Scanning Electron Microscopy Observation of Coir Fibre with Alkali and Drying Method Treatment

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Abstract. The natural fiber, such as bamboo, hemp, ramie, jute, and coir, has been proposed as an alternative fiber to replace the synthetic fibers. Coir fiber, which is physically recognized as coarse and stiff fiber with red-brown colored, comprises lignin, cellulose, hemicellulose, and other plant substance. Coir fibers capability can stretch beyond the elastic limit with no rupture and excellent resistance to microbiological and salt degradation. Therefore coir fiber can be applied as an alternative material for a marine structure such as boat and small crafts. In this paper, the scanning electron microscopy (SEM) observation was made to evaluate the influence of alkali treatment (2% and 6% NaOH) and the drying method, including sun drying, oven, and air drying. Progressive changes in surface morphology due to the two kinds of coir fibre treatments were presented.

1. Introduction

In the last decades, the development of technology has overgrown. Natural fibers are starting to be widely used to replace synthetic fibers, ranging from household appliances to the automotive and manufacturing industries. Natural fibre properties can be affected by several conditions such as the size, the shape, and the method of extracting/process the natural fibers. The size and the formation of natural fibers are indispensable, especially for specific purposes such as fabrication and joining with a matrix to produce the composite materials. The fiber component is an essential element because fiber components determine the material mechanical properties, such as stiffness, toughness, and strength. The primary function of fibers in composites is as load carriers, which provide properties of stiffness, strength, thermal stability, and the other features and give the conductivity to the material composite.

Coir fiber is one of the natural fibers that might be used as reinforcement of composite material. Coir fiber is comprised of 36-43 wt% of cellulose, 41-45 wt% of lignin, and 0.15-0.25 wt% of hemicellulose, [1]. Some references can be found in the study of mechanical properties of coir fiber, Siakeng et al., [2], observed the effects of 6% alkali, 2% silane, and 6% calcium hydroxide on tensile, morphological, thermal, and structural properties of coir fiber. The result shows that the alkali-treated thread has the lowest fibre diameter and the highest interfacial stress strength. Silva et al. [3] investigated the mechanical properties of Native Brazilian Coir Fiber. The tensile strength, elongation at break, and initial modulus were evaluated for coir fiber, treated by alkali and untreated. The result shows that the mechanical properties are improved after treated with an alkali (5 wt% NaOH) for a period of 48-hours.
The thermal stability slightly decreased with thermal events between 28 and 38°C. Bakri and Eichhorn, [4], study on the deformation micromechanics of coir and celery fibers. The mechanical testing shows that the coir fiber possesses a non-linear stress-strain curve. The coir fiber also exhibits high strain to failure. The other study of coir fibers mechanical properties can also be found in the following reference [5-10]. Several processes can be conducted to improve the mechanical characteristics and surface modification of natural fiber with the chemical treatment, [11-13], physical techniques, [14], and biological techniques, [15].

This paper focuses on the scanning electron microscopy observation of the coir fiber, which is treated with the alkali (2 wt% and 6 wt% NaOH) and drying method treatment. After the alkali treatment and drying method, the characterization of coir fiber was performed using the scanning electron microscopy.

![Figure 1. The variety of coconut tree in Indonesia: [a] kelapa hijau; [b] kelapa gading; [c] kelapa pandan wangi; [d] kelapa genjah entok.](image)

2. Experimental Study

2.1. Materials

Coir is a natural fiber which is also known as “Coco” or “Kokos”. It is extracted from the coconut (Cocos nucifera) fruit endocarp (outer shell/husk). The coconut tree is included in the palm tree family, which is called the Arecaceae. There are only one species of the coconut tree. However, there are many different varieties which are considered into various types and group. The primary kind of coconut tree is Tall Coconut Varieties, Dwarf Coconut Varieties, and Hybrid Coconut Varieties. Furthermore, there are several sub-grouped of coconut varieties, which includes:

1. King Coconut
2. East Coast Tall Coconut
3. West Coast Tall Coconut
4. Fiji Dwarf
5. Dwarf Orange Coconut
6. Malayan Yellow Dwarf Coconut
7. Golden Malay Coconut
8. Maypan Coconut
9. Macapuno Coconut

According to the plant population growth location, the selected type of the coconut trees might be included in the sub-varieties of Golden Malay Coconut, Dwarf Orange Coconut, and Malayan Yellow Dwarf Coconut. However, in Indonesia, it is known as “Kelapa Hijau”, “Kelapa Gading”, “Kelapa Pandan Wangi”, and “Kelapa Genjah Entok”, Figure 1.

The coconut fruit husk (endocarp) should be processed to become a good quality coco fiber. The first step is the preparation stage, which is sorting and selecting the husk. The second step is the coir softening stage with the hammer mill as a primary tool. The next step is the coir defibring stage, which is the most critical process to generate the coco fiber. In this process, the coir fibers will be separated from the husk or the coir outer part. The process cannot be done manually. Therefore it must be processed with the fiber separating machine (defibring machine). The next process is the sieving process, which is sorting or separating the coco dust, the soft fiber, and the coarse fiber. After that, the next step is the drying stage, which reduces the coir fiber's water content. Finally, the packing stage was made by the packing machine in order to make secure handling for delivering the product to the consumer.

2.2. Treatment of coir fibres
The coco fibers were washed and rinsed with water to remove the dirt and any surface contaminants. The fibres were then soaked in the alkali solution. The alkali treatment was arranged with a sodium hydroxide solution of 2 wt% and 6 wt%, Figure 2. The soaking period of the alkali treatments were 1 hour and 2 hours. After the soaking process, the coir fibers were rinsed with the water and continued to the drying process. The drying processes were divided into three drying methods: oven drying, air drying, and sun-drying, Figure 3. The oven-drying method is conducted drying procedure using a traditional/conventional oven, which is heated by the stove for 30 minutes. The oven temperature has been arranged around 80°C. In the air-drying method, the coco fiber was drying at room temperature (25°C-30°C) with the blowing fan for 48 hours. The sun-drying process was made with the range temperature 30°C to 45°C for 3 hours. The scenarios of the alkali treatment and drying method can be seen in Table 1.

| Scenario | NaOH Solution Concentration | Soaking Period | Drying Method |
|----------|-----------------------------|----------------|---------------|
| C2P1O    | 2 wt %                      | 1 hr           | Oven Drying   |
| C2P1A    | 2 wt %                      | 1 hr           | Air Drying    |
| C2P1S    | 2 wt %                      | 1 hr           | Sun Drying    |
| C2P2O    | 2 wt %                      | 2 hrs          | Oven Drying   |
| C2P2A    | 2 wt %                      | 2 hrs          | Air Drying    |
| C2P2S    | 2 wt %                      | 2 hrs          | Sun Drying    |
| C6P1O    | 6 wt %                      | 1 hr           | Oven Drying   |
| C6P1A    | 6 wt %                      | 1 hr           | Air Drying    |
| C6P1S    | 6 wt %                      | 1 hr           | Sun Drying    |
| C6P2O    | 6 wt %                      | 2 hrs          | Oven Drying   |
| C6P2A    | 6 wt %                      | 2 hrs          | Air Drying    |
| C6P2S    | 6 wt %                      | 2 hrs          | Sun Drying    |
| C0P0N    |                             |                | No Treatment  |

Table 1. The scenarios of the alkali treatment and drying method of the coir fibers.
Figure 2. The alkali treatment of the coir fibre: [a] 2 wt% NaOH; [b] 6 wt% NaOH.

Figure 3. The drying method of the coir fibre: [a] oven drying; [b] air drying; [c] sun drying.

3. Scanning electron microscopy (SEM) of the coir fibres
The investigation of the coir surface morphology was made by scanning electron microscopy (SEM). The alkali treatment and drying method's influence can be seen from the single coir fibre surface with magnification about 40 times. In the case of no treatment sample and the samples using the sun-drying method with the soaking period of 2 hours (C2P2S, C6P2S, C0P0N), the magnification was determined as large as 500 times to 2000 times. This consideration is selected since it is limited for examining all of the specimens with a very large magnification factor. Therefore the influence of alkali treatment with the more extended soaking period is selected to be prioritized. However, the other specimens still in progress to be evaluated with a very large magnification factor (M.F.). The result of SEM with 40 times magnification can be seen in Figure 4.
Figure 4. The surface morphology of the coir fibre with magnification factor 40 times: [a] C2P1O; [b] C2P1A; [c] C2P1S; [d] C2P2O; [e] C2P2A; [f] C2P2S; [g] C6P1O; [h] C6P1A; [i] C6P1S; [j] C6P2O; [k] C6P2A; [l] C6P2S; [m] C0P0N.

4. Results and discussion
The surface morphology of untreated and alkali-treated coir fibres are illustrated in Figure 4. Figure 4a-4f showed the surface morphology of the treated-alkali with a concentration of 2 wt%. Figure 4g-4l described the surface morphology of the treated-alkali with a concentration of 6 wt%. Finally, Figure 3m has presented the non-treatment specimen of the coir fibre.

It is evident from Figure 4 that the alkali treatment influences the surface morphology of the coir fibre. It can be seen from Figure 4a-4f that the image of the coir surface is different from the coir surface image in Figure 4g-4l. However, Figure 4a-4f has a similar image coir surface with the untreated specimen. It is indicated that alkali concentration 6 wt% has a significant influence on the coir fibre surface morphology than the sample, which is treated with a concentration of 2 wt%. In the case of the soaking period, Figure 4a-4c, and Figure 4g-4i have shown similar images in Figure 4d-4f and Figure
It is indicated that the soaking period between 1 hour and 2 hours has no significant influence on the surface morphology of coir fibre. According to the drying method, Figure 3 does not show any significant difference between the three drying methods. Although the influence of the soaking period and the drying method not presented in Figure 4, however, it might not be seen because of the magnification factor only 40 times. Therefore it should be recommended to do the SEM evaluation with a significantly larger magnification factor such as 500 times to 2000 times.

According to the above recommendation, the SEM evaluation with magnification factor (M.F.), 500 times to 2000 times, was made to present the influence of concentration of the alkali solution (2 wt% and 6 wt%) during 2 hours soaking period with the sun-drying method. The surface morphology of untreated and alkali-treated coir fibre with magnification factor 500 times to 2000 times can be seen in Figure 5-Figure 7.

Figure 5. The surface morphology of specimen C2P2S: [a] MF (×500); [b] MF (×1000); [c] MF (×1500); [d] MF (×2000).
Figure 6. The surface morphology of specimen C6P2S: [a] MF (×500); [b] MF (×1000); [c] MF (×1500); [d] MF (×2000).

Figure 7. The surface morphology of specimen C0P0N: [a] MF (×500); [b] MF (×1000); [c] MF (×1500); [d] MF (×2000).

It can be seen in Figure 5 that the alkali treatment with the concentration solution 2 wt% during the soaking period of about 2 hours (C2P2S) able to smooth the surface morphology. Compared with the untreated one (C0P0N), Figure 7 shows that the alkali-treated coir fibre (C2P2S) has reduced the lignin and the hemicellulose on the surface area, see Figure 5. The texture of the cellulose wall of C2P2S was appeared smoother than C0P0N. It can be identified that the effect of alkali treatment has removed and cleaned the hemicellulose and the lignin on the fibre surface.

In the case of the alkali-treated coir fibre with the concentration solution 6 wt% (C6P2S), Figure 6 shows that the treatment has a significant influence on the cellulose surface. The cellulose wall texture of the coir fibre becomes coarse and rough. Similar to C2P2S, the lignin and the hemicellulose is also
not appear in the fibre surface of C6P2S. However, the cellulose wall of the C6P2S is looked rough while the wall of C2P2S is looked smooth. It is indicated that the alkali treatment can remove the lignin and hemicellulose on the coir fibre surface.

Furthermore, it can be concluded that the higher concentration of alkali able to influence the texture of the cellulose wall. It can be seen that the cellulose wall becomes a rough texture, see Figure 6d. The cellulose wall's coarser texture might be expected to provide a positive contribution, mostly when it is adopted as reinforced fibres of composite material. The improvement can be made by increasing the interfacial adhesion of the coir fibre to the polymer matrix.

5. Conclusions
The characterization results of the surface morphology of untreated and alkali-treated were concluded that alkali treatment significantly influences the fibre surface's texture. The SEM observation indicates that the lignin and the hemicellulose were removed and cleaned by the alkali treatment process on the surface region.

In the magnification factor 40 times, the soaking period between 1 hour and 2 hours not presenting significantly different visual results on the fibre surfaces texture. It is indicated that the soaking period has an optimum time length period, which is the longer soaking time will not have a significant influence on the changes of the coir fibre surface morphology. Although the results can be accepted theoretically, the observation result should be confirmed by the experimental study with a very large magnification factor, such as 500 times or 2000 times.

The concentration of alkali solution has a significant influence on the surface morphology of coir fibre. The higher concentration (6 wt%) able to make the fibre surface texture (cellulose wall) become coarser, while the lower concentration (2 wt%) generated the soft cellulose wall and also removed the lignin and hemicellulose on the surface region. The rougher fibre surface might be expected to improve the interfacial adhesion to the polymer matrix while being used as reinforced fibre in the composite material.

The effect of the drying methods was not captured by the observation using a magnification factor of 40 times. It might be indicated that the drying method does not have any significant influence on the surface morphology of coir fibre. However, to confirm the assumption, it is recommended that the more substantial magnification factor should be adopted for the next observation. The drying method's effect should also be evaluated on the mechanical properties such as the tensile strength, the Young modulus, and the maximum strain.

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