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Effects of alkali solution concentration and soaking time on mechanical properties of coconut fibre

Y. Kondo [a], M. Arsyad [a,*], Ahmad [a], R. Soenoko [b], Arman [a]

[a] Mechanical Engineering Department, Politeknik Negeri Ujung Pandang, Jalan Perintis Kemerdekaan KM 10 Tamalanrea Makassar 90245, South Sulawesi, Indonesia
[b] Mechanical Engineering Department, Universitas Brawajiya, Jalan Jenderal M.T. Haryono Malang 65145, East Java, Indonesia
* Corresponding e-mail address: arsyadhabe@poliupg.ac.id
ORCID identifier: https://orcid.org/0000-0002-3764-5713 (M.A.)

ABSTRACT

Purpose: The study aims to determine the effect of the treatment of alkali solution concentration and soaking time on the mechanical properties of coconut fibre.

Design/methodology/approach: The study consists of preparing materials and equipment, immersion of coconut fibre in an alkali solution, drying in a furnace, testing, analysis of test results, and conclusions. Materials and equipment used are coconut fibre, alkali solution, polyester matrix, distilled water, furnace, hydrolysis test, tensile test, and SEM analysis. The sample had two treatments; the first was coconut fibre, which was soaked in the sodium hydroxide solution with 5%, 10%, 15%, and 20% concentrations for 3 hours. The second treatment was coconut fibre soaked in the sodium hydroxide solution with a concentration of 20% for 1, 5, 7, 9, and 11 hours. The samples were then dried in a furnace at 90ºC for 5 hours, and then a hydrolysis test, tensile test, pull-out test, and SEM analysis were carried out.

Findings: The results suggest that for immersion in an alkali solution of 20%, the highest tensile strength of coconut coir fibre was obtained in soaking for 3 hours at 280.94 N/mm^2, and the highest bonding strength between coconut coir fibres with a matrix polyester was obtained at 5 hours immersion at 7.86 N/mm^2.

Research limitations/implications: In the given study, coconut fibre was treated by soaking it in 5%, 10%, 15%, and 20% sodium hydroxide solution. Then, a single fibre tensile test was carried out, and a pull-out test was carried out to determine the mechanical properties of coconut fibre as a required effect that had been given. Subsequent studies can be carried out with other treatments using other chemical solutions, such as hydrogen peroxide or potassium permanganate.

Originality/value: The tensile strength of coconut fibre without treatment was 186.42 N/mm^2, whereas after being immersed in 20% sodium hydroxide solution, the tensile strength became 280.94 N/mm^2. Likewise, the shear strength of the interface between the fibre and the polyester matrix was 1.85 N/mm^2 for untreated coconut fibre to 3.09 N/mm^2 for coconut fibre soaked in a 20% sodium hydroxide solution. The results of the study are intended as data for the use of coconut fibre as a natural fibre-reinforced composite material, for example, as a raw material for fishing boat walls.

Keywords: Coir, Soaking, Alkali, Interfacial, Shear

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1. Introduction

Development in science and technology has caused the use of materials to increase as well, so engineering in the material field must be carried out, especially composite material engineering. In recent years, biodegradable materials have gained a lot of interest due to their specific properties and, above all, the possibility of degradation in a certain time. Processing additives, such as stabilisers, allow products to be obtained with a controlled degradation time [1]. One of the advantages of composite materials compared to metal materials is that at the same volume, composite materials have a lighter weight than metal materials. It means that if composite materials are used in transportation, the weight can be light to reduce fuel consumption or increase fuel efficiency. Today, widely used composites are glass fibre-reinforced composites made from a mixture of resin as a binder and glass fibre as a reinforcement. One of the uses of glass fibre composites is to make the walls of boats or fast boats.

The composition of the lignocellulose fibre depends on the plant from which it is extracted as well as on agricultural conditions. It mainly consists of three compounds: cellulose, hemicellulose and lignin [2]. Natural fibres contain several chemical compounds, such as cellulose, hemicellulose, lignin, pectin, and waxes. Therefore, natural fibres are often called lignocellulose fibres [3,4]. Lignin is a complex hydrocarbon polymer that gives stiffness to the stem. It protects the fibre from biological attacks. The crystallinity of cellulose determines the strength of the fibre. Hemicellulose forms a cementing matrix and is hydrophilic. Pectin makes fibre flexible. Some natural fibres used as composite reinforcement are coconut fibre, aka fibre, pineapple king fibre, hemp fibre, reeds grass fibre, sisal, flax, hemp, jute, banana, abaca and so on. Even natural materials resins are being researched and developed [3,5,6]. The advantages possessed by natural fibres are abundant, environmentally friendly, low production costs, and elastic. Besides the advantages, natural fibres also have disadvantages, including non-uniform quality, high water absorption, low strength, and difficulty binding to hydrophilic resins.

Composites are known as advanced materials with a combination of more than one group of materials. The combination effect gives the ultimate properties of the combined materials [7]. Composites have become an inseparable part of everyday human life and can be found everywhere. In ancient times, bricks made of flour and mud were one of the classic examples of composites. Composites in the form of wood, teeth, bones, and muscle tissue also have their importance in nature. Composite materials generally consist of resin reinforced with particles or fibres [8]. For natural fibre-reinforced composites to have strength or tenacity, the factors that need to be considered are (1) bonding between the surface of the fibre and resin, (2) how to arrange the fibre, (3) modulus of elasticity of the fibre used is higher than the matrix [9]. Natural fibres such as jute, banana, sisal, and hemp have been discovered to have a good capacity for use as composite material reinforcement [10]. Fibre-reinforced plastic composites are increasingly popular in the manufacturing industry, especially with natural fibre, such as coconut, as a substitute for synthetic fibre [7]. The surface of coconut fibre, which contains many impurities, will affect the ability and strength of the binding of natural fibres with the matrix. One of the methods carried out to remove impurities on the surface of the fibre is the chemical treatment process, such as alkali treatment [11,12]. Chemical treatment of the fibre can be considered in modifying the properties of natural fibres, such as the surface of the fibre, removing impurities, strengthening the fibre, and increasing the interaction between the fibre matrix. Therefore, modification of fibre surface treatment needs to be considered to increase the strength of fibre-reinforced composites [13,14].

2. Material and methods

In the given section, it is necessary to present in detail the assumptions and course of my research to such an extent that a reader could repeat those works if he were going to confirm the achieved results. In short papers, that information should be given in as short a version as possible.

2.1. Materials experiment

The materials used in this research are coconut fibre, polyester resin, MEKPO catalyst, alkali solution, distilled

water, immersion media, hydrolysis test equipment, tensile, SEM, and furnace.

2.2. Experiment procedure

Coconut is obtained from Sidenreng Rappang Regency, South Sulawesi Province, Indonesia. Coconut fibres are obtained by separating fibres from coconut husk traditionally by hand, as shown in Figure 1.

Fig. 1. The process of separating fibres from coconut husk

The fibre treatment consists of two groups. The first group, coconut fibre, were soaked in an alkali solution for 3 hours at room temperature with a concentration of 5%, 10%, 15%, and 20%. The second group, coconut fibre, was soaked in an alkali solution with a concentration of 20% at room temperature for 1, 5, 7, 9, and 11 hours. Then, the coconut fibre and the first and second groups are dried in a furnace for 5 hours at 90°C. Furthermore, the dry coconut fibre is divided for the next process: hydrolysis testing, tensile testing, pull-out testing, and fibre surface observation using a Scanning Electron Microscope (SEM).

2.3. Hydrolysis process

Hydrolysis testing is carried out to determine the number of lignocellulose compounds (hemicellulose, cellulose, lignin) contained in coconut fibres, both coconut fibres before immersion and coconut fibres after soaking. The method used is the "chesson" method using sulfuric acid (H_2SO_4) [6,15,16]. The hydrolysis process was carried out as described below: one gram of coconut coir fibre sample (A) was immersed in a 150 ml distilled solution, then refluxed for 1 hour at 100°C. Then, the filter "residue" results were washed with distilled water as much as 300 ml. Then, the residue was dried in an oven at 100°C until the mass was constant, obtaining residue (B). The residue (B) is immersed in a 150 ml sulfuric acid (H_2SO_4 1N) solution, then refluxed for 1 hour at 100°C. Then filtered, the results of the filter "residue" were washed with distilled water as much as 300 ml to neutral (pH 7), then the residue was dried in an oven at 100°C until the mass was constant, obtained residue (C). The residue (C) was immersed in a 100 ml sulfuric acid (H_2SO_4, 72%) solution for 4 hours at room temperature. Then, refluxed for 1 hour at 100°C. Then filtered, the filter "residue" results were washed with 400 ml distilled water until neutral (pH 7). Then, the residue is dried in an oven at 100°C until the mass is constant and the residue (D) is obtained. The residue (D) is evaporated in a furnace at 600°C for 4 hours until its mass is constant, obtained (E). To find out the percentage of hemicellulose (C_H), cellulose (C_C), and lignin (C_L) content, the values of A, B, C, D, and E are included in the following equations [6,15]:

$$C_H = \frac{B-C}{A} \times 100\% \quad (1)$$

$$C_C = \frac{C-D}{A} \times 100\% \quad (2)$$

$$C_L = \frac{D-E}{A} \times 100\% \quad (3)$$

2.4. Tensile test

In tensile testing, the specimen is given a load (F) in stages. As a result of the loading, there is a change in cross-section (A) and length (L) to the amount of load by the tensile testing machine, so there is a relationship between stress and strain. Maximum tensile stress values can be obtained using eq. 4, while strain values are obtained with eq. 5 [16].

$$\sigma = \frac{F}{A} \quad (4)$$

$$\varepsilon = \frac{L_1 - L_0}{L_0} \quad (5)$$

where σ = tensile strength (N/mm^2), F = load (N), A = cross-sectional area (mm^2), ε = strain, L_0 = initial length (mm), L_1 = final length (mm). Form of tensile test specimens using ASTM 3379-02 standard, as shown in Figure 2.

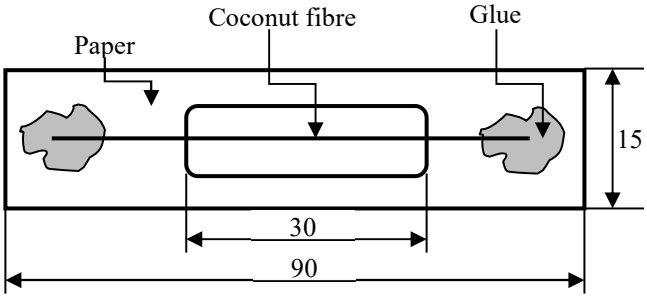

Fig. 2. Tensile test specimens according to ASTM 3379-02

2.5. Pull-out test

Quantitative single fibre pull-out tests can provide information about the interfacial shear stress of fibres

embedded in the matrix that can be converted to an interface shear stress. In addition, the onset of fibre-matrix debonding causes microcrack growth until macro failure can be observed [6]. The pull-out test is expected to provide information about the direct interaction of the interface area of the fibre with the matrix and the failure behaviour between the fibre and the matrix due to the low compatibility of the two materials. Figure 3 shows the interfacial shear stress test procedure where a single fibre is planted in a matrix with a planting depth of L and given an axial tensile load of *F*. Load *F* is expected to be able to pull out the embedded fibre. It is assumed that shear stress occurs along the uniformly embedded fibre surface [17,18]. The value of the interfacial shear stress between the surface of the fibre and the matrix can be calculated by equation 6.

$$\tau = \frac{F}{\pi dL} \quad (6)$$

where τ = interfacial shear stress (N/mm^2), F = load (N), d = fibre diameter (mm), L = fibre embedded length (mm).

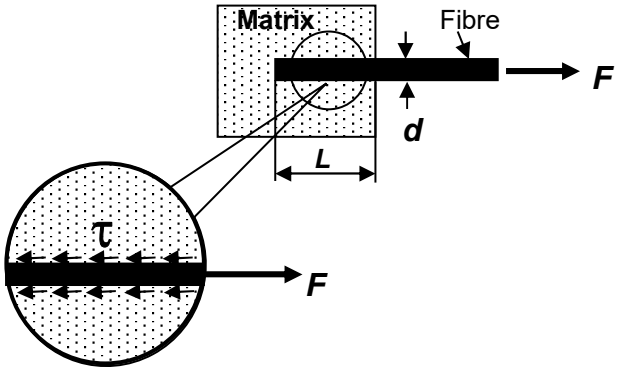

Fig. 3. Pull out test process

2.6. SEM observation

SEM observations were made to observe the surface morphology of the sample. In this case, the cavities can be seen by the interaction of the fibre material with the immersion media or the surface of the fibre with the matrix. The analysis will provide information on the effect of the immersion media on the surface of the fibre or how the matrix with the fibre can interact.

3. Results and discussion

Table 1 shows the results of tests carried out for coconut fibre without treatment and coconut fibre which has been soaked in an alkali solution in various alkali concentrations and soaking times.

3.1. Lignocellulose content

Figure 4 shows the lignocellulose content after the coconut fibre has been soaked for 3 hours in an alkali solution with concentrations of 5%, 10%, 15%, and 20%. The amount of hemicellulose generally decreases with increasing alkali levels except at a concentration of 20% compared to coconut fibre, which is not soaked. Likewise, lignin decreased with increasing alkali levels compared to coconut fibre, which was not soaked. It shows that at high alkali concentrations, delignification occurs in natural fibres and changes the surface morphology of the fibres [4]. The alkali treatment will reduce the amount of hemicellulose compared with coconut fibre without treatment. The decrease in cellulose and lignin is proportional to the increase in alkali solution concentration [6,19]. Alkali treatment can improve natural fibres such as surfaces, remove impurities, strengthen fibres, and increase interaction between fibres and the matrix [16,19].

Table 1.
The Values of Result Testing

Treatment		Amount, %			σ	ε	D	F	τ
		Hemicellulose	Cellulose	Lignin	N/mm^2	%	Mm	N	N/mm^2
Original		15.5	37.9	33.5	186.42	28.33	0.310	3.70	1.85
5% alkali	3 hours	11.0	37.0	29.0	144.00	50.00	0.390	5.43	2.32
10% alkali		11.3	36.8	27.3	113.09	29.17	0.290	3.03	1.72
15% alkali		12.7	22.2	37.5	52.65	11.67	0.330	3.40	1.57
20% alkali		40.9	22.0	6.1	280.94	11.25	0.350	7.38	3.09
1 hour	20%	12.6	27.1	29.6	116.12	13.32	0.250	5.40	6.88
3 hours	alkali	40.9	22.0	6.1	280.94	11.25	0.350	7.38	3.09
5 hours		15.1	24.4	39.7	120.19	17.66	0.300	7.40	7.86
7 hours		8.9	18.4	39.4	150.75	10.77	0.250	5.90	7.52
9 hours		13.3	35.2	28.6	116.12	8.30	0.225	5.10	7.22
11 hours		11.0	21.8	43.8	101.86	8.17	0.225	5.10	7.22

σ = tensile strength, ε = strain, D = fibre diameter, F = load, τ = interfacial shear stress

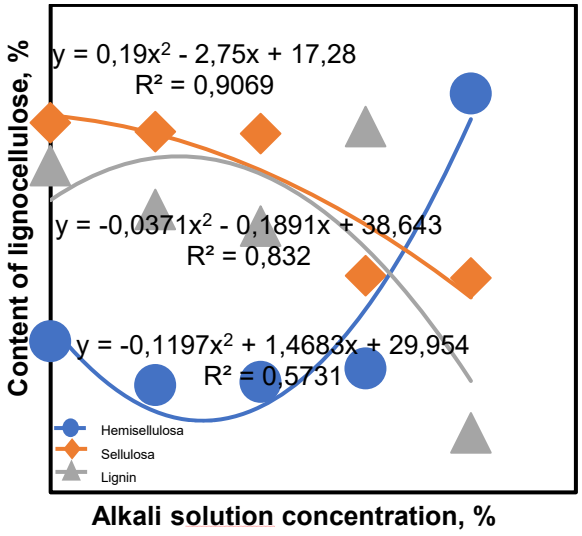

Fig. 4. Soaking in an alkali solution for 3 hours

Figure 5 shows the lignocellulose content after coconut coir fibre was immersed in an alkali solution of 20% concentration for 1, 3, 5, 7, 9, and 11 hours. The amount of hemicellulose generally decreases with the duration of immersion except at the immersion of 3 hours compared with coconut fibre, which is not soaked. Meanwhile, the amount of cellulose decreased with increasing immersion compared to untreated coconut fibre. However, the amount of lignin content increased with increasing immersion time, and the highest amount of lignin obtained in the longest immersion was 11 hours by 43.75%. The lowest amount was obtained at 3 hours of immersion at 6.1%.

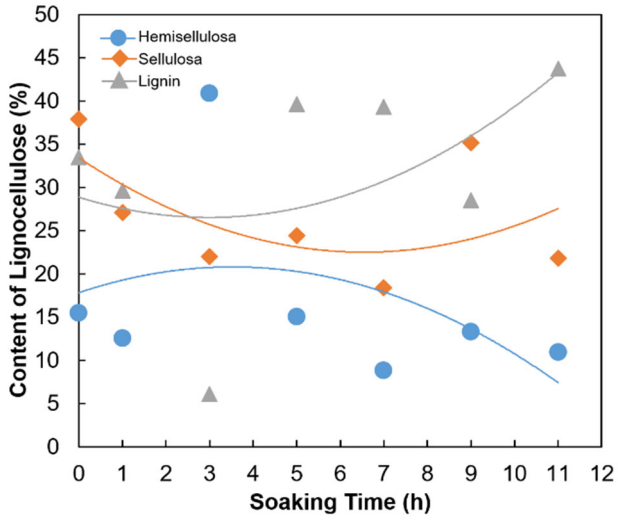

Fig. 5. Soaking in a 20% alkali solution

3.2. Tensile test

Figure 6 shows the tensile strength and strain values of coconut fibre after being soaked for 3 hours in an alkali solution with concentrations of 5%, 10%, 15%, and 20%. The tensile strength of coconut fibre, which has been soaked, is lower than that of coconut fibre, which has not been soaked except for coconut fibre, which has been soaked in alkali, with a concentration of 20% of 280.94 N/mm^2. However, when the tensile strength is highest, the strain is the smallest at only 11.25%. Figure 7 shows the tensile strength and strain values of coconut fibre after being immersed in an alkali solution with a concentration of 20% for 1, 3, 5, 7, 9, and 11 hours. The tensile strength of coconut fibre, which has been soaked, is lower than that of coconut fibre which has not been soaked, except for coconut fibre, which has been soaked for 3 hours at 280.94 N/mm^2. However, the strain decreases with increasing immersion time. Figure 7 shows a decreasing strain trend with treatment time [18].

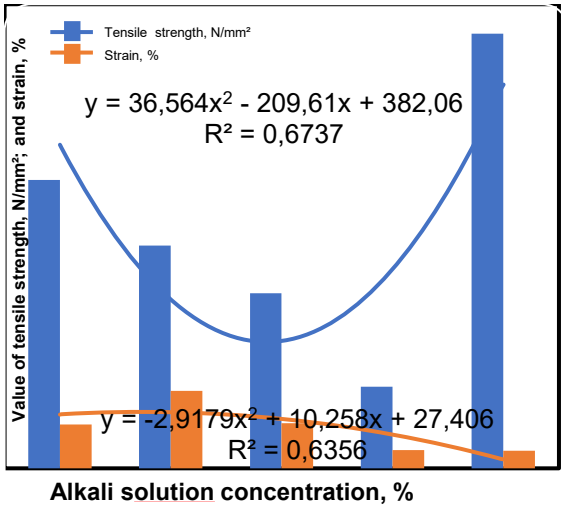

Fig. 6. Tensile strength and strain of coconut fibre after being immersed in an alkali solution for 3 hours

3.3. Pull-out test

Figure 8 shows the bond strength value between coconut coir fibre and polyester resin, wherein coconut coir fibre is immersed for 3 hours in an alkali solution with a concentration of 5%, 10%, 15%, and 20%. The highest Interfacial Shear Stress of coconut coir fibre with polyester resin was obtained at 20% alkali soaking, 3.09 N/mm^2. Long immersion increases the Interfacial Shear Stress between fibre and matrix [20].

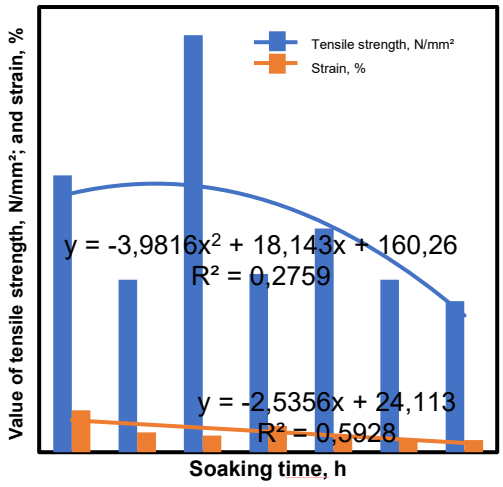

Fig. 7. Tensile strength and strain of coconut fibre after being immersed in a 20% alkali solution

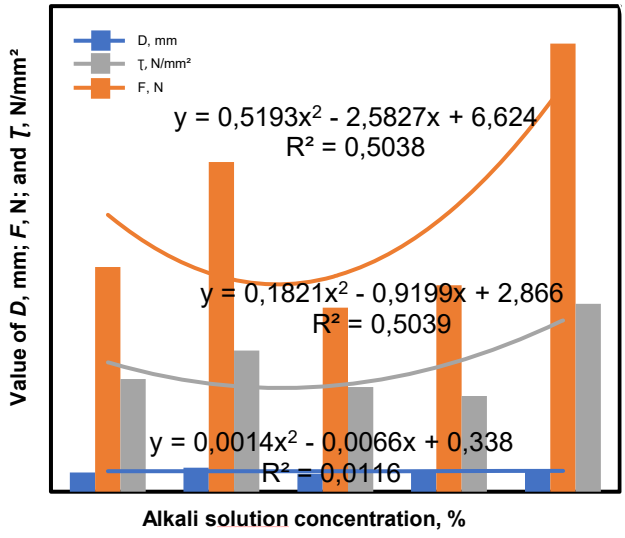

Fig. 8. Interfacial shear stress of coconut fibre after immersion in alkali solution for 3 hours

Figure 9 shows the bond strength between coconut coir fibres and polyester resins, where coconut coir fibres are immersed in an alkali solution at a concentration of 20% for 1, 3, 5, 7, 9, and 11 hours. The strength of the binding of coconut fibre that has been soaked is higher than that of the coconut fibre that has not been soaked. The highest binding strength was obtained at a 5-hour immersion time of 7.856 N/mm^2. The larger the molecule size, the weaker the bond; the smaller the molecular size, the stronger the bonds. Hence, microstructures affect the tensile strength of fibres [20].

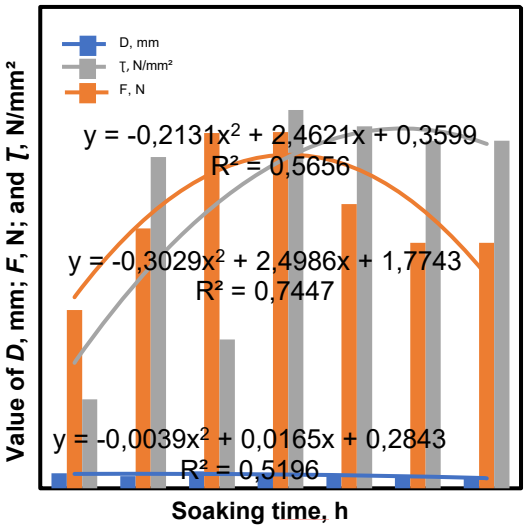

Fig. 9. Interfacial shear stress of coconut fibre after immersion in 20% alkali solution

3.4. Scanning electron microscope analysis

Figure 10 shows coconut coir fibres before soaking (a) and after soaking (b to f). The surface of untreated native coir fibre is smooth, as shown in Figure 10a. It shows that there are still impurities and other substances on the surface of coconut fibre. At the same time, the surface of coconut fibre soaked in an alkali solution of 20% looks rough compared to coconut fibre without immersion due to the release of a waxy coating on the surface of the fibre [16,21,22]. The SEM observations show that the surface morphology of the fibre becomes rough after the alkali treatment and roughness increases with increasing immersion period. It occurs due to the degradation of the amount of hemicellulose and lignin. Thus, the alkali treatment results in significant and effective changes in the surface morphology of the fibres to increase the bond between the fibre and the matrix [3,23,24]. It is in accordance with the interfacial shear stress (IFSS) shown in Figure 9. The IFSS is greater with the longer immersion, although the highest is obtained at 5 hours. However, after soaking for 5, 7, 9 and 11 hours, the value of the average IFSS of more than 7 N/mm^2 is shown in Table 1.

Figures 11 and 12 show the results of SEM photographs (a) of coconut fibres pulled out from (b) polyester matrix in the pull-out test process. Figure 11a shows coconut fibres being pulled out from the polyester matrix. A small portion of the polyester matrix is still attached to the surface of the coconut fibre. Figure 11b shows the former coconut fibres forming small trenches in the polyester matrix. Figure 12a

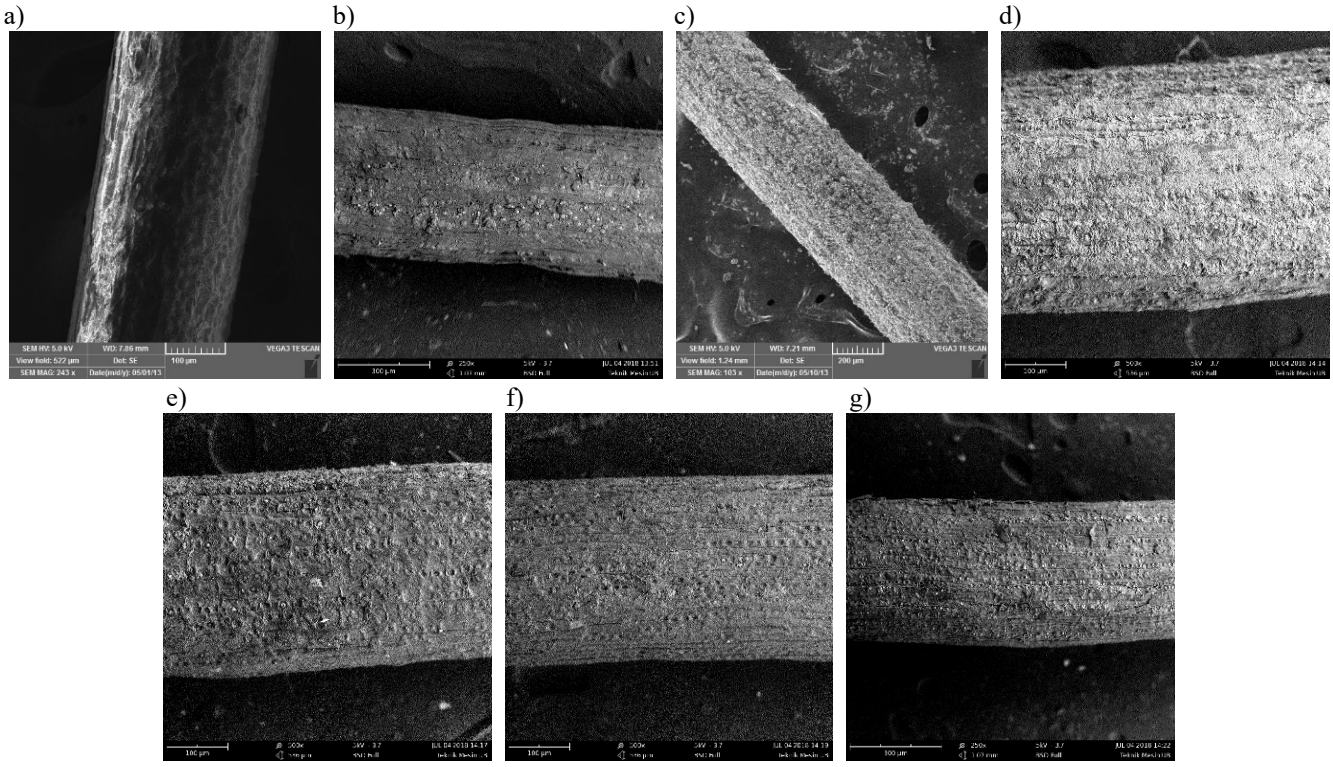

Fig. 10. SEM of coconut fibre without and with alkali treatment: a) original, b) 1 hour, c) 3 hours, d) 5 hours, e) 7 hours, f) 9 hours, g) 11 hours

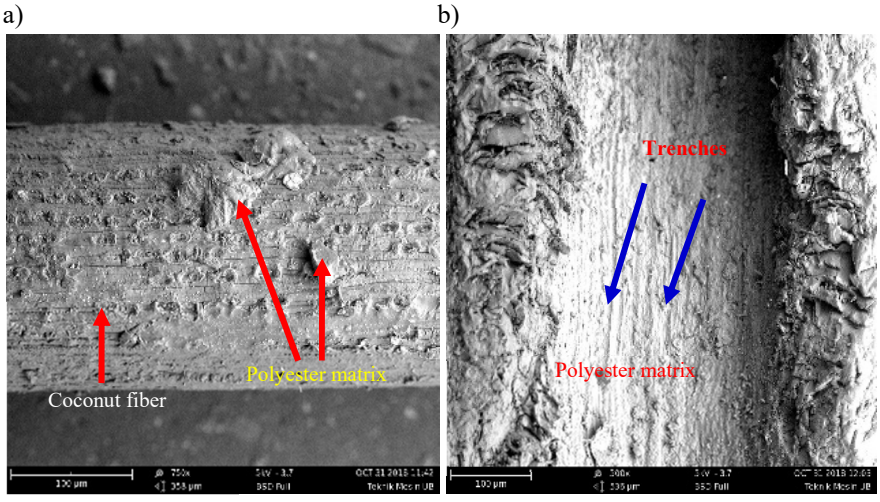

Fig. 11. Result of pull out test after treatment 20% alkali for 1 hour immersions: a) coconut fibre, b) polyester matrix

shows a coconut fibre that is covered by a polyester matrix attached to the coconut fibre. At the same time, Figure 12b shows a trench from the former coconut fibre on a polyester matrix. It shows that there has been a bonding between the coconut fibre and a polyester matrix, so there is an interfacial shear force between the coconut fibre and the polyester matrix. When compared with Figure 11 and Figure 12, the latter shows that the bond between the coconut fibre and the polyester matrix is very strong, so when the coconut fibre is pulled out from the polyester matrix, it is still

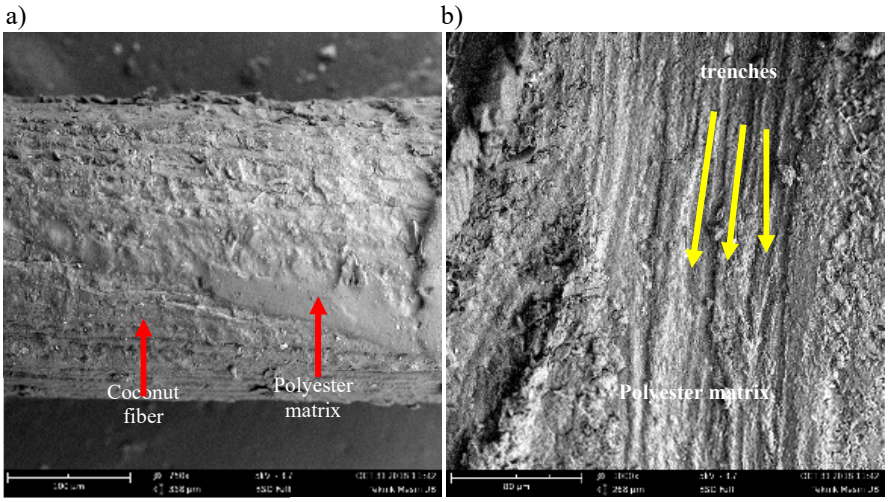

Fig. 12. Result of pull out test after treatment 20% alkali for 5 hours of immersion: a) coconut fibre, b) polyester matrix

attached to the coconut fibre. It shows that the alkali treatment increased the interface adhesion between the coconut fibre and the polyester matrix [21]. In Figure 12a, coconut fibre soaked for 5 hours in a 20% alkaline solution obtained the highest value of the interface shear stress, namely 7.86 N/mm^2, as shown in Figure 9 and Table 1.

4. Conclusions

Based on the results and discussion, it is concluded that:

a. Alkali treatment can reduce the amount of hemicellulose and lignin and eliminate impurities that are attached to the surface of the coconut fibre so that the surface of the coconut fibre becomes coarser,
b. The highest tensile strength is obtained at a concentration of 20% NaOH, namely 280.94 N/mm^2,
c. Long immersion increases interfacial shear strength, and the maximum interfacial shear strength is obtained at 5 hours of immersion, namely 7.86 N/mm^2.

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