need for materials (mostly composite materials) as industrial support.

7. Conclusions

1. Chemical analysis at 8 hours immersion content of cellulose has the highest percentage, namely 36.99 % when compared to other submerged and untreated fibers. This was because limestone water can clean the dirt that sticks to the

surface of the coconut coir fibers. Lignin and hemicellulose are degraded by limestone water.

2. After 8 hours of immersion, the wettability of the fibers increases, this was because the surface of the fibers was clean, rough and grooved. The matrix can penetrate inside, which causes the bond between the matrix and the fiber to be better.

3. The interfacial shear stress between the matrix and fibers increases. Coconut coir fiber has excellent compatibility with polyester resin, because of the Pull Out test results and fiber density and matrix. The fiber immersed in lime water for 8 hours has a shear stress value of 3.80 MPa, because the fiber for 8 hours of immersion has a good bond between the fiber and resin, according to the results of the scanning electron microscope. The immersion of the fibers in limestone water indicates the presence of resin that enters the fiber or the fiber surface can be firmly bound to the matrix.

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