Microstructural, Mechanical, and Physicochemical Behaviours of Alkali Pre-treated Oil Palm Stalk Fibres

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The effect of alkali pre-treatment (sodium hydroxide, NaOH) on the microstructural, mechanical, and chemical composition of oil palm stalk fibres (OPSF) is reported for future bioconversion processes. The OPSF was pre-treated with various concentrations of NaOH (5, 10, 20, 30, and 40% w/v). Scanning electron microscopy analysis revealed that 5% w/v alkali concentration caused complete removal of silica bodies and waxy layers, whereas pronounced degradation of the fibres occurred at 40% w/v NaOH concentration. Mechanical test results showed that the maximum elastic modulus of untreated OPSF was 2.5 GPa and the modulus was not sensitive to alkali concentration. Permanent set (plastic strain) and viscoelastic behaviours of OPSF were observed from the loading-unloading and stress relaxation test results, respectively. Agreement was observed between the Prony series viscoelastic model and test results, which provided further evidence of the viscoelastic behaviour of OPSF.

Keywords: Oil palm stalk fibres (OPSF); Pre-treatment; Sodium hydroxide (NaOH); Mechanical behaviours; Morphology; Lignocellulosic composition

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INTRODUCTION

Empty fruit bunches (EFB) are the largest source of lignocellulosic biomass from oil palm mills (Bahrin *et al.* 2012), consisting of 20 to 25% stalk and 75 to 80% spikelet (Han and May 2012). Currently, oil palm biomass has been exploited in various applications such as biocomposites (Kalam *et al.* 2005; Khalid *et al.* 2008), biosugar (Rahman *et al.* 2007; Shamsudin *et al.* 2012), biocompost (Yahya *et al.* 2010; Zainudin *et al.* 2013), biofuel (Chiesa and Gnansounou 2014; Ishola *et al.* 2014), and cellulose derivatives (Soom *et al.* 2009; Wanrosli *et al.* 2011; Nazir *et al.* 2013). Some of these applications, however, are hindered because of the presence of a waxy layer and silica bodies that are embedded on the surface of oil palm biomass (Omar *et al.* 2014a,b). As reported by Shalwan and Yousif (2014), the waxy layer and silica bodies reduce the contact between the fibre and matrix (*i.e.*, resin), thus affecting the fibre performance in biocomposite applications. On the other hand, the removal of silica bodies is crucial for the bioconversion of oil palm biomass, as it can open up the siliceous pathway and expose more of the amorphous region of the fibres, thus increasing the efficiency of the

hydrolysis process (Omar *et al.* 2014a). Apart from that, the bioconversion of oil palm biomass is also greatly affected by the lignocellulosic content of the fibres. For example, lignin reduces the hydrolysis rate by acting as a physical barrier, which hinders the cellulosic accessibility to the enzymes (Chaturvedi and Verma 2013).

Various pre-treatment methods have been proposed to alter the physical and chemical structure of the fibres, which will be beneficial for the fibres used in various applications as the main structural component or as the filler agent. According to Chen *et al.* (2013), alkali pre-treatment is one of the most widely investigated chemical pre-treatment methods and is frequently used to pre-treat agricultural residues rather than wood materials (Baharuddin *et al.* 2012). Various alkali reagents have been used for the pre-treatment process, but sodium hydroxide (NaOH) has been studied the most (Kumar *et al.* 2009); however, to the best of the authors' knowledge, no report on alkali pre-treatment using NaOH on oil palm stalk fibres (OPSF) is available.

It is still not clear if there is any change in the strength of OPSF after pre-treatment under various pre-treatment conditions. Norul Izani *et al.* (2013) reported that the tensile strength and elastic modulus of NaOH pre-treated fibres were higher than those of untreated fibres; however, these findings contradicted the results reported by Nishiyama and Okano (1998), who found that the tensile strength of the fibres decreased after alkali pre-treatment. Moreover, the exact mechanical behaviour of OPSF is still not clear; it can be either viscoelastic or viscoplastic, in which the former has been reported by Sreekala *et al.* (2001b). The effect of pre-treatment on the structural and mechanical behaviour of OPSF is important for the applications discussed above. For example, it would be beneficial to know the alkali concentration that causes silica bodies and the waxy layer to be removed from OPSF and the maximum concentration that causes pronounced stiffness and microstructure degradation of OPSF. This study, therefore, aimed to address the effects of alkali pre-treatment (NaOH) at different concentrations toward the microstructure, mechanical, and physicochemical behaviour of OPSF.

EXPERIMENTAL

Materials

Oil palm stalk fibres (OPSF) were collected from empty fruit bunches (EFB) after ripe fruitlets were removed for oil extraction. The samples were obtained from Besout Palm Oil Mill in Perak, Malaysia. The EFB was then kept in a controlled environmental condition of -20 °C to prevent fungal contamination. The spikelets were separated from the EFB stalks, and the OPSF were then separated manually from the stalk bundle. The fibres were washed with tap water and a 2% detergent solution to remove any residual oil and dust and then dried in an oven for 24 h at 105 °C (Ariffin *et al.* 2008).

A portable USB microscope (Dino-Lite AM 4113 series, Taiwan) was used to measure the diameter of the fibres. At least three measurements were taken at different cross sections of the fibre, and the final diameter reported was the average. The untreated stalk fibre has a wide range of diameters (*e.g.* 0.2 to 0.6 mm). In our observation, the stalk fibres diameter was in the range of 0.25 to 0.30 mm (for samples pre-treated with 40% w/v NaOH). Gunawan *et al.* (2009) have reported that the fibres with larger diameter (>0.5 mm) had void or holes inside, which could affect the measured elastic modulus. The elastic modulus increases with reduction of fibres diameter, which according to Gunawan *et al.* (2009) can be due to the void or holds inside the larger

diameter fibre. Similar findings were also reported by Omar *et al.* (2014b), where the SEM images showed no large voids in the EFB fibres with the diameter less than 0.40 mm. Therefore, in order to standardise the samples dimension and obtain reliable results, the diameter of the fibres was kept in the range of 0.25 to 0.30 mm (for all untreated and pre-treated samples), while the height of the fibres was kept constant at 50 mm.

Methods

Alkali pre-treatment

The OPSF from 10 different oil palm bunches after the threshing process were mixed and treated with 500 mL of NaOH at different concentrations (5, 10, 20, 30, and 40% w/v), with a fibre to NaOH ratio of 1:10 (g/ ml^{-1}) as described by Harun *et al.* (2013) with slight modification. The OPSF were soaked in NaOH solution for 30 min at room temperature and then autoclaved at 121°C, 15 psi for 5 min. The treated OPSF was washed several times with plain water until no traces of base could be detected. The fibres were then dried in an oven at 105 °C overnight

Morphological analysis

The surface morphology of the OPSF before and after pre-treatment with different concentrations of NaOH was observed using a scanning electron microscope (SEM; E-1010, Hitachi, Japan). The OPSF was cut into pieces in the range of 2 to 5 mm. Individual fibres were then mounted on an aluminium stub and sputter-coated with platinum prior to a morphological assessment. The scanning electron micrographs were obtained at an accelerating voltage of 15 to 25 kV.

Mechanical tests

Tensile tests were performed using a texture analyser (Texture Analyzer model TA-XT, Stable Micro System Ltd., UK) following the method of Omar *et al*. (2014b). The length of each sample for the tests was fixed at 50 mm. The fibres were glued (cynoacrylate adhesive) to a 'C-shaped' paper to make sure the fibres stayed aligned, as shown in Fig. 1a. Then, the paper with fibre glued on it was loaded to the testing machine with both ends carefully clamped using a specialised tensile grip. Uniaxial tension tests were performed by pulling each end of the sample in opposite directions at a constant crosshead speed (*i.e.*, 1 mm/s).

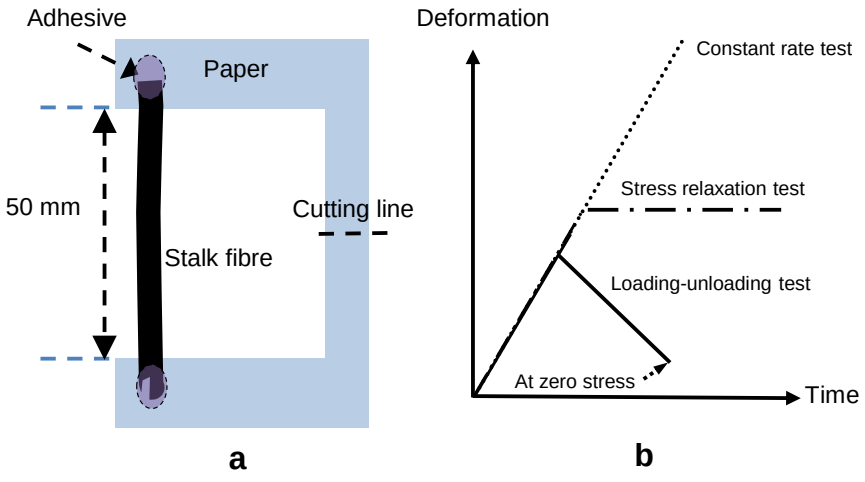

Fig. 1. (a) Sample setup for tensile test and (b) different deformation modes for the tensile tests

Loading-unloading and relaxation tests were performed at a constant crosshead speed of 1 mm/s and at a max deformation of 5 mm. The former test was conducted by loading and unloading the fibres under tensile mode (Fig. 1b). In comparison, the stress relaxation test was performed by stretching the fibre to a required strain, with the strain being held constant for a period of time while the stress decay was measured (Fig. 1b).

True stress, σ, was measured as: $\sigma = 4Fl/\pi D^2 l_0$; whereas the true (log) strain, ε, was calculated using: $\varepsilon = \ln(l/l_0)$; where F is the applied force obtained from the tensile test conducted, D is the diameter of the OPSF, and l and l_0 are the deformed length and gauge length, respectively. The fibre shape was assumed to be cylindrical based on the diameter measurements of the fibres at three different locations along the length of the fibre.

Viscoelastic model derivation

The viscoelastic model used in this work considers a separable time- and strain-dependent material behaviour for a homogeneous and isotropic material (Goh *et al.* 2004). The relaxation stress under a step strain loading history is defined as a function of time, $g(t)$, and strain, $\sigma_0(\varepsilon)$ through $\sigma(\varepsilon,t) = \sigma_0(\varepsilon)g(t)$. The time function is represented by the Prony series,

$$g(t) = g_\infty + \sum_{i=1}^{N} g_i \exp\left(-\frac{t}{\xi_i}\right) \tag{1}$$

where t and ξ_i are the time and relaxation time constants, respectively, and g_i and g_∞ are dimensionless constants. The Prony series consists of a series of Maxwell elements (springs and dampers) connected in parallel with a spring.

The total stress can be obtained using the Leaderman form of the convolution integral (Williams 1980), which is given by the algebraic sum of the entire past loading history at time t:

$$\sigma(\varepsilon,t) = \int_0^t g(t-s)\frac{d\sigma_0(\varepsilon)}{ds}ds \tag{2}$$

Combining the previous two equations using the numerical algorithm of finite time increments (Kaliske and Rothert 1997; Goh *et al.* 2004; Mohammed 2012) yields,

$$\begin{aligned}\sigma(t_{n+1}) &= g_\infty \sigma_0(t_{n+1}) \\ &+ \lambda(t_{n+1})\sum_{i=1}^{N}\left(\exp\left(-\frac{\Delta t}{\xi_i}\right)h_i(t_n) + g_i\frac{1-\exp\left(-\frac{\Delta t}{\xi_i}\right)}{\frac{\Delta t}{\xi_i}}\left[P_0(t_{n+1}) - P_0(t_n)\right]\right).\end{aligned} \tag{3}$$

where P_0 represents the nominal stress term, which is related to the true stress, σ_0 through: $\sigma_0(t_n) = P_0(t_n) \cdot \lambda(t_n)$. The term, $h_i(t) = \int_0^t g_i \exp\left(-\frac{t-s}{\xi_i}\right)\frac{d\sigma_0(s)}{ds} ds$ is updated at different time steps. The stress, σ_0, in Eq. 3 is obtained using a linear elastic equation as: $\sigma_0(t_{n+1}) = E_f \varepsilon(t_{n+1})$, where E_f and $\varepsilon(t_{n+1})$ are the fibre elastic modulus and true strain (at different time step) respectively.

Lignocellulosic composition

The cellulose, hemicellulose, and lignin contents were determined using neutral detergent fibre (NDF), acid detergent fibre (ADF), and acid detergent lignin (ADL) (Goering and Van Soest 1970). Neutral detergent fibre (NDF) represents a portion of fibres that contain cellulose, hemicellulose, and lignin. Acid detergent fibre (ADF) is a portion of fibres composed of cellulose and lignin, while acid detergent lignin (ADL) is the portion of fibres that only consist of lignin.

In general, the NDF content was determined by refluxing the fibres in a boiling neutral detergent. After being refluxed for one hour, the solution was cooled and filtered. The residues were washed with distilled water and acetone, before being dried. On the other hand, the ADF content was determined using a procedure like NDF, except that different detergent solution was used, which is known as acid detergent solution. The ADL analysis was then conducted on the residue fibres from the ADF analysis by using 72% w/v sulphuric acid. The fibres were washed, dried, and ignited in a furnace. The percentage of NDF, ADF, and ADL were calculated based on the initial and final weight difference. The percentages of cellulose, hemicelluloses, and lignin were then calculated using the following equations:

$$\text{Cellulose (\%)} = \text{ADF} - \text{ADL} \quad (4)$$

$$\text{Hemicellulose (\%)} = \text{NDF} - \text{ADF} \quad (5)$$

$$\text{Lignin (\%)} = \text{ADL} \quad (6)$$

RESULTS AND DISCUSSION

Microstructure Analysis

The microstructure of the OPSF is shown in Fig. 2. Silica bodies and a waxy layer covering the fibre can be seen in the untreated sample (Fig. 2a). The appearance of the silica bodies (Fig. 2b) is similar to those from previous experiments on oil palm fibres (Bahrin *et al.* 2012; Omar *et al.* 2014a,b). When the fibres were pre-treated with 5% w/v NaOH concentration, the protrusions and waxy layer were removed, leaving craters because of the absence of the silica bodies, as shown in Figs. 2c and 2d. This indicates that both the waxy layer and silica bodies are less resistant to alkali pre-treatment. The absence of these surface impurities (waxy layer and silica bodies) has been claimed to improve the fibre-matrix adhesion through better bonding or interface, as reported in previous studies (Norul Izani *et al.* 2013; Shalwan and Yousif 2014; Mahjoub *et al.* 2014). No significant microstructural changes were observed for fibres pre-treated by 10 to 30% w/v NaOH concentrations; however, at 40% w/v NaOH concentration, the

internal structure of the fibre was revealed (Figs. 2e and 2f), which indicates pronounced degradation of the fibre microstructure.

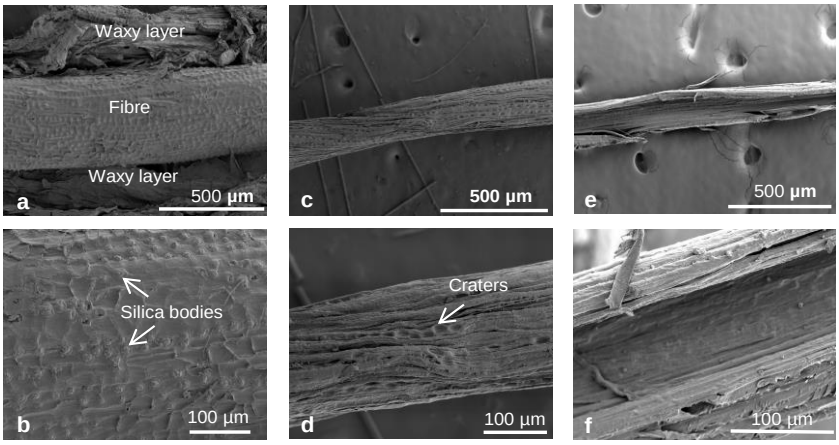

Fig. 2. SEM images of: (a) and (b) untreated fibre, (c) and (d) 5% NaOH concentration pre-treated fibre, and (e) and (f) 40% NaOH concentration pre-treated fibre

Lignocellulosic Composition

Table 1 shows the chemical compositions of the untreated and treated OPSF under various NaOH concentrations. The cellulose, hemicellulose, and lignin contents of the untreated OPSF were 23.7%, 35.9%, and 29.2%, respectively. The values reported in this study are comparable to the results of a study by Zaharah and Lim (2000), in which the hemicellulose and lignin contents of EFB stalk fibres were 28.7% and 28.1%, respectively (no cellulose content was reported). It is worth noting that to the authors' knowledge, these are the first reported results on the compositions of OPSF. In particular, the cellulose percentage increased with increasing alkali pre-treatment concentration. This is in agreement with other pre-treatment studies using palm oil fibres (Ariffin *et al.* 2008; Baharuddin *et al.* 2012).

Table 1. Cellulose, Hemicellulose, and Lignin Compositions of Untreated and NaOH-Treated OPSF

NaOH Concentration (% (w/v))	Cellulose (%)	Hemicellulose (%)	Lignin (%)
0	23.70 (1.31)	35.87 (1.13)	29.20 (0.33)
5	27.50 (0.67)	19.17 (0.38)	48.20 (0.43)
10	29.00 (0.71)	16.70 (0.99)	49.17 (0.31)
20	41.37 (0.33)	20.40 (0.86)	31.17 (0.24)
30	54.33 (1.23)	25.73 (1.03)	13.10 (0.65)
40	58.03 (0.83)	29.23 (0.17)	9.33 (0.45)

Standard deviation is enclosed in parentheses

The results suggested that alkali pre-treatment by NaOH is an effective pre-treatment method in exposing the cellulose on the fibre surface, which benefits the bioconversion process of cellulose stalk fibres. Results also showed that the lignin percentage was increased and hemicellulose percentage was decreased when the fibres were pre-treated at 5 to 20% w/v NaOH. This could be attributed to the removal of hemicellulose, which had less resistance to alkali degradation. The results obtained were in agreement with the findings of Nordin *et al.* (2013), who used superheated steam to

pre-treat the oil palm mesocarp fibres. Note that the complex structure of lignin which consists of Phenylpropane units joined together by different types of linkages makes it difficult to be degraded as compared to the branched structure of hemicellulose (Pérez *et al.* 2002).

Mechanical Behaviour

An example of the stress-strain curve of a single stalk fibre is shown in Fig. 3, which can be divided into elastic, plastic, and fracture regions, where the approximate boundaries between the regions are highlighted. Similar stress-strain curve patterns were observed for the stalk fibres pre-treated with various concentrations of NaOH. The linear elastic region suggested that no damage occurs within the stalk fibre when deformed under small deformation; however, in the plastic region, it is likely that damages within the fibre caused deviation from the elastic line. Omar *et al.* (2014a), for example, through a numerical study, showed that this is due to silica bodies and a fibre debonding effect. Finally, the sudden drop of stress at the fracture region suggested complete failure of the fibre.

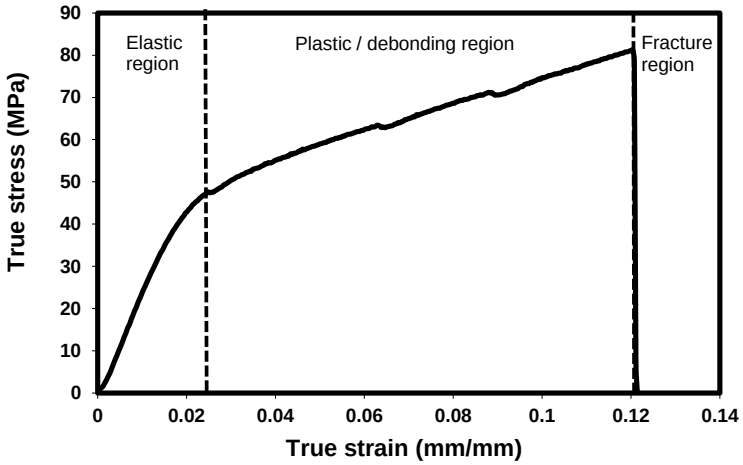

Fig. 3. A typical stress-strain curve of a single stalk fibre

The elastic modulus obtained from the initial slope of the stress-strain curve in the elastic region for the fibres pre-treated under different alkali concentrations is shown in Fig. 4. The maximum elastic modulus obtained in this study for the untreated OPSF was approximately 2500 MPa. This can be compared to other findings by Sreekala *et al.* (2001a), Jacob *et al.* (2004), and Norul Izani *et al.* (2013), with elastic moduli of oil palm single fibres of 1000 to 9000 MPa, 6700 MPa, and 2400 MPa, respectively. Except for with the 40% w/v NaOH concentration, minor changes in the elastic modulus were observed for samples pre-treated at various NaOH concentrations. This indicates that the elastic modulus of OPSF is not sensitive to pre-treatment using NaOH, which is in agreement with the results reported by Shalwan and Yousif (2014), which showed that NaOH concentration had no remarkable influence on the elastic modulus of oil palm fibres with fibre diameters of 0.3 and 0.5 mm; therefore, more mechanical tests were performed to further investigate the effects of alkali pre-treatment on the mechanical behaviour of OPSF, namely loading-unloading and stress relaxation tests.

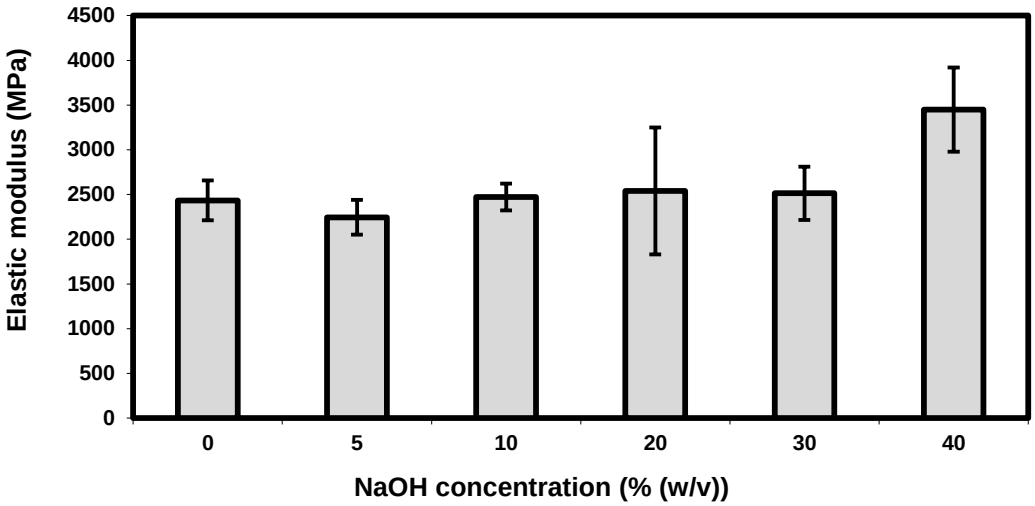

Fig. 4. Elastic modulus of OPSF pre-treated at various alkali concentrations. Data reported as the average ± standard deviation.

Loading-unloading tests were conducted in the plastic region (strain range 0.02 to 0.12 in Fig. 3). For this, the deformation used was 5 mm, which corresponds to a true strain of ~0.09. An example of the loading-unloading test results is shown in Fig. 5. For a perfectly elastic material, the unloading strain coincides with the loading curve; however, the elastic strain in Fig. 5 does not recover to zero at zero stress, indicating damage or plastic behaviour of the fibres. The strains at zero stress are divided into plastic and elastic strain, as shown in Fig. 5.

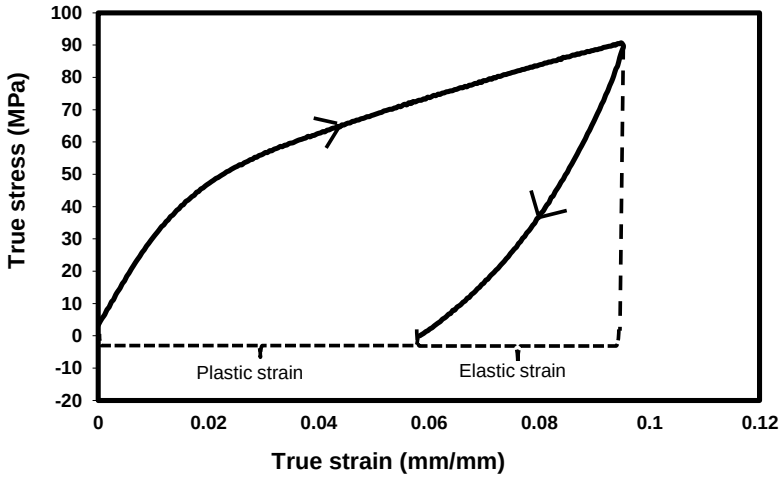

Fig. 5. Loading-unloading test results of OPSF at 5 mm deformation

Plots of the elastic and plastic strains for fibres pre-treated at various NaOH concentrations are shown in Fig. 6. When the samples were first treated with 5% w/v NaOH, a sudden reduction of elastic strain was observed, which in turn caused a sudden increase in plastic strain when compared to untreated samples. When compared to the microstructure observation (Figs. 2a and 2b), the changes in the strains may be due to silica body removal, as suggested by Omar *et al.* (2014a) through a numerical model. The changes may also be due to disruption of the chemical composition of OPSF during the alkali pre-treatment, as shown in Table 1. The presence of the major lignocellulosic

components and surface impurities (Figs. 2a and 2b) in the untreated fibres tend to keep the microfibrils in their original positions, as reported by Goda *et al.* (2006), which resulted in less plastic deformation in untreated OPSF. For samples pre-treated at higher alkali concentrations, minor changes in both strains were observed, where slight reduction of the plastic strain was observed at 30% w/v and 40% w/v concentrations of NaOH. Note that in addition to the plasticity behaviour of OPSF reported, Sreekala *et al.* (2001b) showed that oil palm fibres behaved as a viscoelastic material. Viscoelastic behaviour in this case refers to decay of stress at a constant applied strain when held over time.

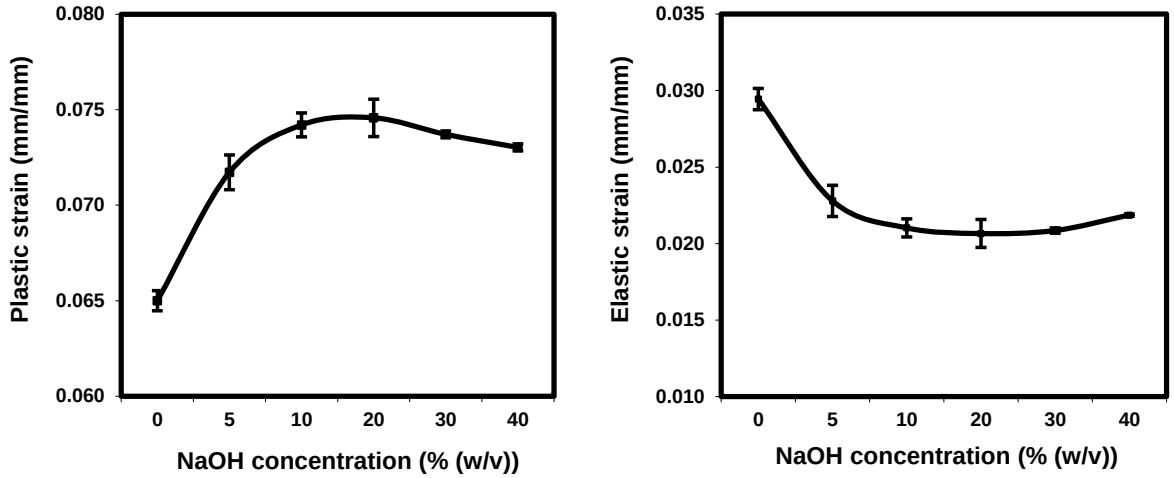

Fig. 6. Loading-unloading test results: (a) plastic strain and (b) elastic strain for OPSF pre-treated at various NaOH concentrations. Data reported as the average ± standard deviation.

To investigate the possible viscoelastic behaviour of OPSF, stress relaxation tests were performed under tensile mode. An example of a test result for treated OPSF (40% w/v NaOH concentration) at a deformation of 1 mm is depicted in Fig. 7.

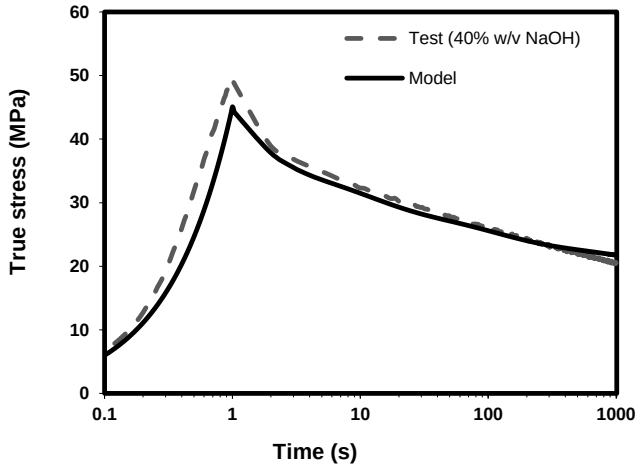

Fig. 7. An example stress relaxation test for the sample pre-treated at 40% w/v NaOH concentration and model fit using Eq. 3

From Fig. 7, viscoelastic behaviour is suggested by the steady reduction of stress at constant strain over time. The relaxation stresses at different times (*e.g.* 1 to 1000 s) for untreated and pre-treated OPSF at different NaOH concentrations are shown in Fig. 8. The stresses were reduced at a low NaOH concentration, but increased at a higher NaOH concentration for all time steps. The sudden reduction at a low concentration can be related to the absence of silica bodies (as seen from the SEM image in Fig. 2d) and the disruption of the cellulose-hemicellulose-lignin interface. The increase in relaxation stress at an NaOH concentration of 10 to 30% w/v is believed to be related to the higher cellulose content of OPSF (Table 1). This could be attributed to the fact that alkali pre-treatment increases the amount of cellulose exposed on the fibre surface (Valadez-Gonzalez *et al.* 1999), which can lead to greater packing of cellulose chains. Finally, reduction of relaxation stress of the fibres pre-treated at 40% w/v NaOH concentration can be related to further degradation of OPSF (Fig. 2f) and its cellulosic component.

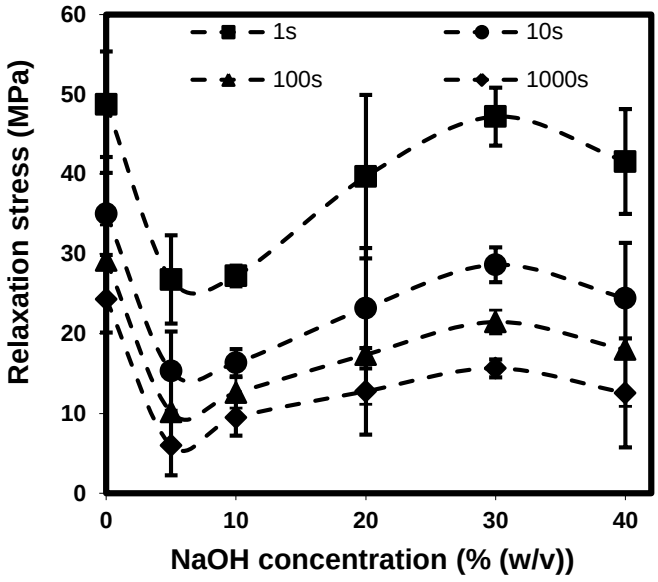

Fig. 8. Stresses at different relaxation times for untreated and NaOH-treated OPSF. Data reported as the average ± standard deviation.

It is worth noting that the relaxation stress results (Fig. 7) can be modelled using the Prony series viscoelastic model, which is available in commercial finite element software such as Abaqus (Abaqus 2009). Details on the model derivation are provided in the Experimental section. The viscoelastic model (Eq. 3) was fitted to the stress relaxation test data sample treated at 40% w/v NaOH concentration, as shown in Fig. 7, where the model parameters used are shown in Table 2.

The fibre elastic modulus shown in Table 2 was within the values of the elastic modulus test results displayed in Fig. 4, whereas the Prony series was fitted to the relaxation test data (1 mm deformation) using a least squares method (Goh *et al.* 2004); at this small deformation, OPSF are assumed to have minimal microstructural damage. The agreement observed between the model and test results provides further evidence of the viscoelastic behaviour of OPSF.

Table 2. Viscoelastic Model Parameters Used to Simulate Stress Relaxation Test Data for the Sample Treated at 40% w/v NaOH Concentration

OPSF elastic modulus, E_f (MPa)	3490
OPSF Prony series constants, g_i (at 0.1, 1, 10, 100, 1000, and ∞ seconds)	0.3, 0.17, 0.1, 0.08, 0.05, 0.3

CONCLUSIONS

1. An investigation of the effects of alkali pre-treatment using various concentrations of NaOH on the microstructure, chemical compositions, and mechanical behaviours of OPSF was performed in this study.
2. The SEM micrographs showed that 5% w/v NaOH was sufficient to completely remove the silica bodies and waxy layers from the OPSF, whereas pronounced degradation of the fibres was observed at a 40% w/v NaOH concentration.
3. Loading-unloading and relaxation tests showed the plastic and viscoelastic behaviours of OPSF, respectively. A Prony series viscoelastic model was developed and provided further evidence of the viscoelastic behaviour of OPSF.

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