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On the cover a fragment of Jolanta Owidzka's structural tapestry 'Prayer book'

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Contents

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Influence of the Soaking Time on the Mechanical Properties of Coir as a Natural Composite Reinforcement

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Abstract
This study aimed to determine the effect of the long soaking of coir in sodium hydroxide solution on its mechanical properties. The materials used, namely coir, sodium hydroxide solution, polyester matrix, catalyst, and aquades. The coir was soaked in a sodium hydroxide solution for 1, 5, 7, 9, and 11 hours. After that, coconut fibre was washed using distilled water, and then dried in an oven at 90 °C for 5 hours. Next, a single fibre tensile and pull out test was performed. Based on the results and discussion, it was concluded that immersing coconut fibre in sodium hydroxide solution for 7-hours gave the maximum tensile strength and interfacial shear strength of 223.91 N/mm^2, and 9.8 N/mm^2, respectively Coir as a composite reinforcement has the potential to be used for engineering material such as ceiling boards and fishing boat walls.

Key words: *soaking, NaOH, tensile, shear, interfacial.*

Introduction

Factors that need to be considered to increase the strength of composites reinforced with natural fibre are (1) the bonding between the surface of the fibre and the matrix, (2) the way of preparing the fibre, and (3) the fibre elasticity modulus used being higher than the matrix. The surface of coir containing a lot of impurities will affect the process of engagement with the matrix. One way to remove dirt on the fibre surface is the chemical treatment process. The chemical treatment of fibres can be considered in modifying the properties of natural fibres, such as fibre surfaces, removing impurities, fibre strength, and increasing the interaction between the fibre-matrix. Therefore, modification of the surface treatment of fibres should be considered to increase the strength of fibre-packed composites [1].

The characteristics of composite reinforced natural fibre depend on the properties of the fibre, fibre growth, bonding interactions between the fibre and the matrix, fibre size, fibre form, number of fibres in the matrix, and processing techniques. Also, the chemical treatment can determine the nature of a composite product. It is also affected by some fibre conditions such as how the fibres are obtained, as well as their size and shape fibre [2]. The size and shape of the fibre are necessary for certain purposes such as processing and engagement with the matrix. Fibre content can also affect the composite mechanical strength. Two factors that may affect the distribution of fillers are interactions between fillers and the filler length. The interaction between fellow lignocellulosic fillers through hydrogen bonds causes the buildup of fibres, resulting in the cracking or breaking of fibres. Also, this type of filler may also affect the composite strength because different lignocellulosic fillers have different cellulose, lignin and hemicellulose contents [3]. Coir is treated with 0.7% $NaClO_2$ with a long boiling variation of 0, 15, and 90 minutes, where the longer the fibre is boiled, the more the lignin decreases and the cellulose and hemicellulose increase, while the tensile strength decreases, as compared to the fibre without treatment [4]. The chemical composition is a crucial criterion that influences the mechanical properties of natural fibre [5].

The interconnection of the fibre with the matrix consists of several bonding models: (a) chemical bonds, (b) ionic electrostatic bonds, (c) molecular reaction interconnections, and (d) mechanical bonds [6]. The NaOH treatment of fibres will give two effects fibres: (1) increasing the surface roughness of the fibres to produce better interlocking, and (2) increasing the amount of loosened cellulose [7]. The composite strength of the treated NaOH composite increased significantly by about 53% compared to composites made from untreated fibres and by 33% compared to the non-fibre composite, where the fibre was soaed in 10% NaOH for 3-hours [8]. IFSS is a direct measurement of the bond between a fibre and a matrix [9]. Nam et al. also reported that coir-PLA (polylactic acid) matrix soaked in 5% NaOH solution for 24, 48, 72, and 96 hours obtained the highest IFSS value at 72 hours immersion [10].

In the recent decades, recycling, reusing and recovering cellulosic fibres have increased considerably. Cellulosic fibres, like wood, sisal, coir (coir), jute, palm, bamboo, wood, paper in its natural condition, as well as several waste cellulosic products such as shell flour, wood flour and pulp have been used as reinforcing agents of different thermosetting and thermoplastic matrix. The global production of cellulosic fibres from wood pulp in 2012 was 173.8 million tonnes. Chemical pulp made up 93% of market pulp. From these values, it can be observed that the availability of this raw material is really important and its importance will grow when the consumption per capita of paper in developing countries reaches

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values comparable to those of developed countries [11].

Experimental

Materials

Coconut fibre (Coir) was obtained from the Sidenreng Rappang Regency of South Sulawesi Province, Indonesia. The coir was separated from coconut husk by a mechanical extraction method and then cleaned. Sodium hydroxide (NaOH) solution with 20% concentration, polyester matrix (Yukalac BQTN-EX Lot No. T1580818TS), catalyst ME-KPO (Metyl Ethyl Ketone Peroxide), and aquades was obtained from chemical stores in Makassar City.

Fibre treatment

The Coir was soaked in a NaOH solution for 1, 5, 7, 9, and 11 hours at room temperature. After that, the coir was washed using distilled water, and then dried in an oven at 90 °C for 5 hours [12].

Hydrolysis process

The chemical composition of the coir was determined using the Chesson method [12]. A mixture containing 1 g of dry coir, and 150 ml of distilled water was heated in a glass tube at a temperature of 90-100 °C for 1 hour, and called sample (A). Sample (A) was filtered, then the residue was washed with 300 ml of hot water and next, dried in an oven to a constant weight, which was called sample (B). Sample (B) was mixed with 150 ml of 1 N H_2SO_4, then heated in a glass tube at a temperature of 90-100 °C for 1 hour. Next, Sample (B) was filtered, and then washed with 300 ml of distilled water. Atferwards, the residue of sample (B) was dried, and called sample (C). Sample (C) was then immersed in 10 ml of 72% H_2SO_4 at room temperature (28 °C) for 4 hours. After that, 150 ml of 1N H_2SO_4 was added to sample (C) and refluxed in glass tubes at 90-100 °C for 1 hour. The solid was next washed with 400 ml of distilled water, then heated in an oven at 105 °C and weighed to a constant weight; it was called sample (D). Finally, sample (D) was heated to ash and weighed; this was then called sample (E). The percentage of hemicellulose (H), cellulose (S), and lignin (L) were calculated using the following ***Equations (1-3)***:

$$H = (B - A)/A \times 100\% \quad (1)$$

$$S = (C - D)/A \times 100\% \quad (2)$$

$$L = (D - E)/A \times 100\% \quad (3)$$

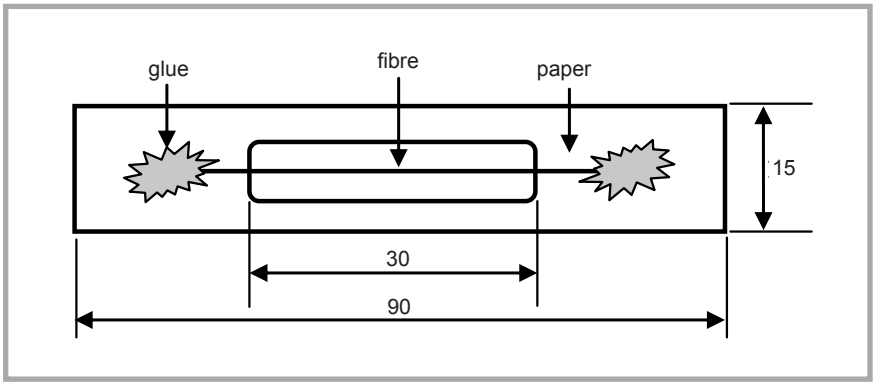

Figure 1. *Specimen of single fibre test ASTM 3379-02 for the tensile test.*

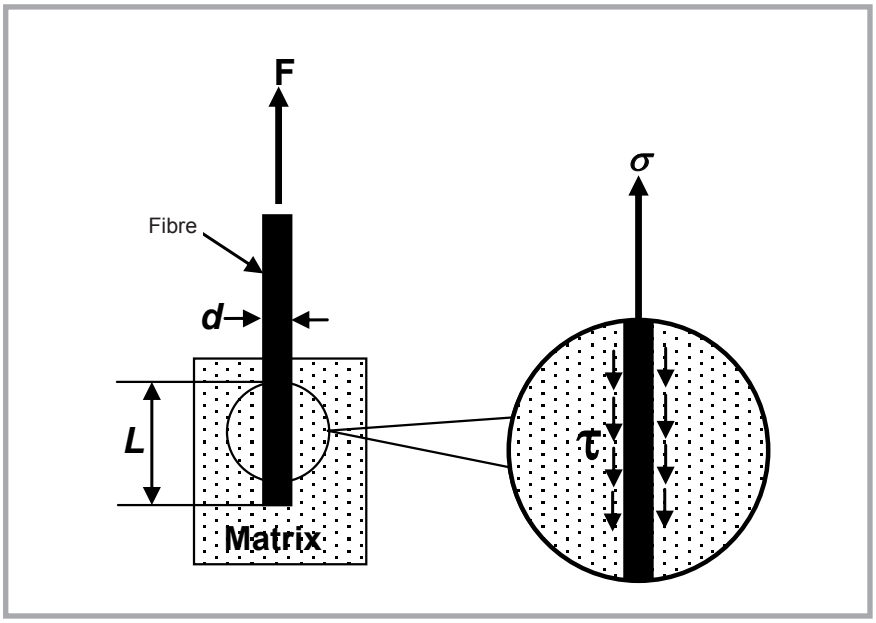

Figure 2. *Process of single fibre pull out test.*

Tensile and pull out test

Tensile and pullout tests of single fibre were performed using the Testometric M500-25CT DBBMTCL pull-2500 kg Test, Rochdale, England. A single fibre tensile test was carried out in accordance with ASTM 3379-02, shown in ***Figure 1***. Each test variable was performed 3 (three) times. The tensile strength can be determined by applying ***Equation (4)***:

$$s = F/A \quad (4)$$

Where, s – maximum tensile stress (N/mm^2), F – maximum load (N), A – sectional area (m^2).

Single fibre pull out (SFPO) testing was carried out to determine the reaction or bonding ability between the fibre and the matrix. This test is performed over the length of the fibre embedded in the matrix which was 1 mm, with a tensile speed of 1 mm/min [10, 22]. The process of interfacial shear stress between the surface of the coir and the polyester matrix is shown in ***Figure 2*** [9, 12]. The strength of the shear force that occurs can be calculated using the following equation (5) [9, 12-14]:

$$t = F/pdL \quad (5)$$

Where, t – interfacial shear strength (N/mm^2), F – maximum load (N), d – fibre diameter (mm), L – embedded fibre length (mm).

In the SFPO test, the maximum tensile load value of the fibre until fibre released from the matrix was established. Based on the value of the tensile load and ***Equation (5)***, we obtained the value of the interfacial shear strength (IFSS) [10].

SEM observation

Observation of the surface of the coir was made using an SEM (Scanning

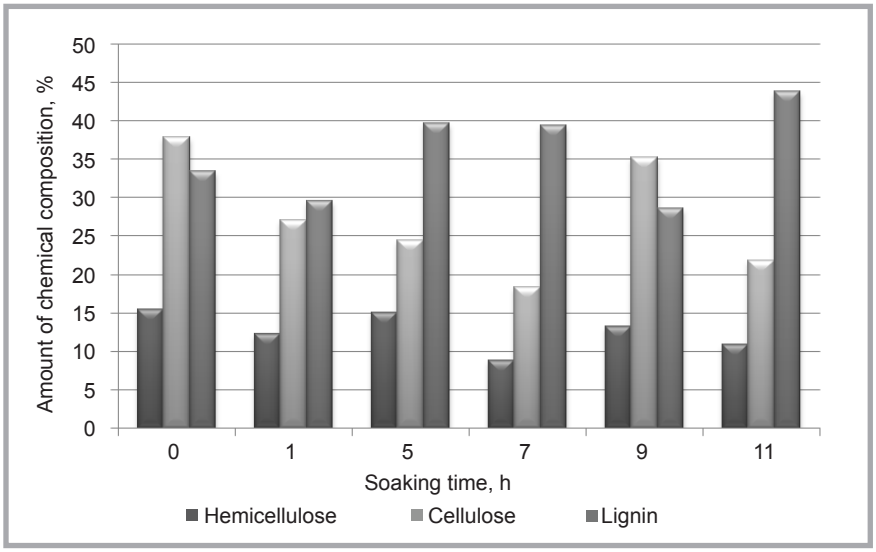

Figure 3. *Amounts of the chemical composition of coir.*

Table 1. *Amounts of the chemical composition of coir.*

Soaking time, h	Hemicellulosem, %	Cellulose, %	Lignin, %
0	15.5	37.9	33.5
1	12.25	27.07	29.62
5	15.08	24.4	39.65
7	8.85	18.4	39.34
9	13.29	35.18	28.55
11	10.96	21.84	43.75

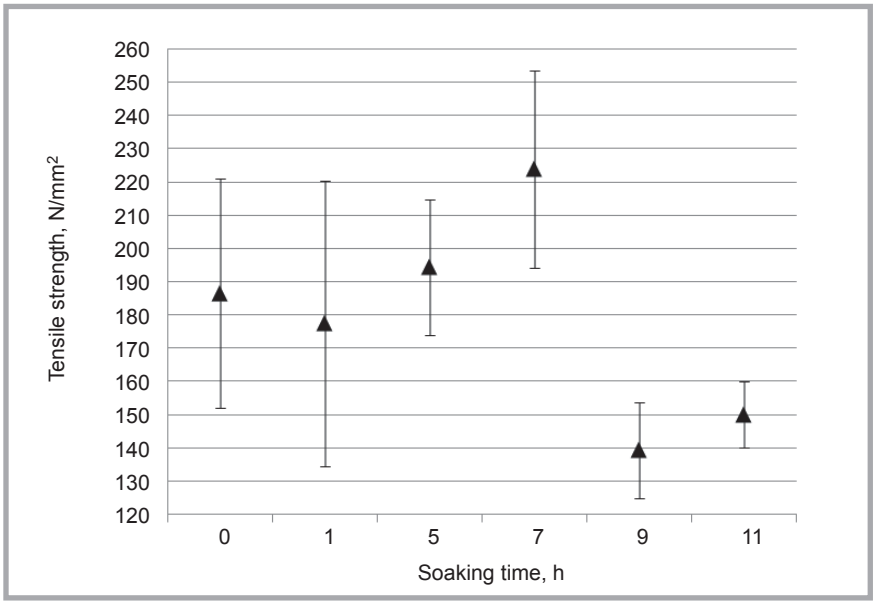

Figure 4. *Tensile strength of coir.*

Table 2. *Tensile strength of coir.*

Soaking time, h	D, mm	F, N	σ, N/mm²	ε, %
0	0.31	14.02	186.42	28.33
1	0.28	10.30	177.55	29.64
5	0.25	9.53	194.31	35.40
7	0.23	9.47	223.91	27.23
9	0.30	10.15	139.39	26.55
11	0.30	10.60	150.04	28.85

Electron Microscope, Phenom G2Pro). This is done to see changes in the surface roughness of the coconut fibre, as well as the grooves in the polyester matrix, which are the location of former coir fibres.

Results and discussion

Hydrolysis process

Table 1 and ***Figure 3*** show the hemicellulose, cellulose, and lignin content of coir before and after immersion in NaOH solution. Coir without soaking contains hemicellulose 15.5%, cellulose 37.9%, and lignin 33.5%. This is different from that reported by Ramakrishna, namely hemicellulose 31.1%, cellulose 33.2%, and lignin 20.5% [15], and from that reported by Khan, namely hemicellulose 7.6%, cellulose 44.1%, and lignin 37.1% [16]. These differences indicate that despite the same type of fibre, the place of growing the fibre will determine the amount of hemicellulose, cellulose, and lignin [7, 17, 18]. In this study, the lowest amount of hemicellulose and cellulose content was obtained for a 7-hour immersion i.e. 8.85% and 18.40 %, respectively. This shows that the longer the fibre is soaked in NaOH solution, the greater the degradation of hemicellulose, and cellulose [7]. However, when coir is soaked in a solution of 0.7% $NaClO_2$ for 0, 15 and 90 minutes, the amount of cellulose and hemicellulose increases along with the time of fibre soaking [4]. This shows that the type of chemical solution used in soaking will also determine the chemical composition of the fibre.

Tensile strength

Table 2 and ***Figure 4*** show the tensile strength of coir before and after being soaked. The tensile strength of coir before soaking in NaOH solution was 186.420 N/mm^2, while after soaking the highest was obtained for a 7-hour immersion i.e. 223.907 N/mm^2 ; decreasing after that. A close agreement is found with the data of a previous study of Ray where the percentage of the tensile strength decreased by 23% after being immersed in 5% NaOH solution for 8 hours, despite an increase in the modulus of elasticity of flax fibre of 12%, 68%, and 79% after 4, 6 & 8 hours [19]. Muensri reported that for coir which was immersed in 0.7% $NaClO_2$ solution for 0, 15, and 90 minutes, the tensile strength increased with increasing immersion time, as compared to untreated fibres [4].

Interfacial shear strength

In the SFPO test, the maximum tensile load value is given to the fibre until the fibre pulls out of the matrix. Based on the value of the tensile load and ***Equation (5)***, we obtain IFSS values as shown in ***Table 3***, and ***Figure 5***.

The low value of IFSS can be caused by a mismatch between hydrophilic fibres and hydrophobic matrix and the presence of layers or impurity particles on the surface of coir, which results in fewer mechanical interactions between the coir and the polyester matrix [13]. The value of IFSS of coir-polyester matrix that was immersed in NaOH solution increased compared with that which was not immersed. As shown in ***Table 3***, the IFSS of coir- polyester matrix soaked in NaOH solution increases with increasing immersion time from 0 to 11 hours. This occurs due to the removal of layers or impurity particles on the surface of the coir during immersion in NaOH solution, which causes increased adhesion between the coir and polyester matrix [9]. However, the IFSS after soaking for 9 and 11 hours is lower than after soaking for 7 hours. This can be caused by the less adhesion of the coir- polyester matrix and mechan-

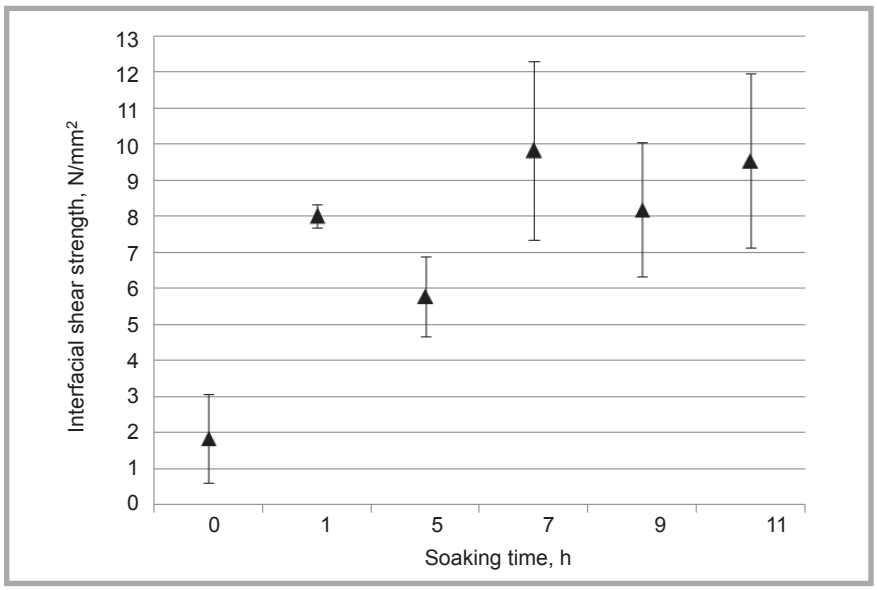

Figure 5. *IFSS of coir-polyester matrix*

Table 3. *IFSS of coir-polyester matrix.*

Soaking time, h	d, mm	F, N	τ, N/mm^2
0	0.31	3.70	1.85
1	0.28	6.90	8.01
5	0.28	5.17	5.78
7	0.26	7.90	9.82
9	0.28	7.17	8.18
11	0.25	7.37	9.54

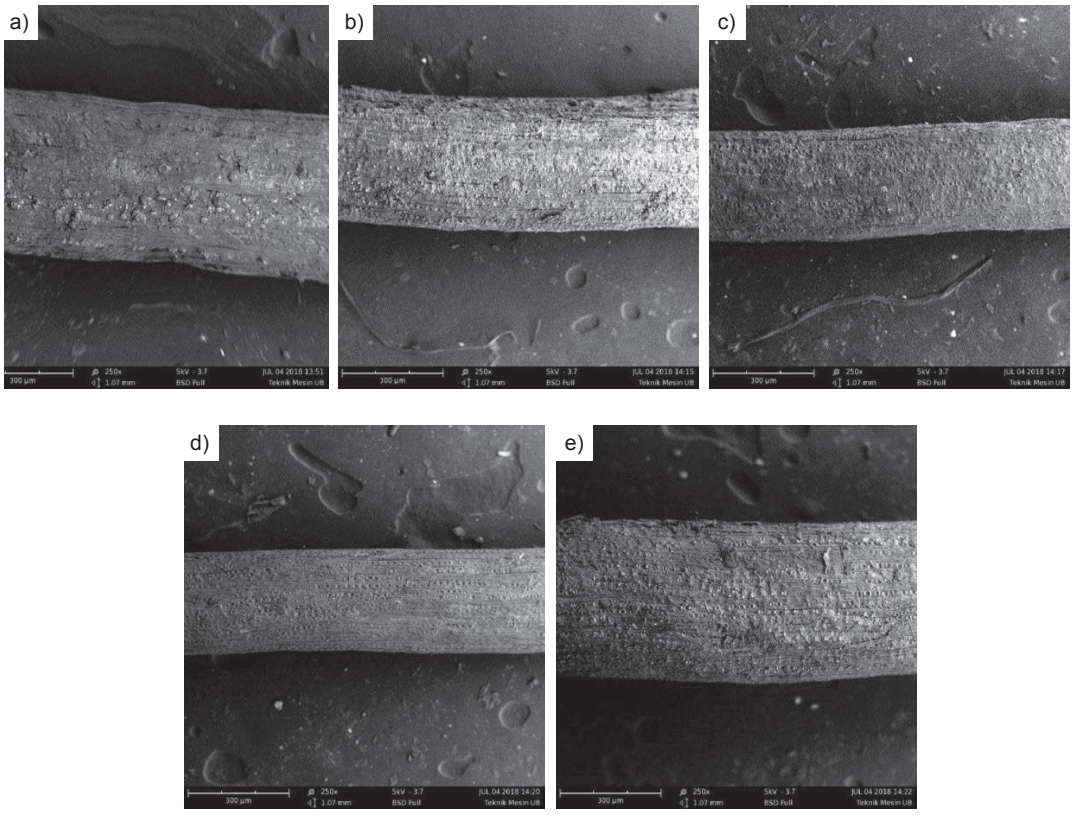

Figure 6. *Surface roughness of coir after soaking in NaOH solution: a) 1 hour, b) 5 hours, c) 7 hours, d) 9 hours and e) 11 hours,*

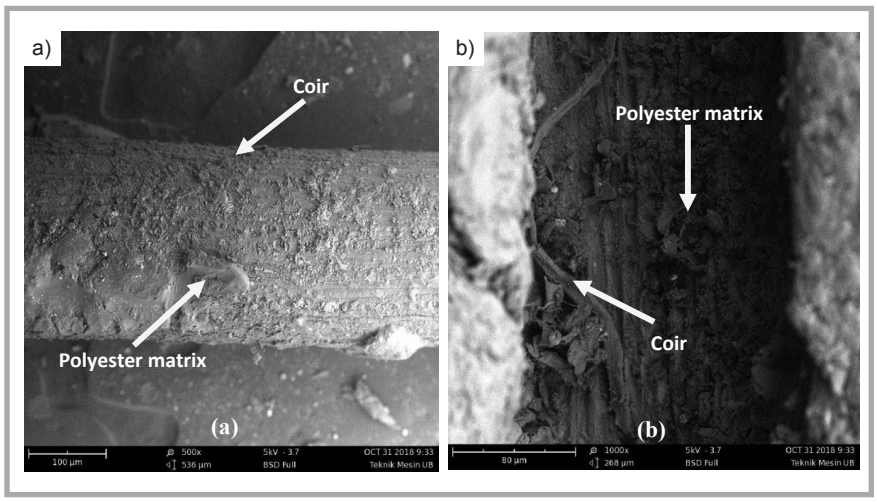

Figure 7. *SFPO test results of coir soaked for 7 hours in a NaOH solution: a) coir is removed from the matrix after a pull out test, b) matrix where extracted coir is removed.*

ical interaction between the fibre and the matrix. In short, the best NaOH treatment of coir in this study was 7 hours soaking. Soaking coir in a NaOH solution increases the strength of the interface bond [10]. The immersion of coir in NaOH solution increases the bond strength of the interface and the wetting of the fibre by the polyester matrix, which causes an increase in bonding between the fibre and the matrix [13].

SEM observation

Figure 5 shows the IFSS of coir before and after soaking in a NaOH solution. ***Figure 5*** shows that the soaking time of coir in the NaOH solution has a very significant effect. The highest IFSS is obtained for 7 hours' soaking. This shows that after 7 hours' immersion, the IFSS decreased. As Nam reported, forcoir soaked in a 5% NaOH solution for 24, 48, 72, and 96 hours, the highest IFSS was obtained for an immersion of 72 hours, after which the IFSS decreased [10]. The value of IFFS for 7 hours' soaking is due to the surface of the coir being coarser compared to that after another soaking, as shown in ***Figure 6*** [13]. Surface roughness will affect the bond strength between the fibre and matrix. Rougher surfaces will increase the bonding ability between fibres and the matrix [12, 20, 21].

Figure 7.a shows the surface of coir removed from the polyester matrix in a SFPO test. On the surface of the coir, the matrix is still attached. This shows the strong bond between the matrix and the coir. ***Figure 7.b*** shows traces of coir left behind in the matrix as a friction effect between the coir and polyester matrix. On the groove, a visible part of the coir left behind is attached to the matrix. This shows the strong bond between the coir and the polyester matrix [22-24]. Soaking in NaOH solution can increase the adhesion between the fibre and the matrix because it removes natural and artificial impurities from the surface of the fibre and changes the unit's arrangement in cellulose macromolecules. In addition, it can also increase the surface roughness of the fibres and the amount of cellulose exposed to the fibre surface, resulting in better mechanical interlocking. As in ***Table 1***, the amount of coir cellulose that has been soaked in NaOH solution is smaller than that of coir that has not been soaked. Therefore, the development of fibre surface tomography and the bonding of the fibre interface with the matrix will improve mechanical properties [13].

Conclusions

Coir was soaked in NaOH solution with a concentration of 20% for 1, 5, 7, 9, and 11 hours to determine the effect of the soaking time. After testing the chemical composition, tensile strength, and interfacial shear strength, as well as observation of the surface of the coir, it was concluded that the soaking time of the coir in NaOH solution decreased the amount of hemicellulose and cellulose, making the surface of the fibre coarser. The tensile strength and interfacial shear strength maximum were obtained after 7-hours immersion – 223.907 N/mm^2 and 9.8 N/mm^2, respectively

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