

Modification of Oil Palm Empty Fruit Bunch and Sugarcane Bagasse Biomass as Potential Reinforcement for Composites Panel and Thermal Insulation Materials

Nor Azlina Ramlee¹, Mohammad Jawaid^{1*}, Edi Syams Zainudin², Shaikh Abdul Karim Yamani³

1. Department of Biocomposite Laboratory, Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

2. Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

3. Faculty of Applied Science Wood Industry Universiti Teknologi MARA, Campus of Jengka 26400 Bandar Tun Abdul Razak Jengka, Pahang, Malaysia

Abstract

This paper focuses on the study of the effect of modification of Oil Palm Empty Fruit Bunch (OPEFB) and sugarcane bagasse (SCB) biomass as potential reinforcement for composites panel and thermal insulation. Both fibres were treated with three types of chemicals: 2% silane, 4% H₂O₂ and 4% H₂O₂-2% silane for 3 h. The influence of modified fibres content in composites was examined by structural changes using image analyser, Fourier transform infrared (FTIR), Scanning Electron Microscopy (SEM), tensile, interfacial shear strength (IFSS) and thermal characteristic. The diameter of both fibres was reduced after treatment and showed decreasing of lignin and hemicellulose in fibre. Tensile strength has been increased by 2% silane treatment for both fibres and 4% H₂O₂ treatment displays higher result for IFSS. Thermal properties of treated SCB fibre with silane display higher residual content and better thermal stability. SEM characterization showed that 2% silane treatment removed silica bodies of OPEFB fibre while 4% H₂O₂ treatment uniformly filled porosity of SCB fibre. Finally, results revealed that treated OPEFB fibre is enough to improve compatibility and mechanical properties, while treated SCB fibre was effective in thermal stability for fabrication of composite materials.

Keywords: oil palm empty fruit bunch, sugarcane bagasse, fibre treatment, tensile properties, thermal properties, interfacial shear strength

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1 Introduction

Nowadays, a high attention about concerning global warming and depletion of petrochemical based material reserves have become interest to many researchers to focus on the use of natural fibres as an alternative material for various industrial components. The abundant of biomass from natural fibre residue has attracted researchers to develop biodegradable, recyclable and eco-friendly composites to solve current environmental issue. The natural fibres such as jute, kenaf, coir, sisal, hemp, bamboo, rice husk and flax^[1] provide many advantages to the composite materials due to their acceptable specific strength, lightweight, good thermal insulation, biodegradable, good acoustic properties and low cost^[2]. Previous study proved that natural fibres from agriculture biowaste have potential application in

aircraft, automotive, food packaging, and building sector^[3]. Scientifically previous researches noticed that oil palm fibres are porous, short in length and have varying diameter which affect the mechanical properties of composites^[4]. In addition, oil palm fibres have low cellulose content as compared to coconut and bagasse fibres, which makes it easy to do extraction process^[4].

Among the agriculture in Malaysia, sugarcane is one of higher demand in economic and productivity plant, that more than 700000 tonnes of sugarcane are produced per year^[5]. Abundant by-product from sugarcane known as sugarcane bagasse (SCB) is produced after extracting juice process and generates 40% wastes from sugar production^[5]. Generally, sugar is produced by crushing sugarcane in mills and the juice is squeezed out after the cane stalk broken the small size. It was reported that the SCB contains 60% to 80% of carbo-

*Corresponding author: Mohammad Jawaid

E-mail: jawaid_md@yahoo.co.in

hydrates made up by 22.02% to 44.3% cellulose, 24.5% to 29.34% hemicellulose and 18% to 22% of lignin^[6]. The components in sugarcane bagasse are obtained as low cost materials, high mechanical strength, long lasting and have good thermal insulations which are suitable and have high quality performance structural properties in order to making composite for building purposes^[7].

Recently, different pre-treatment methods for fibre modification such as physical, chemical or mechanical methods have been studied by researchers in order to select high or low performance of fibre compositions and mechanisms for different applications. Proper chemical pre-treatments have been applied to lignocellulose and apparently shown the improvement of interfacial adhesion between fibre and matrix; increasing the surface area or remove lignin and hemicellulose from the lignocellulosic matrix; rising incompatibility between hydrophilic of fibre and hydrophobic matrix that may affects to the adhesion of the fibre matrix^[8]. Researchers reported that silane treatment of fibre affects the surface morphology of fibres and increases fibre dimension whereas silane have a unique property to enhanced fibre/matrix interface bonding and modifying fibre orientation^[9]. Rachini *et al.*^[10] investigated the optimization on hemp fibres, which treated with silane agents before mixing with clay based materials for building materials applications, results showed that it improved the thermal degradation temperature.

Lately many studies have employed the common treatments for natural fibre such alkalization treatment in order to remove lignin and waxy substances^[11]. Faizi *et al.*^[12] have reported tensile properties of OPEFB fibres increased after treatment with 5% sodium hydroxide, NaOH for 7 h of soaking time. In other studies found that soaking time from 4 h to 12 h, dissolving ratio and temperature in alkaline treatment using extract of Oil Palm Empty Fruit Bunch (OPEFB) ash significantly affects the mechanical and physical properties of oil palm frond fiber reinforced composite^[13]. Fatra *et al.*^[14] studied OPEFB fibre treated with 5% NaOH alkaline solution for 12 h soaking time resulted highest flexural strength (30.21 MPa) of oil palm composites. Jawaid *et al.*^[15] found that OPEFB and jute fibres treated with 2-hydroxy ethyl acrylate (2-HEA) improved interfacial bonding with epoxy matrix, it enhanced flexural and

impact properties of treated hybrid composites. Furthermore, 4 h treatment of sugarcane bagasse with NaOH was applied in composites. The study reported that weight ratio 25/75 of sugarcane bagasse fibre: polypropylene was increased the tensile strength of composites to 24.92 MPa^[16]. The similar observation were found in the study of Acharya *et al.*^[17] that alkali treated bagasse/epoxy composites significantly improved the flexural strength of the composites. Moreover Mulinari *et al.*^[18] carried out combination of treatment with sulfuric acid, H₂SO₄ in sugarcane bagasse fibres, it was found that tensile modulus improved by 66.1% in composites which suitable for fabrication of automotive parts.

Particularly, due to specific characteristics of lignocellulosic material, several authors have focussed on the influence of fibre treatment on the thermal properties of composites for building, automobile, aerospace, packaging and furniture industry application. By the way, the proper use of thermal insulation in building becomes major concern due to thermal material contributes in reducing the annual energy consumption^[19]. A modification of the fibre with alkaline treatment increases 24% in thermal conductivity of hemp fibre^[20]. The thermal conductivity of oil palm fibre reinforced with phenolic resin is effective with silane surface treatment. It was found that the higher thermal conductivity of the silane treated composites, $0.59 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ is due to the high polarity of the silane treated fibre and to the Si-OH group in the treated fibre^[21]. The importance of increasing the thermal performance in building sector is also highlighted worldwide nowadays and becomes more interesting issue due to sustainability and energy efficiency.

Generally, thermal materials are purposely designed to reduce the transmission of heat flow, and thermal conductivity λ is defined as the steady state heat flow passing through a unit area of material expressed in $\text{W} \cdot (\text{m} \cdot \text{K})^{-1}$ ^[22]. Insulating panels from sugarcane by-product has higher thermal conductivity about $0.046 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ with density of $100 \text{ kg} \cdot \text{m}^{-3}$ ^[23]. The thermal conductivities of sunflower composite materials with different matrix: epoxy and binder were $0.1642 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ and $0.0728 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ ^[24], respectively. Previous studies evaluated the thermal conduc-

tivity of composite made from rice hull was between $0.0464 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ and $0.0566 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ with lower density $154 \text{ kg}\cdot\text{m}^{-3}$ ^[25]. The residues from leaves, petioles and bunches from date palm plant are considered as natural waste that have bigger potential to innovate bio composite material. Composite made of date palm fibre and gypsum was studied and the thermal conductivity of board is between $0.15 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ and $0.17 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ with density $753 \text{ kg}\cdot\text{m}^{-3}$ ^[26].

In this paper, we evaluate the potential of hydrogen peroxide (H_2O_2) and silane as alternative methods of fibre treatment due to limited study reported on weak acid and coupling agent in fibre modification. We study the mechanical, structural, morphological and thermal properties of untreated and treated OPEFB and SCB fibres in order to enhance high performance development of eco-friendly thermal insulation material and composites panel.

2 Materials and methods

2.1 Chemicals and materials

The chemical used in this research are Triethoxy(ethyl) silane (96% purity, Sigma Aldrich, Germany) and commercial hydrogen peroxide (H_2O_2) with 30% concentration (Evergreen Sdn. Bhd., Malaysia). For interfacial testing, phenolic resin in powder form was used (Chemovate Giringar, Bangalore, India).

2.2 Samples collection and preparation

Oil Palm Empty Fruit Bunch (OPEFB) was obtained from Malaysia Palm Oil Board (MPOB), Bangi and sugarcane bagasse (SCB) was collected from local market located at Banting, Selangor. The SCB fibre was initially immersed in clean tap water for 24 h, washed and rinsed with hot water. The purpose of this cleaning process is to remove the impurities and reduce sugar content in the sugarcane bagasse^[27]. SCB fibre was sundried naturally for 2 weeks until moisture content constant to 10% – 13%. The dried OPEFB and SCB fibres were cut to 5 cm – 10 cm for efficient pre-treatment size. The fibre was kept in dry container for subsequent fibre treatment process.

2.3 Soaking in chemicals

OPEFB and SCB fibres were treated with three

types of chemicals: 2% v/v silane, 4% v/v H_2O_2 and combination of 4% v/v H_2O_2 and 2% v/v silane (H_2O_2 -silane) for 3 h separately.

2.3.1 Hydrogen peroxide treatment

Raw of OPEFB and SCB fibres from the previous preparation were immersed and stirred continuously in 4% v/v H_2O_2 solution at room temperature for 3 h separately. The weight ratio of H_2O_2 solution per fibre content was fixed at 20:1. Next, the mixture was neutralized by distilled water for several times until no alkalinity detected and pH 7. The fibres were filtered and then dried in an oven at 80°C for 48 h. The fibres obtained from this treatment were designated as H_2O_2 -OPEFB and H_2O_2 -SCB fibres.

2.3.2 Silane treatment

Triethoxy(ethyl) silane 96% concentration was diluted into 2 wt.%. The solution was dissolved in water and was stirred for 10 min to ensure that silane solution was completely mixed well. OPEFB fibre was added in the solution and immersed for 3 h, and then it was washed by distilled water for several times. The treated fibres were oven dried at 80°C for 24 h. Repeat the same step for SCB fibre. The fibres obtained from this silane treatment were designated as Si-OPEFB and Si-SCB.

2.3.3 Combination of hydrogen peroxide and silane treatment

OPEFB and SCB fibres underwent H_2O_2 treatment first for 3 h. Then, the fibres were washed clearly until pH 7 and continue with treatment with silane with the same step above.

3 Characterization

3.1 Diameter measurement

Optical diameter characterization of single fibre was conducted at room temperature using Digital Image Analyser (SZX12, Olympus Matrix Optics (M) Sdn. Bhd. Petaling Jaya) machine. Using 1000 times magnification of the images, the outer diameter of single fibre was estimated. It is shown that the accuracy of the fibre diameter measurement can be enhanced by generating multiple point sources but the irregular shape of single fibre make impossible to achieve constant



Fig. 1 Measurement of fibre diameter.

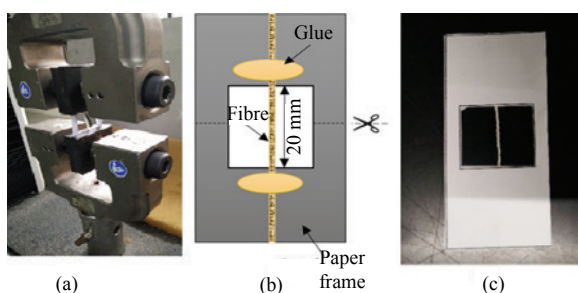


Fig. 2 (a) INSTRON 3365 machine; (b) schematic drawing designed paper frame; (c) fibre filament attached on paper frame.

measurement^[28] as shown in Fig. 1. Particularly, the unique properties of natural fibre composed by microfibrils of hemicellulose and lignin produces the un-informed size of fibre cross section. The diameter was measured at three different points. Six replicates of OPEFB and SCB was analysed for each different treatment and the average value was taken.

3.2 Tensile testing

Both untreated and treated OPEFB and SCB fibres were compared by strength. A single fibre tensile test was conducted using an INSTRON 3365 Dual Column Table Top Universal Testing Machine (UTM) as Fig. 2a based on ASTM D3379. Paper holder frame was designed as shown in Fig. 2b with 20 mm gauge length and this tests required to attached single fibre filament to a paper holder and glued it a shown in Fig. 2c. The frame sides were carefully cut in the middle part after clamping on the ends of the paper frame by the grips of the INSTRON machine. The total length of fibre used is approximately ± 70 mm. The maximum load is 5 kN with rate of speed $1 \text{ mm} \cdot \text{min}^{-1}$.

The tensile strength of fibres was calculated based

on calculation stated in standard:

$$T = F/A, \quad (1)$$

where, T tensile strength, MPa, F force to maximum stress in N and A average filament area, m^2 . The average of five replicates for each different treatment was recorded.

3.3 Fourier Transformed Infrared Spectroscopy (FTIR) analysis

The chemical changes and functional group of untreated and treated OPEFB and SCB fibres were analysed using Thermoscientific Nicolet 6700 Instrument with Attenuated Total Reflectance (ATR) capability method. A resolution spectra setting ranging are from 4000 cm^{-1} to 400 cm^{-1} .

3.4 Thermogravimetric analysis (TGA)

Mettler Toledo TGA/DSC 1 (Schwerzenbach Switzerland) analyser was used to evaluate the thermal stability of untreated and treated OPEFB and SCB fibres. Sample OPEFB and SCB fibres with ± 10.60 mg was placed in an alumina crucible and undergo to pyrolysis process with nitrogen ($30 \text{ mL} \cdot \text{min}^{-1}$). The temperature was setting between $30 \text{ }^\circ\text{C}$ to $500 \text{ }^\circ\text{C}$ and the heating rate $20 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$. TGA analysis is a technique that has been used to measure changes in the mass loss of sample that is subjected to changing of temperature in controlled atmosphere condition^[29].

3.5 Micro-droplet test

To analyse the interfacial shear strength between fibre and matrix, micro-droplet bonding test was conducted using a technique from previous studies^[30,31]. OPEFB and SCB fibres were fixed with designed paper properly. The microdroplet test was done by firstly applying a resin drop onto the surface of a single fibre, curing the fibre-resin to form the droplet and then applying a shearing force to pull the fibre out of the droplet. The diameters of the single fibres (D) and embedded length of the droplets (L) were measured using an optical microscope model Olympus SZX. Five replicates from each different treatment were carried out for tensile test using INSTRON 3365 Dual Column Table Top Universal Testing Machine (UTM). 5 kN maximum

Table 1 Length and diameter of OPEFB and SCB fibres

Fibres	Average length (mm)	Diameter (µm)			
		Untreated	Silane 2%	H ₂ O ₂ 4%	H ₂ O ₂ 4%-silane 2%
OPEFB	70 ± 0.201	472 ± 0.307	324 ± 0.401	364 ± 0.341	384 ± 0.307
SCB	60 ± 0.166	434 ± 0.384	256 ± 0.281	308 ± 0.311	315 ± 0.304

load and 0.5 mm·min⁻¹ speed rate was applied, and the analysis was conducted using the following equation^[30]:

$$\tau = F/(\pi DL), \tag{2}$$

where τ is the interfacial shear strength (MPa), F is maximum load before fibre pull out, D is diameter of single fibre and L is the length of embedded fibre in matrix.

4 Results and discussion

4.1 Length and diameter of OPEFB and SCB fibres

Surface modification of fibre was carried out in order to increase better interfacial adhesion between fibre and matrix and enhance mechanical properties of composite materials^[32]. Table 1 presents an average result of length and diameter of untreated and treated OPEFB and SCB fibres. OPEFB fibre recorded the higher average length 70 mm as compare to SCB fibre, 60 mm respectively.

The result shows that diameter of untreated OPEB and SCB fibres are larger than that of treated fibres. Both treated fibres were shows decrease trend due to different influence interface bonding between fibre and chemical attacked. It was clear from results that 2% silane treatment displays effective treatment for both fibres which performed high removal of lignin and hemicellulose. The movement of the liquid in the cell wall occurs mainly at the elementary fibril level of hemicellulose and contraction and swelling processes also mainly occur at this fibre surface part^[33]. Results show that treatment by 4% H₂O₂ for both fibres are higher than 2% silane treatment. This finding related due to increase the concentration of fibre could make fibre more brittle and weak^[34].

4.2 Tensile properties

The average tensile strength of untreated and treated OPEFB and SCB fibres are presented in Fig. 3. OPEFB and SCB fibres treated by 2% silane show

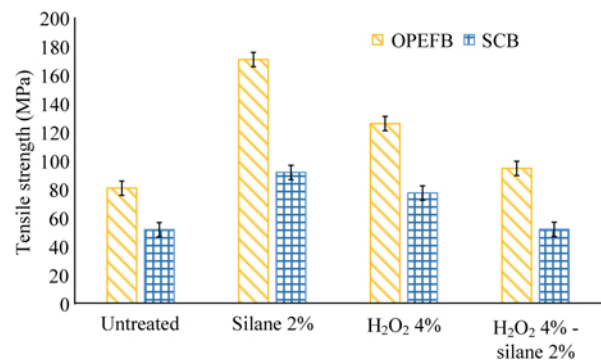


Fig. 3 Tensile strength of OPEFB and SCB fibres.

slightly higher tensile strength as compared to other treatments. In this study, we found that tensile strength of OPEFB fibre was increasing around 120% (170.96 MPa) after treatment by silane as compared with untreated (80.92 MPa). These siylation modifications are categorized as most effective treatment. The similar finding reported by Sreekala *et al.*^[35] in silane treated OPEFB fibre, which show maximum tensile strength, 273 MPa as compared to untreated 248 MPa and alkali treatment.

OPEFB fibre treated by 4% H₂O₂-2% silane showed less strength than OPEFB fibre treated with 4% H₂O₂, which indicates same trend with SCB fibre treated by 4% H₂O₂ show higher value compared than H₂O₂-silane. Hydrogen peroxide help to the improvement in physical appearance, and fibre surface treatment can also enhance the mechanical performance of polymeric composites^[32]. From the graph it clearly shows similar tensile strength for SCB fibre treated by H₂O₂-silane and untreated. Here, it can notice the combined chemical treatment is not an effective treatment for SCB fibre due to increasing time treatment and high concentration affect the strength of fibre. This result is probably due to the mixture of both chemical does not give any effect of SPF fibers^[36]. Previous study proved that hydrogen peroxide pre-treatment requires less time to have high cellulose and less hemicellulose^[37]. The tensile strength of OPEFB and SCB fibres after

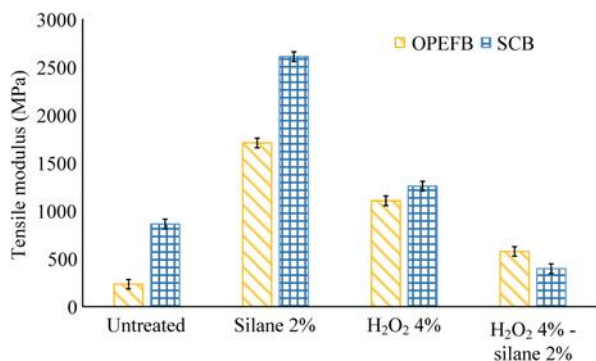


Fig. 4 Tensile modulus of OPEFB and SCB fibres.

treatment with H₂O₂-silane are very less improvement among the treatment. This finding are similar results with previous studies were reported combination of chemical NaOH-silane in sugar palm fibre^[31] and kenaf and sisal treatment had a lower tensile strength^[38].

The tensile modulus behaviour of single OPEFB and SCB fibres are shown in Fig. 4. Chemical treatment with silane 2% indicates maximum tensile modulus among all treatment. The tensile modulus of SCB fibre treated with silane 2% increased three times compared to untreated SCB whereas tensile modulus of OPEFB fibre increase from 234 MPa to 1709 MPa with silane 2% treatment. OPEFB fibre with 4% H₂O₂ treatment showed 15% and SCB fibre showed 40% less than treated with silane solution. However, tensile modulus of H₂O₂-silane displays low tensile modulus. This finding is consistent with the previous study reported the tensile modulus after treatment of fibres, it may be after treatment, hydrogen peroxide etching and silane application reduced the bond strength^[39] and due to high concentration of solution.

4.3 Fourier Transforms Infrared Spectroscopy (FT-IR) Analysis

The FTIR spectroscopy is employed to study the functional groups and tracking the chemical changes in before and after treatment of fibres structures. In this work focusing to discuss on cellulose, hemicellulose and lignin molecular structure, which has the most influence on the mechanical characteristics of fibres because of its high tensile strength. FTIR spectra were recorded with wavenumber range from 400 cm⁻¹ to 4000 cm⁻¹. The result for OPEFB and SCB fibres FTIR analyses are shown in Figs. 5 and 6. Peak assignments based on

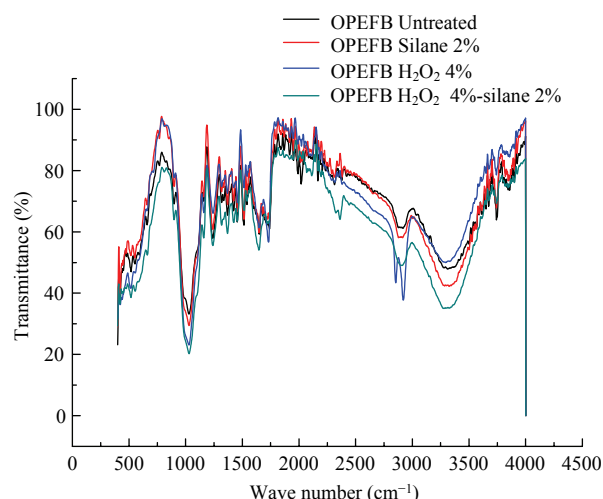


Fig. 5 FTIR spectrum of OPEFB fibre.

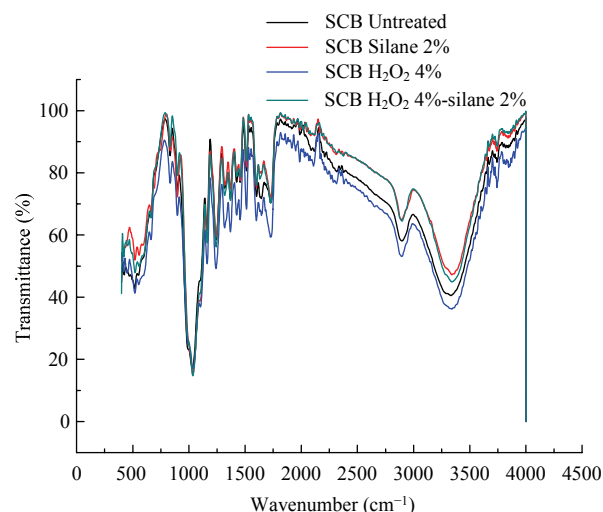


Fig. 6 FTIR spectrum of SCB fibre.

literature are listed in Table 2 for OPEFB fibre and Table 3 for SCB fibre.

The spectra bands of OPEFB fibre are around 3279 cm⁻¹ to 3324.66 cm⁻¹ region corresponds to the strong band's vibration of O-H stretching absorption. Table 3 shows band appeared in OPEFB fibre after treated with silane increase the wavenumber and assigned to increase the cellulose content compared with untreated. The interpretations of this spectrum were supported by the works of Siyamak *et al.*^[3] stated that broad band around 3400 cm⁻¹ regions corresponds to the stretching of the O-H bond of cellulose molecules and water absorbed by fibre. The spectrum exhibits CH₂ stretching around 2900 cm⁻¹ assigned to asymmetric stretch vibration. The peak at 1732 cm⁻¹ is associated

Table 2 Band assignment for oil palm empty fruit bunch fibre components

Peak assignment	Sources	Reference	Untreated	Silane 2%	H ₂ O ₂ 4%	H ₂ O ₂ 4%-silane 2%
			Wavelength (cm ⁻¹)			
O–H stretching	Cellulose	Ref. [40]	3310.70	3324.66	3293.61	3279.25
CH ₂ valence vibration hydrogen bonded	Cellulose	Ref. [40]	2918.39	2915.36	2920.82	2906.16
C=O stretching	Hemicellulose, Polysaccharides	Refs. [41,42]	1739.34	1714.84	1731.12	1724.03
C–H in plane deformation CH ₂ scissoring	Lignin	Ref. [42]	1421.21	1424.74	1421.67	1425.01
C–H deformation vibration	Cellulose	Ref. [43]	1335.81	1335.66	–	–
O–H blending of alcohol groups	Carbohydrate	Ref. [44]	1318.02	1317.50	1317.15	1317.59
C=O stretching	Lignin	Ref. [45]	1238.33	1237.94	1240.64	1238.55
C(1)–H deformation, ring valence vibration	β -glycosidic linkage	Ref. [44]	896.86	897.76	897.87	897.24
C–H out of plane deformation in positions 2,5,6	Lignin	Ref. [44]	849.00	847.83	813.04	811.08
CH ₂ vibration in cellulose I α	Cellulose	Ref. [46]	768.56	769.57	–	–
C–O bending out of plane	Cellulose	Ref. [47]	659.59	659.64	–	661.39

Table 3 Peak assignments of sugarcane bagasse components

Peak assignment	Sources	Ref.	Untreated	Silane 2%	H ₂ O ₂ 4%	H ₂ O ₂ 4%-silane 2%
			Wavelength (cm ⁻¹)			
O–H stretching	Cellulose	Ref. [48]	3330.93	3339.93	3340.87	3343.25
C–H vibration	Cellulose	Ref. [49]	2896.44	2895.78	2891.63	2896.58
C=O stretching	Hemicellulose	Ref. [48]	1726.55	1724.83	1725.37	1726.93
C=C stretching	Lignin	Ref. [50]	1644.72	1645.10	1645.68	1631.95
C=C stretching	Lignin	Ref. [49]	1512.65	1512.24	1513.17	1512.77
CH ₂ symmetric bending	Cellulose	Ref. [49]	1425.63	1425.40	1426.03	1427.30
CH stretching	Lignin	Ref. [49]	1370.40	1370.82	1370.39	1370.91
C–O–C asymmetric deformation	Cellulose and hemicellulose	Ref. [51]	1159.55	1161.03	1161.26	1162.42

with C=O stretching of the carbonyl group. This peak can be attributed to the uronic acids of the xylan in hemicelluloses^[40]. Structural changes in lignin and loss of aromatic units of OPEFB fibre were shown by the intensities in the changes in the 1646 cm⁻¹, 1514 cm⁻¹ and 1421 cm⁻¹ band. IR intensities at band wavenumber 1335 cm⁻¹ was assigned to C–H deformations in cellulose and hemicellulose. From Table 2, it clearly shown that intensity of this band was disappearing after H₂O₂ and combination of H₂O₂-silane pre-treatment. It could be related to the higher loss of hemicellulose content. This finding is similar with previous studies reporting a higher decrease and loss of hemicellulose and cellulose content after phosphoric acid pre-treatment^[40]. The band at wavenumber 1238 cm⁻¹ is due to the C=O stretching of the aryl group in lignin. The increase intensities seen at peak 1240 cm⁻¹ after treatment with H₂O₂. The peak at 1028 cm⁻¹ is due to C–O deformation in primary alcohols and C=O in secondary alcohol was assigned to lignin and there is no changes for untreated and treatment OPEFB fibre. In the frequency range 890 cm⁻¹. All

the treated OPEFB fibre sample showed increasing C–H–O stretching of the β -glycosidic linkage than untreated at wavelength 896 cm⁻¹. Deformation of C–H and lignin only occurred after treatment process at wavelength around 847 cm⁻¹, 813 cm⁻¹, and 811 cm⁻¹ and silane treatment shows higher peak respectively. From Table 2 shows there is no CH₂ vibration in cellulose I α after treatment by hydrogen peroxide and both chemicals treatment. Wavelength 769 cm⁻¹ for cellulose I α showed constant intensities after treatment with silane solution. However, the region 659 cm⁻¹ indicates C–O bending but hydrogen peroxide treatment was removed the cellulosic contents at this bands and might due to higher concentration of solution.

Table 3 shows the absorption spectrum for the SCB fibre presents some similarities with OPEFB fibre. At 3310 cm⁻¹ the band is related to the O–H stretching and wavenumber 2896 cm⁻¹ observed absorption band is axial deformation of C–H vibration where indicates cellulosic contents. Fig. 6 shows FT-IR spectrum of SCB fibre. The peak 1726 cm⁻¹ belongs to carbonyl band

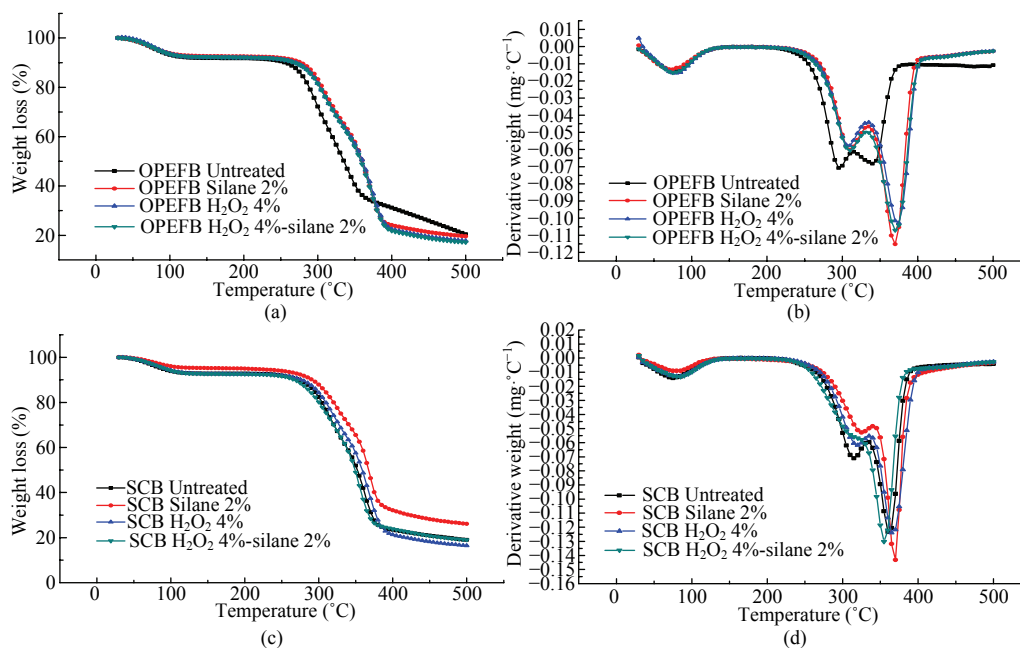


Fig. 7 A plot of (a) TGA (b) DTG thermograms of untreated and treated OPEFB single fibre and (c) TGA, (d) DTG thermograms for untreated and treated SCB single fibre at heating rate $20\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$.

C=O stretching of the hemicellulose in the SCB fibre. The lignin group gives overlapping signals at 1644 cm^{-1} and 1512 cm^{-1} due to C=C stretching. The region at 1425 cm^{-1} is corresponding to the symmetric bending deformation of CH_2 group of cellulose, while the band at 1243 cm^{-1} refers to the C–O–C in cellulose chain. The spectrum band at 1159 cm^{-1} is in connection with the asymmetric deformation of C–O–C of the cellulose and hemicellulose^[48].

4.4 Thermal properties of OPEFB and SCB fibres

Fig. 7 shows the thermogravimetric (TG) and derivative thermogravimetric (DTG) thermograms graphs of untreated and treated OPEFB and SCB single fibres. From the TG graph in Fig. 7a, it clearly shows a small weight loss for all OPEFB samples occurs between $40\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$ at the first stage. Probably, the same trend resulted 5% to 10% loss of weight occurred on the first region of OPEFB fibres due to evaporation of water content. These changes attributed to the removal of absorbed water in cellulose^[52]. At the second stage, it can be observed the thermal degradation of OPEFB single fibre process obtained extremely slope high range around $260\text{ }^{\circ}\text{C}$ and final degradation at $400\text{ }^{\circ}\text{C}$. This is due to high degradation of hemicellulose and cellulose and the cleavage of the glycosidic linkages of cellulose

which reduces the polymerization degree leading to the formation of CO_2 , H_2O and a variety of hydrocarbon derivatives^[53]. Untreated OPEFB fibre shows decompose earlier than treated fibre at temperature $245\text{ }^{\circ}\text{C}$. The third region indicates temperature over than $400\text{ }^{\circ}\text{C}$ and related to the mass loss caused the decomposition of lignin content. Fig. 7c shows three thermal degradation steps of SCB fibre. The moisture was released on the first stage with minimum temperature between $40\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$. The second stage contributes the large mass loss part of thermal decomposition hemicellulose and cellulose occurred around. Final stage occurred within temperature $360\text{ }^{\circ}\text{C}$ to $400\text{ }^{\circ}\text{C}$ where related to lignin content. Based on previous studies, it was shown that TG curve of SCB fibre and other natural biomass presented similar behaviour with the TG curve obtained in this work^[54]. These behaviour and alterations possibly are due to destruction of molecular structures and linkages of cell walls after treatment^[55]. In comparatively, DTG profile curves in Figs. 7b and 7d shows initial and final decomposition temperature of thermal stability. This is might be probably due to the difference affected of the fibre surface structure from the different chemical used in pre-treatment. However, from the Table 4 show the treated sugarcane bagasse fibres with silane 2% display higher residual content 26.18% compare to others and

Table 4 Thermal properties of untreated and treated of OPEFB and SCB single fibre

Samples	Transition temperature (°C)		Residual weight (%) at 500 °C
	$T_{initial}$	T_{final}	
OPEFB Untreated	245	365	20.41
OPEFB Silane 2%	270	405	19.59
OPEFB H ₂ O ₂ 4%	260	395	17.69
OPEFB H ₂ O ₂ 4%-silane 2%	265	380	17.17
SCB Untreated	260	365	18.99
SCB Silane 2%	290	420	26.18
SCB H ₂ O ₂ 4%	270	385	18.24
SCB H ₂ O ₂ 4%-silane 2%	265	370	19.04

have better thermal stability. The pre-treatment process and increasing the heating rate influencing the result on mass loss rates but the start of thermal decomposition of sugarcane bagasse become delayed to the higher temperature^[56]. By comparing to OPEFB fibre, it noticed there is second highest of remaining residual 20.41% but the degradation process occurred fastest within the temperature 245 °C to 365 °C. From this study, we expected treated fibres could build a better interface than the non-treated fibres.

4.5 Droplet test

The structural of fibre-matrix can be determine by having interfacial shear strength test (IFSS) in order to identified the affected of fibre-matrix condition of the fibre surface layer. Untreated and treated of OPEFB and SCB fibres were investigated by interfacial shear strength test as demonstrate in Fig. 8. Fig. 8a clearly shows fibre with the adhesion droplet under microscopic view before test. These figures indicate the micro-droplets are exploitable for pull-out testing. For Fig. 8b showed breaking part fibre is pulled out from matrix on the same fibre after test. Fibre was cracked and pulled out from the droplet after 5 kN load applied.

Fig. 9 showed the highest IFSS improvement in OPEFB fibre treated with 4% H₂O₂, 6.32 MPa then followed by fibre treated with 2% silane solution. This result was probably due to lignocellulose fibre become more hydrophobic after treatments and elimination of lignin occur in surface layer^[28,57]. Other than that, previous researcher reported natural fibre can be treated and effectively with hydrogen peroxide which normally used in textile industry^[58]. But only a few studies have reported about the effect of this treatment on the

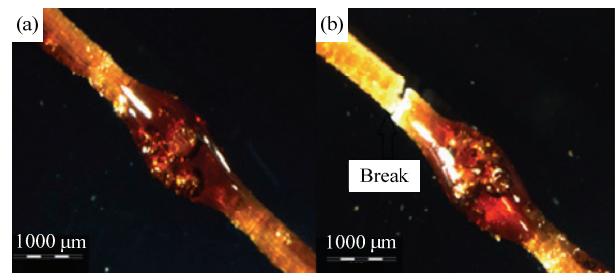


Fig 8 (a) Before test; (b) breaking part after IFSS test.

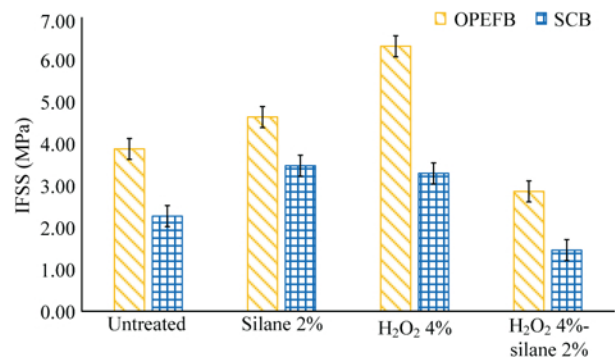


Fig. 9 IFSS test of OPEFB and SCB fibres.

properties of polymeric composites applications. Therefore, treated fibres show better wetted out by hydrophobic matrix compare to untreated. Both SCB fibre treatments with 2% silane and 4% H₂O₂ resulted similar shear strength, however for combined treatment the shear strength become lower than untreated. It might be due to high concentration treatment affected damaging structure of fibre. However, Garcia *et al.*^[59] explained modified sugarcane bagasse fibres by surface treatment important to improve fibre adhesion to polystyrene matrices and composite material^[60]. From the experimental work, expected that the enhancement in the IFSS of the OPEFB and SCB fibres after treatment in these studies may be attributed to the phenomenon called fibrillation where the packed alignments of the fibres are broken into smaller ones through the dissolution of hemicellulose^[61].

4.6 Scanning Electron Microscopy (SEM)

In this study, SEM was used to investigate the surface morphological of OPEFB and SCB fibres before and after treatment. Fig. 10a was showed and highlighted the area of silica bodies found embedded on the surface of the untreated OPEFB fibres. From the image, it's illustrated the silica bodies are attached to circular

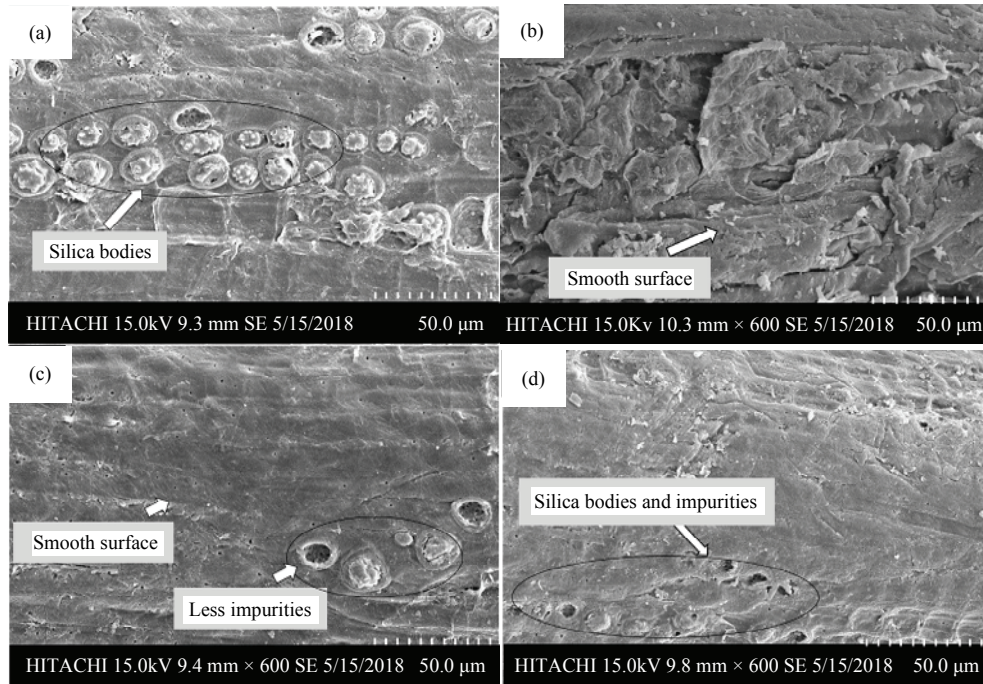


Fig. 10 SEM morphology on (a) untreated and treated with (b) 2% silane, (c) 4% H₂O₂ and (d) 4% H₂O₂-2% silane of OPEFB fibre.

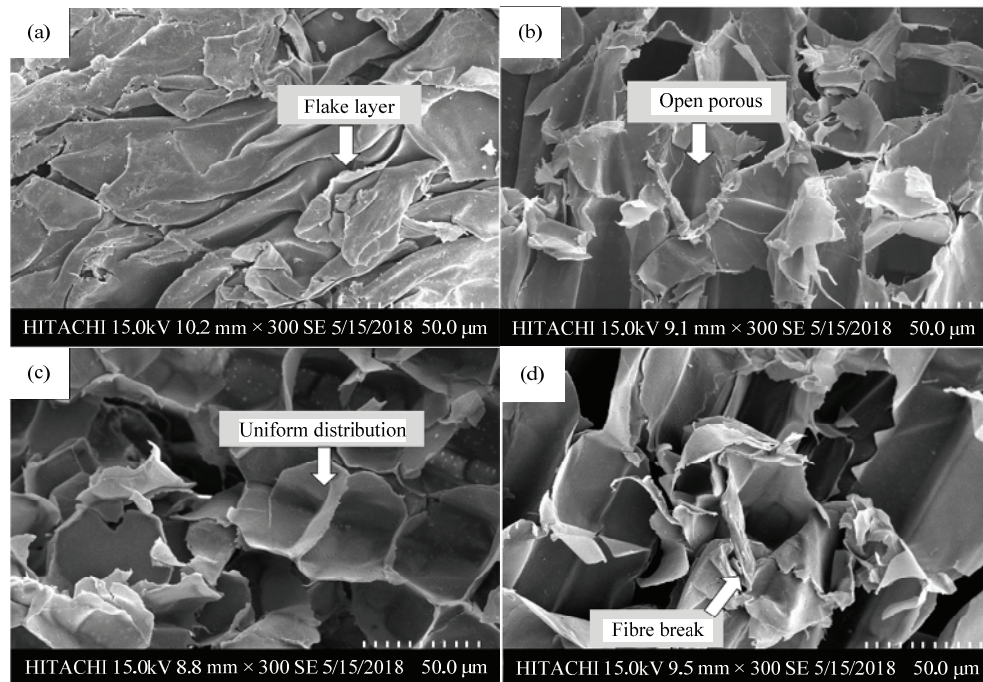


Fig. 11 SEM morphology on (a) untreated and treated with (b) 2% silane, (c) 4% H₂O₂ and (d) 4% H₂O₂-2% silane of SCB fibre.

craters which are spread uniformly over the strand's and impure material on surface layer. This finding is similar with previous research reported that OPEFB fibre contains hemicellulose, cellulose, lignin, silica and metal ions^[62]. Treated OPEFB fibre with silane in Fig. 10b shows the treatment was removed the silica bodies and

inorganic materials on the surface layer. Based on literature, silane treatment on fibre is helpful to removal lignin and hemicellulose from natural fibre and also improving the interfacial bonding of fibre-matrix^[63]. The SEM images of pretreated OPEFB fibre with H₂O₂ in Fig. 10c show that some of the silica bodies were still

attached to the fibre and some had been removed. This result was supported by Yunus *et al.*^[64] that was found low temperature acid pre-treatment caused silica bodies to erode but still remained intact with circular craters. Fig. 10d shows that H₂O₂-silane treatment of OPEFB has no obvious effect in comparison with H₂O₂ treatment.

Fig. 11 illustrated untreated and treated surfaces of SCB fibre. Fig. 11a shows clearly of flake layer surface and impurities droplet on unmodified of SCB fibre. The filaments in the untreated bagasse fibre were pack together in flake form however the morphological changes after pre-treatment. After fibre treatment with 2% silane solution, the detachment of fibres improves and exposes open porous formation on fibril capillary and cell wall surfaces were seen in Fig. 11b. The elimination of superficial layer after treatment was able to increase the contact area and exposed the fibrils^[65].

Fig. 11c shows the microfibril of SCB fibre was exposed to uniform distribution of fibre after 4% H₂O₂ concentrations of pre-treatment. The effect of acid pre-treatment caused exposure of fibre strips where decreased the lignin content in the cell wall and in turn changed the surface texture^[66]. SCB fibre treated with combination H₂O₂-silane in Fig. 11d shows the microfibril in the break structure. This occurred due to high concentration of treatment solution affected to the strength of microstructure around fibre. Previous study found similar finding where once the weakest microfibril is broken, it causes a flaw in the fibre structure and might be act as a microcrack, which swiftly propagates in a brittle mode^[60].

5 Conclusion

In the present work, 2% silane treatment allows the elimination of major parts hemicellulose and lignin of OPEFB and SCB fibres and was reduced fibre dimension. Tensile strength of OPEFB and SCB fibres gives optimum result after treated with 2% silane; 170.96 MPa and 91.91 MPa. Treatment studies were found 2% silane improve the thermal resistance and degradation process of SCB fibre by displayed maximum residual content 26.18% compared to OPEFB fibre with 500 °C temperature. IFSS studies indicated that treatment on OPEFB fibre with 4% H₂O₂ give optimum result

6.32 MPa. This is attributed to the effectiveness of lignin elimination in weak acid treatment and also improvement in fibre-matrix bonding increase in surface roughness of cellulose fibrillation. SEM micrographs analysis of OPEFB fibre showed that the silica bodies in surface layer was removed after treated with 2% silane due to loss of binding material such as lignin and hemicellulose. While 4% H₂O₂ treatment was effective to SCB fibre due to revealed uniform distribution of porous formation. Results obtained in this study are promising and focused on sustainability of biomass composite product for future development as thermal insulation for building wall material. These works found that treated OPEFB fibre sufficient to improve in mechanical properties while treated SCB fibre effective in thermal stability performance.

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