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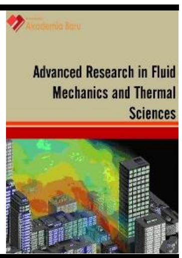

Properties of Chemically Treated Oil Palm Empty Fruit Bunch (EFB) Fibres

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ABSTRACT

Empty Fruit Bunch (EFB) fibres have been used in the production of many types of bio-composites, such as fibreboards and fibre-reinforced plastic composite. Hence, the understanding of the EFB characteristic is very important in determining the quality of the products. In this study, the physical, mechanical, anatomical and surface properties of the EFB fibres have been analysed, and the properties of two types of treated EFB fibres; 1) alkaline treated fibre, and 2) acid treated fibres were investigated. The fibres were treated with sodium hydroxide (NaOH) and acetic acid at different concentration levels (0.2, 0.4, 0.6 and 0.8%). The effects of the treatments towards the physical, chemical, mechanical and anatomical properties of EFB fibre after they were subjected to thermo-mechanical pulping (TMP) were studied along with the chemical composition, tensile strength and fibre morphology of the treated fibres. The results from the study indicated that the concentration levels of both NaOH and acetic acid had influenced the fibre properties.

1. Introduction

Agricultural wastes have become the alternative raw materials for fibre production. They have played an important role in the wood industry, especially during the recent global shortage of wood and forest materials. This shortage stems from the resistance towards deforestation and forest degradation, and the increasing demand for wood from wood-based industries [1]. In this light, the advantages of using these materials include their low cost, lighter weight, low density, recyclability, and a very high strength-to-weight ratio [2-4]. Oil palm (*Elaeis guineensis*) is a major export commodity for Malaysia and there were 5.84 million hectares of oil palm plantation area in 2018 [5].

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In the meantime, oil only represents about 10% of the total biomass produced from the oil palm tree while 90% of the biomass are residues [6] and it is estimated that the oil palm industry had generated more than 90 million tons of oil palm biomass [7].

Empty fruit bunch (EFB) is a potential resource for the production of composites. However, it has inherent properties that hinder the production of high-quality products, especially oil residues that are absorbed by fibres during their extraction in the mill. Rozman *et al.,* [8] reported that oil residue forms a layer on the outer surface of EFB fibre. In this regard, the residual oil has a marked influence on fibre wettability, glue penetration, and bonding properties while the gluing properties of EFB fibres are improved by removing the residual oil.

Khalil *et al.,* [9] reported that modified EFB fibres improve the mechanical properties and water resistance ability of EFB composites. As the mechanical properties of EFB composites are very important, treatment is crucial to obtain better fibre properties and produce high quality composites. The treatments can be physical, thermal, mechanical, or chemical, and they can be performed before, during, or after fibre processing. In principle, physical treatment does not make changes to the structural composition and only modifies the surface properties; such treatments include stretching, calendaring, hybrid yarning, electric discharging, ball-milling [10], and cold plasma treatment, meanwhile thermal treatments include boiling, UV heating, and hot air application. Furthermore, chemical treatments involving acid [11], alkaline [12], silane [13], and ethanol [14] modify the fibre chemistry to improve its mechanical performance [15].

Alkaline treatment, also called mercerisation, is a simple, economical, and effective treatment to remove lignin, wax, or oil on the external fibre surface, with only some fibre chemical modifications [16,17]. Alkaline treatment could improve the tensile properties of fibres [18,19], and increase the fibre surface free energy, surface area, and roughness by removing waxes, pectin, some lignin, and oil from the fibre surface. A study conducted by Ibrahim *et al.,* [20] found that tensile strength of kenaf fibre was improved when treated with sufficient amount of alkaline solution.

Sodium hydroxide (NaOH) is a commonly used alkaline treatment; NaOH modifies the interface between plant fibres and the matrix, which produces high quality fibres. By removing natural fats, waxes, and impurities from fibre surfaces, this treatment increases the reactive functional hydroxyl groups [21], as follows

$$\text{Cellulose-OH} + \text{NaOH} \rightarrow \text{Cellulose-O}^{-}\text{Na} + H_2O + \text{impurities} \tag{1}$$

Saiful Islam *et al.,* [22] observed that removing fibre surface impurities improves the surface roughness, leading to more available hydroxyl groups and other reactive functional groups. Moreover, Mwaikambo and Ansell [23] treated hemp, jute, sisal, and kapok fibres with a NaOH solution at different concentrations, and found that the optimum concentration for cleaning fibre bundles is 6% NaOH.

Oxalic acid, acetic anhydride, acetic acid, and nitric acid are generally used; acetic acid are commonly used for acid treatment due to their low cost. Osmani *et al.,* [24] found that acid treatment changed the chemical and morphological properties of pineapple leaf fibre which is important in composite production. Tengku Faisal *et al.,* [25] studied the effect of an acetic acid treatment on the mechanical, thermal, and morphological properties of coconut shell fibres. The study found that the acetic acid treatment improved the tensile strength, elongation at break, and the Young's modulus of the produced composites. Furthermore, the treated fibres exhibited better thermal stability and interfacial interactions compared to untreated fibres. Acetic acid treatment could also result in a rough surface topography with fewer voids, providing better mechanical interlocking with the matrix [26]. Rowell *et al.,* [27] investigated this acid treatment on several types of cellulosic fibres and reported that the treatment improved the moisture resistance properties, as explained by the removal of hemicelluloses and lignin from the treated fibre. Another study explored the use of acetic acid and xylanase to treat rice straw fibres with acetic acid [28]. The finding showed a decrease in the percentage of water absorption, as a result of the removal of hemicelluloses, which increased the crystallinity of the cellulose from 63.4% to 65.6%. However, eliminating the hemicelluloses and amorphous substances produced fine voids in the surface of the rice straw fibres.

This study focuses on the treatment of EFB fibres with NaOH and acetic acid at low concentration. The objective of the study is to determine the effect of treatments on the chemical composition, tensile strength, and morphology of the treated EFB fibres.

2. Methodology

2.1 Material

Shredded empty fruit bunches were obtained from Sri Langat Oil Palm Mill in Dengkil, Selangor, Malaysia. The initial moisture content of the shredded EFB was 110% (dry basis) and they were used directly after sampling.

2.2 Methods

2.2.1 Fibre treatments

The EFB shreds were soaked in NaOH and acetic acid solutions at different concentrations of 0.2%, 0.4%, 0.6%, and 0.8%, at room temperature for 24 h. The fibres were then filtered, washed with water to remove impurities, and air-dried for 5 h, then, the fibres were pulped and refined with a Sprout-Bauer refiner (Andritz, Graz, Austria) to produce single fibres.

2.2.2 Fibre morphology

About 20 g of EFB fibres were used from each treatment batch. The fibres were examined under a stereo light microscope (Leitz DMRB, Bachmuhlweg, Germany) that was attached to an image analyser (Olympus, Tokyo, Japan). The length, width, and lumen diameter of 500 randomly picked fibres were measured, and the aspect ratio (length/width) was calculated.

2.2.3 Chemical composition

The lignin, cellulose, and hemicellulose contents of the treated EFB fibres were determined according to ASTM D1106-96 [29], ASTM D1103-60 [30], and Zhang *et al.,* [31], respectively.

2.2.4 Scanning electron microscopy (SEM)

A Hitachi 3400 scanning electron microscope (Tokyo, Japan) was used to study the morphology of the fibre surface with the acceleration voltage at 15 kV. All of the samples were sputter-coated with gold prior to observation.

2.2.5 Mechanical strength tests

The tensile strength of a single EFB treated fibre was measured according to ASTM D3379-75 [32]. The diameter was measured at five points for each fibre, using an optical microscope with a calibrated eyepiece, and the average diameter was used to calculate the tensile properties of the fibres. An Instron Universal Testing Machine (Norwood, MA, USA) with a crosshead speed of 2 mm/min was used to determine the tensile strength of the specimen as shown in Figure 1. The mounted fibres were then carefully gripped by the testing machine, and cut with scissors along the cutting line as indicated in Figure 2.

Fig. 1. Universal Testing Machine for fibre tensile test

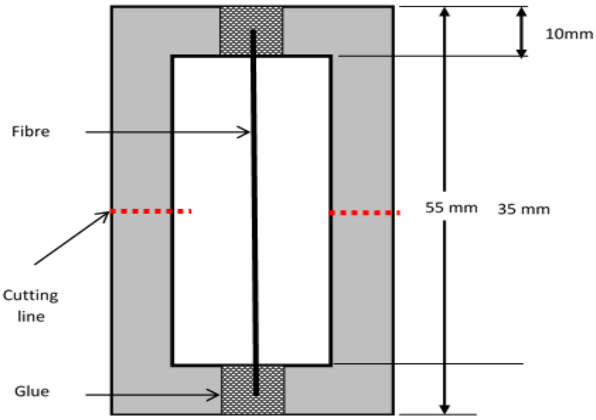

Fig. 2. Schematic representation of the experimental setup of tensile test

2.2.6 Statistical analysis

The data was statistically analysed using the analysis of variance (ANOVA) and the least significant different (LSD) method using the SAS software version 7 (Cary, NC, USA). The LSD method calculates the least difference that occurs between two means and compares them with the LSD value; the means are considered significantly different if $p < 0.01$ and ranked with different letters (a, b, c etc.) and are not significantly different with means ranked with the same letters if $p > 0.05$.

3. Results and Discussion

3.1 Fibre Morphology

Table 1 shows the average values of the fibre length, diameter, and aspect ratio of treated EFB fibres after the refining process. In general, fibre measurements declined with increased NaOH and acetic acid concentrations, with the highest reduction in length occurring in the fibres treated with acetic acid. Fibre length was not affected greatly by the concentration of the chemicals, although the type of chemical used did greatly influence the fibre characteristics. However, the reduction in fibre length was not significantly different when using a 0.2% concentration in both treatments. The aspect ratio of the fibre length to the fibre diameter decreased as the concentration of chemicals was increased.

Table 1
The length, diameter, and aspect ratio of treated EFB fibres

Treatment	Concentration (%)	Fibre Morphology		
		Length (mm)	Diameter (µm)	Aspect ratio
NaOH	0.2	1.23^{a}	15.91^{b}	79^{a}
	0.4	1.20^{a}	15.56^{b}	78^{a}
	0.6	1.16^{a}	15.48^{b}	77^{a}
	0.8	1.14^{b}	15.11^{c}	77^{a}
Acetic Acid	0.2	1.16^{a}	15.96^{a}	77^{a}
	0.4	1.13^{b}	16.02^{a}	75^{a}
	0.6	1.14^{b}	15.49^{b}	75^{a}
	0.8	1.06^{c}	14.92^{c}	68^{b}

*Note: Means followed by the same letters in each column are not significantly different ($p>0.05$) according to the least significant difference statistical test

Soaking the fibres in certain concentrations of NaOH and acetic acid prior to refining caused the cellulose to swell, and the separation of fibres during refining was enhanced when the hydrogen bonds between the cellulose molecules were broken. As the concentration of the NaOH solution increased, free hydroxyl groups within the cellulose molecule were subjected to substitution. At this state, the swelling of the cellulose are increase, and this affected the development of the fibres. The fibres experienced unravelling and peeling of the outer layers, while the cutting of fibres was observed during the process of refining [33]. The use of chemicals prior to refining had successfully swelled the cellulose and reduced the cellulose chain, which fragmented the fibres.

As shown in Table 1, the NaOH treatment produced longer fibres compared to the acetic acid treatment. As swelling agents, both NaOH and acetic acid penetrated the crystalline cellulose structure, resulting in higher swelling and contributing to greater fibre separation in refining. However, the fibre length was reduced greatly during the acetic acid treatment, indicating that severe cellulose hydrolysis had occurred during the reaction between the fibres and acetyl groups

[13]. Consequently, the reaction between the acetic acid and the fibres had increased the swelling, peeling, and shortening of the cellulose fibres during refining.

The diameter of the fibres was reduced for both chemical treatments from a concentration level of 0.2% to 0.8%, due to the modification of the fibre structures. Kabir *et al.,* [34] stated that chemical treatments change the orientation of the crystalline cellulose order and form an amorphous region, allowing more chemicals to penetrate the fibre structure. This process broke the hydrogen bonds and formed new reactive hydrogen bonds between the cellulose molecular chains. The hydroxyl groups were partially removed, and along with pectin, oil, lignin, and wax. This had reduced the fibre diameter and the aspect ratio [35].

A higher concentration of chemicals removed more fibre substances, which caused a higher reduction in the fibre diameter. This result is consistent with a study by Karthikeyan and Balamurugan [36], which stated that a higher NaOH concentration provides more Na+ and OH ions to react with the fibre substances, causing more lignin, pectin, wax, and oil to leach out. During the acetic acid treatment, a higher concentration caused more O-acetyl groups and OH ion reactions to take place, leading to the higher removal of these substances.

3.2 Chemical Composition

Table 2 shows the chemical compositions of the treated EFB fibres. In general, the cellulose content in the EFB fibres after treatments with NaOH and acetic acid were higher compared to the EFB fibres before the treatments. Conversely, the hemicellulose and lignin content were reduced after the treatment, with the hemicellulose content decreasing greatly compared to the lignin content. Moreover, in comparison to the acetic acid treatment, the NaOH treatment produced lower cellulose and lignin contents and higher hemicellulose content.

Table 2
Chemical composition of refined EFB fibres

Treatment	Concentration (%)	Chemical Composition		
		Cellulose	Hemicellulose	Lignin
NaOH	0.2	55.92^{a}	18.75^{b}	7.17^{a}
	0.4	57.03^{a}	15.53^{d}	6.89^{a}
	0.6	56.91^{a}	17.47^{c}	6.95^{a}
	0.8	58.93^{a}	22.75^{a}	7.03^{a}
Acetic Acid	0.2	63.65^{a}	13.97^{b}	7.42^{a}
	0.4	64.32^{a}	12.74^{d}	7.69^{a}
	0.6	63.57^{a}	13.59^{c}	7.49^{a}
	0.8	63.21^{a}	16.30^{a}	7.45^{a}
Untreated	0	53.37	19.88	10.74

*Note: Means followed by the same letters in each column are not significantly different ($p>0.05$) according to the least significant difference statistical test

The increase in the cellulose content in the EFB fibres after the chemical treatments is associated with the modification of the fibre structure. In this regard, the treatment disrupted hydrogen bonding in the fibre network, thus increasing the cellulose content at the expense of the crystalline cellulose. According to John *et al.,* [37], Na+ is able to widen the pores in the crystalline structures and penetrate them. This resulted in the formation of ONa-group from the converted structures of the OH-group of the cellulose, and expanding the dimensions of the molecules. Moreover, the NaOH treatment delignified the EFB fibre, thus exposing more cellulose and allowing the fibres to absorb more water molecules as the hydrophilic properties increased [38]. The high cellulose content in the fibres treated with acetic acid, compared to those treated with NaOH, was possibly due to the low acid characteristic that hydrolysed the cellulose while the similar concentration of NaOH induced better swelling of the EFB fibre, which reduced the cellulose. Meanwhile, a higher concentration of both chemicals could give contradictory results, like the presence of acetic acid could result to lower cellulose content compared to the NaOH treatment [38].

Both chemical treatments removed lignin and the hemicellulose from the EFB fibres. A fibre treated with 0.2% NaOH achieves the reduction of about 7% in the hemicellulose content and 35% in lignin content, compared to a 29% and 30% reduction, respectively, of the acetic acid treated fibre. This finding is also supported by a study from Shahriarinour *et al.,* [39], which found that the use of an acid solution was more effective in removing hemicellulose, while the use of an alkali solution is more effective for removing lignin. Mwaikambo and Ansell [23] also observed that after an alkaline treatment, hemp fibres showed the removal of wax and oil, a higher cellulose and a lower lignin contents.

3.3 Chemical Composition

The SEM image of the untreated EFB fibre surface clearly shows the presence of embedded silica bodies (Figure 3). Oil and other impurities were also observed on the untreated fibre surface.

Figures 4 and 5 show the changes in fibre surface morphology after both chemical treatments. The NaOH treatment reduced the visible amount of silica bodies, and the surface became rougher, as a result of the voids created by the removal of silica bodies. This result is in agreement with the study by Law *et al.,* [40], which used a gravimetric technique to show that EFB fibres treated with a chemical exhibited 28% silica removal. The SEM image of the fibre treated with 0.6% NaOH (Figure 4(c)) shows a rough surface with more exposed pores. At 0.8% NaOH (Figure 4(d)), the surface looks irregular with obvious bump fibres, which may be due to the swelling of the fibres.

The surface morphology of EFB fibres treated with acetic acid is shown in Figure 5. The removal of the silica bodies was more prominent in the acetic acid treated fibres than in the NaOH treated fibres. As a result, more voids were noticed on the surface of the fibre treated with 0.6% acetic acid (Figure 5(c)). One of the advantages of the removal of the silica bodies is that it enhances chemical penetration during the pulping process and exposes more amorphous regions of the fibres [40,41]. Consequently, composites with better properties are created; at 0.8% acetic acid (Figure 5(d)), the fibres had a rougher surface with some segmentation lines, indicating that hydrolysis and delamination had occurred. Many studies claim that increasing the surface roughness result in better mechanical interlocking, which improves the adhesion of fibres with the matrix [13,42].

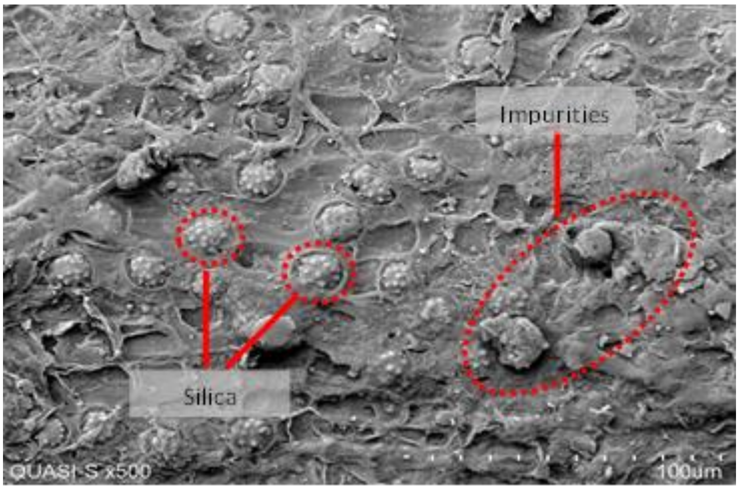

Fig. 3. SEM image of untreated EFB fibre at 500x magnification

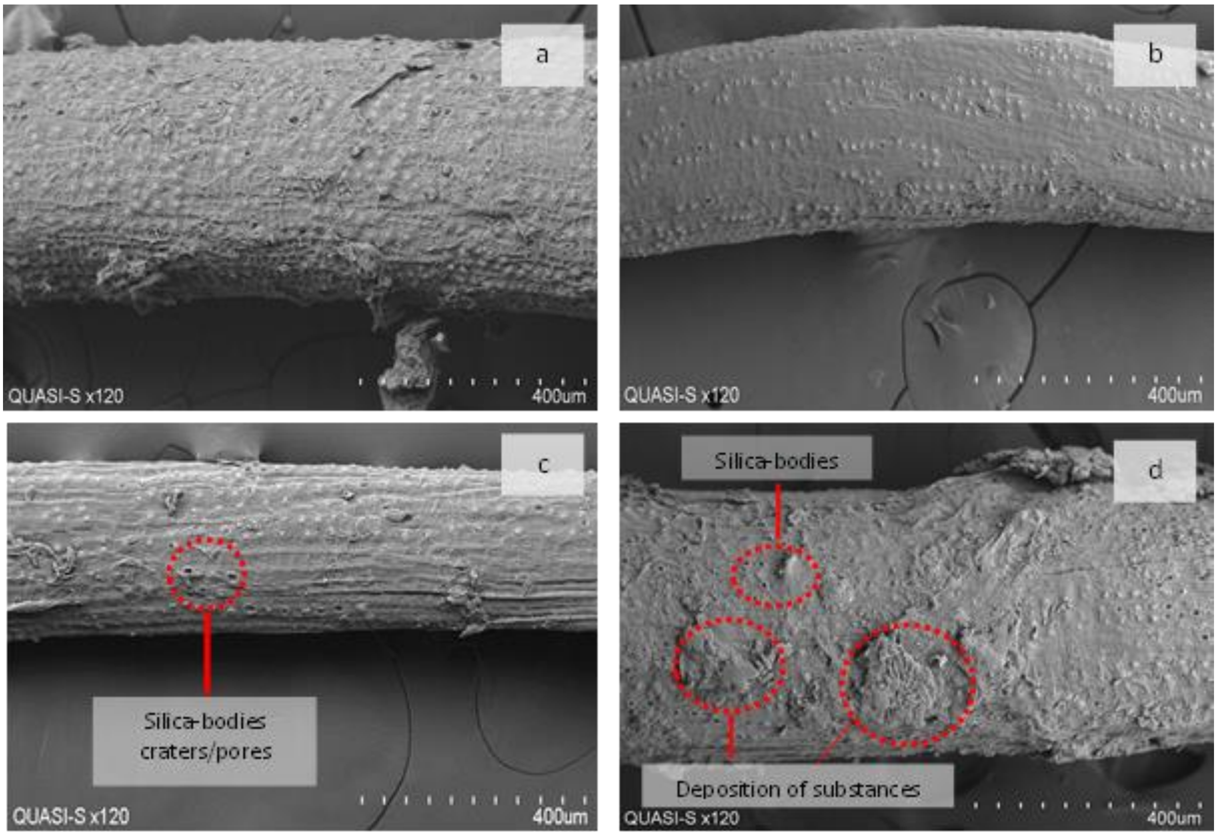

Fig. 4. SEM images of EFB fibre treated with (a) 0.2%, (b) 0.4%, (c) 0.6%, and (d) 0.8% NaOH at 120x magnification

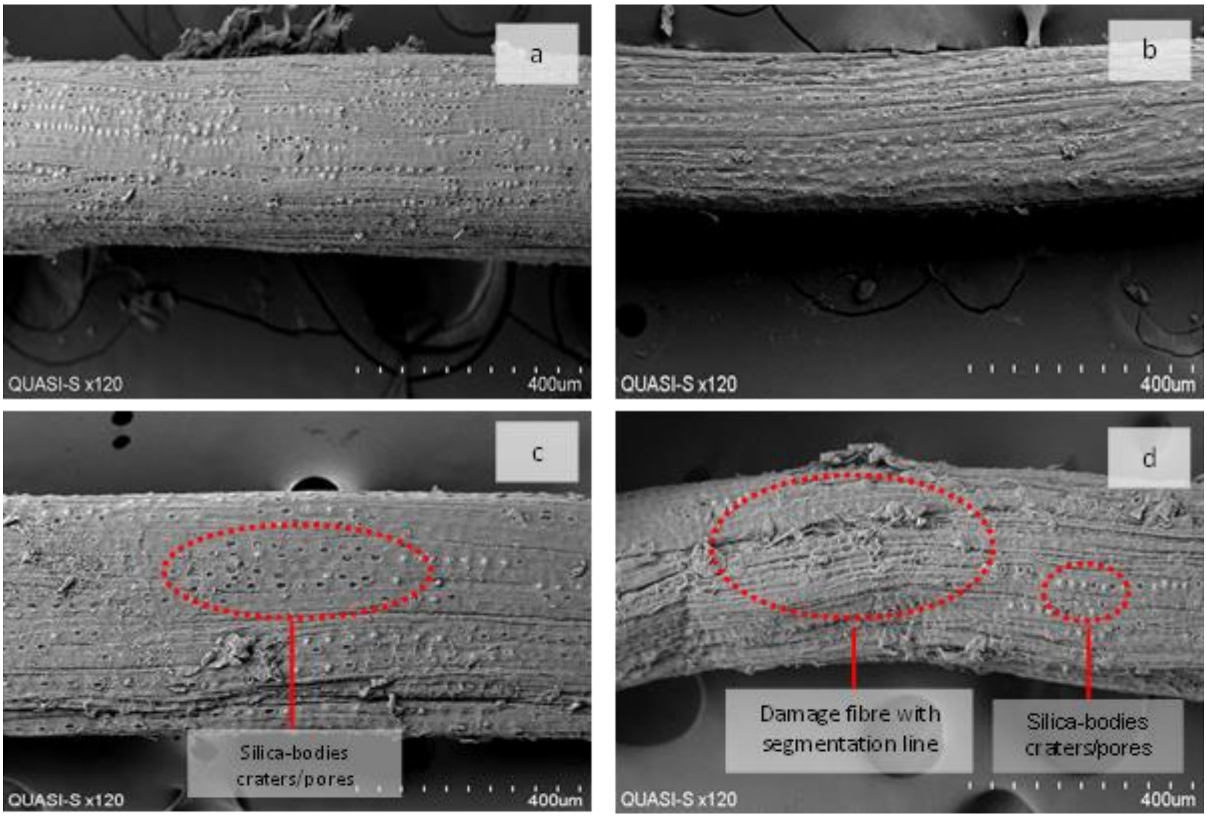

Fig. 5. SEM images of EFB fibre treated with (a) 0.2%, (b) 0.4%, (c) 0.6%, and (d) 0.8% acetic acid at 120x magnification

3.4 Tensile Strength

The tensile strength of the treated EFB fibres is presented in Table 3. The fibres treated with both chemicals had slightly increased in tensile strength with increasing chemical concentration. The highest tensile strength of 48 MPa was obtained from treatment with 0.8% NaOH, could be related to changes in fibre morphology, specifically the length and diameter. Rosa *et al.,* [43] mentioned that the fibre diameter affects the tensile properties, where generally the tensile strength increases when the diameter decreases. In this case, the diameter of the treated fibre decreased with the increasing NaOH concentration. Taha *et al.,* [19] claimed that NaOH treatments decreased the spiral angle and increased the molecular orientation of the cellulose chains, which improved the strength of date palm fibres.

In addition to the morphology changes, the chemical composition may also influence the tensile strength of fibres. Both Rosa *et al.,* [43] and Taha *et al.,* [19] found that increasing cellulose content caused an increase in the tensile strength of date palm fibres. Furthermore, Aji *et al.,* [44] reported that the tensile strength of a kenaf fibre increased with increasing cellulose content, which makes up the orientation of the cellulose microfibrils with respect to the fibre axis. Therefore, natural fibres are more ductile if the microfibrils have a spiral orientation to the fibre axis, and fibres are inflexible, rigid, and have a high strength if the microfibrils are oriented parallel to the fibre axis [45].

At 0.8% acetic acid, the tensile strength of the treated fibres was slightly reduced. This decrease could be due to the reduction of fibre stiffness after the acetyl groups was introduced to the fibre surface. In addition, the reduced fibre strength could be due to cellulose degradation during treatment at higher acid concentrations. A likely explanation is that the removal of the silica bodies increased surface roughness, including the cleavage line and the presence of pores, which caused an increased stress concentration and therefore, decreased the fibre strength [46].

Table 3
Mean value of tensile strength of treated EFB fibres

Treatment	Concentration (%)	Tensile Strength
NaOH	0.2	38^c
	0.4	39^c
	0.6	46^b
	0.8	48^b
Acetic Acid	0.2	48^b
	0.4	52^a
	0.6	60^a
	0.8	58^a
Untreated	0	35

4. Conclusions

The chemical composition, tensile strength, and morphology of the treated EFB fibres were affected by the chemical treatments. NaOH treatment produced fibres with a higher aspect ratio compared to acetic acid-treated fibres. Acetic acid treatment produced fibres with a low cellulose content and high hemicellulose content. Neither chemical treatment had an effect on lignin content, at any concentration. There were great differences in the surface conditions after the treatments. The acetic acid-treated fibre surface was rougher than that of the NaOH-treated fibres. At a higher NaOH concentration, the fibre surface became uneven and contained some residual substances that were deposited onto the surface, while the surface of fibres treated with acetic acid had a clear cleavage line. Surface conditions of treated EFB fibres affected the tensile strength of the fibres, where fibres treated with NaOH showed a low tensile strength, compared to fibres treated with acetic acid treated fibres.

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