The effect of alkaline treatments soaking time on oil palm empty fruit bunch (OPEFB) fibre structure

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Abstract. The primary objective of this work was to study the effect of different soaking time in an alkaline solution for oil palm empty fruit bunch (OPEFB) fibre. For this purpose, the OPEFB fibres were treated with the sodium hydroxide (NaOH) at 5% concentration for ½ hour, 1-hour, 3-hour, 5-hour, 7-hour and 24-hours. The single tests of treated and untreated fibre were then performed in accordance with the ASTM D3822-07 standard. Next, the surfaces of the fibres prior and after the treatment were observed with a scanning electron microscope (SEM) TM3000. The result shows that at 7 hours of soaking time exhibits the highest tensile strength compared to the other soaking time and untreated fibre.

1. Introduction

The enthusiasm for utilising natural fibre, for example, another plant and wood fibre as support material in plastics have expanded significantly in a recent couple of years. Due of the light weight, high quality to weight proportion and decomposition resistance natural fibre based composites are getting to be necessary composite materials in building and designing areas [1]. Oil palm trees can develop well in a Malaysian atmosphere. Without a doubt, it has turned into the most critical rural product in Malaysia and has been the way to the national financial development [2]. The oil palm empty fruit bunch (OPEFB) is another potential natural fibre to be explored [3]. Just 10% of the aggregate biomass delivered in the estate comprises of oil, the staying 90% of the generation consists of large amounts of lignocellulosic material, for example, OPEFB, trunks and oil palm fronds [4]. In the process produce bunch remains as compost; OPEFB are mostly incinerated, which exposes to an ecological issue. These agriculture waste, particularly of OPEFB, can be changed to useful by-products utilising fibre based (natural fibre) composite as opposed to blazing the OPEFB. The structure of OPEFB comprises of three primary constituents; celluloses, hemicellulose and lignin. Steady holding among matrix and fibre can increase the composites mechanical properties. In order to achieve good consolidation between fibre and matrix, surface treatment on fibre is the common approach adopted via chemical treatments [5]-[8]. The common chemicals that used for surface treatments were sodium hydroxide, acetyl acid, and silane [9]-[14]. This research focuses on the effect
of soaking time during alkali treatment of the OPEFB fibre. The elastic properties; tensile, modulus of elasticity and morphological structure of the fibres are discussed.

2. Materials and methods
The oil palm empty fruit bunch (OPEFB) fibres are obtained from United Oil Palm Industries, Nibong Tebal, Pulau Pinang. The fibres were manually extracted hair by hair and continue to water retting process. The water retting process takes about one week. After a week, the fibre was cleaned using distilled water and dried under the sun to ensure removal of the moisture content of the fibres. The processed oil palm empty fruit bunch (OEFB) fibre was then treated with 5% aqueous solutions of NaOH at room temperature for 1/2, 1, 3, 5, 7 and 24 hours, maintaining the liquor ratio 40:1 to remove the hemicelluloses and surface impurities of the fibre. Finally, the fibres were cleaned using distilled water and dried at room temperature. The physical properties of the treated and untreated fibres are presented in Table 1. The measurement of the length and diameter were taken before the tensile test to ensure the properties of the specimens were maintained for analysis purpose.

Table 1. Physical properties of OPEFB fibre. (20 samples for each soaking time).

| Soaking time | Average diameter (mm) | Standard deviation | Average length (mm) |
|--------------|-----------------------|--------------------|--------------------|
| Untreated    | 0.52                  | 0.09               | 70                 |
| ½ hour       | 0.34                  | 0.05               | 70                 |
| 1 hour       | 0.30                  | 0.04               | 70                 |
| 3 hours      | 0.32                  | 0.04               | 70                 |
| 5 hours      | 0.33                  | 0.04               | 70                 |
| 7 hours      | 0.34                  | 0.04               | 70                 |
| 24 hours     | 0.34                  | 0.03               | 70                 |

The tensile strength of the fibre was determined through a single fibre tensile testing in accordance with the ASTMD3822-07 standard. The treated and untreated fibres were weighed using an analytical balance device, and the length of the fibre strand was measured microscopically. The surface and the diameter of the fibres were observed using Scanning Electron Microscope (SEM) TM3000. Prior to the test, the fibres were dried by using oven to the temperature of 60°C to 100°C about 30 minutes to an hour. The fibre was mounted onto a tab-shaped piece of paper with the gauge length greater than 50 mm. The tensile strengths of the treated and untreated fibres were then determined using an INSTRON 5848 Micro universal testing machine with a load cell of 2kN.

3. Result and discussion
The SEM micrographs of the surfaces of untreated and treated of OPEFB fibre before performing the single test are shown in Figure 1. The surface morphology of the fibre is important to observe the changes that happen on the surface of the OPEFB fibre because of the alkali treatment. The surface of the fibre after the treatment is found rougher than the untreated fibre. This is because the alkali treatment causes reduce in hemicellulose and lignin contents within the fibre thus make it more compact. The compact structure of the treated fibre will lead to making the treated fibre increase in tensile strength.

Figure 1 shows the stress–strain responses for selected of untreated and alkali-treated OPEFB fibres. The treated fibres exhibit a higher ultimate tensile stress because of the changes in the cellulose crystallinity. The graph shows that the 7 hours of soaking time have the highest ultimate tensile test.
The removal enables the fibril to rearrange them more compactly manner, thus enhancing the tensile strength of the fibre. Following the 3 hours, 24 hours, 1 hour, 5 hours, ½ hour of soaking time, there is an increase in the stiffness and this result in the increase in the tensile stress but a decrease in strain compared to untreated fibres.

![Figure 1](link-to-figure1.png)

**Figure 1.** Stress–strain responses for selected untreated and alkali-treated OPEFB fibre.

As shown in figure 2, it is seen that the highest strength obtained is at 7 hours of soaking time in 5% concentration of NaOH for OPEFB fibre with its 77.93 MPa. The second highest is for 24 hours, followed by 3 hours, 1 hour, untreated, ½ hour and 5 hours. The lowest strength is 43.42 MPa recorded for 5 hours of soaking time in alkali treatment. This indicates that the 7 hours of soaking time had increased almost 49% from the untreated fibre. The alkali treatment causes fibrillation, which is a process that causes the fibre bundle to break into smaller bundles. Smaller bundles help to distribute the load applied throughout the fibres.

![Figure 2](link-to-figure2.png)

**Figure 2.** The average tensile stress of OPEFB fibre (20 samples for each soaking time).

Based on figure 3, the highest average Young’s Modulus of OPEFB fibre recorded is 1.4 GPa for 5 hours of soaking time in 5% of NaOH. The lowest average modulus of elasticity, on the other
hand, is; after 1-hour treatment fibre with 0.67 GPa. Seeing these results, the alkali treatment indicates changes of the fibre elasticity concerning the soaking time applied during the treatment. Also, the elastic modulus remains below 1 GPa between specimens under 3 hours of treatment, registering between 0.67 GPa to 0.96 GPa. However, the fibre showed improved elasticity after being treated for more than 5 hours, recording at 1.4GPa, an increase of almost 65%. The modulus then indicates a slight reduction in elasticity with prolonged soaking times though higher than untreated fibres. Hence, the different modulus elasticity may result in the different strain to failure of specific fibre.

![Figure 3. Young’s Modulus of OPEFB fibre](image)

The SEM micrographs of the surface of the untreated and alkali treated OPEFB fibres are shown in figure 4. Figure 4 (a) shows the images of the untreated fibre. The white spots represent the presence of impurities on the surface of the fibre. Figure 4(b), (c), (d), (e), (f), and (g) represents the images of ½ hours, 1 hour, 3 hours, 5 hours, 7 hours, and 24 hours of soaking time respectively. Based on the observation, these alkalis treated OPEFB fibres showed cleaned surfaces due to the removal of impurities.
4. Conclusions
The mechanical properties of the treated and untreated OPEFB fibre with 5% NaOH aqueous solutions accompanied by different soaking times were investigated in this paper. It is shown that the OPEFB fibre is increased in their mechanical properties when treated in alkaline solution. The structure of the fibre also differences between the treated and untreated fibre. The result shows that the treated fibre exhibited better tensile strength whereas the 7 hours of soaking time is the highest compared to the other soaking time and untreated fibre. The surface observation through Scanning Electron Machine (SEM) shows the texture of the fibres achieved surface roughness after treating the OPEFB fibres. For future work, the surface roughness of fibre is an important factor to form it into composite since it promotes the best fibre-matrix bonding compared to untreated. The study further supported the feasibility of utilising OPEFB fibres as reinforcing materials in polymer composites.

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