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Original Article

Characterization of silane treated Malaysian Yankee Pineapple AC6 leaf fiber (PALF) towards industrial applications

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ABSTRACT

This research studied the effects of silane treatment at different soaking time: 1, 3, 5 h, on the properties of new variant Yankee's Pineapple AC6 leaf fiber (PALF). The properties of untreated and treated PALF was evaluated through several testing. The Si element was found on all treated fiber's surface through Energy-Dispersive X-ray, while significant peaks were clearly seen for these treated fibers at 1317.81 and 1100 cm^{-1} by Fourier Transform Infrared Spectroscopy. X-Ray Diffractor analyses showed small changes on the crystallinity of all treated fiber disregards the treatment and soaking time as compared to untreated fiber. Improvement on the degradation temperature of all treated fibers to 360 °C from 340 °C was seen from the thermogravimetric analysis. Maximum surface roughness and tensile strength were found for treated fibers at 3 h soaking time by atomic force microscope and single fiber testing respectively. The analyses suggested the potential Yankee's PALF to be used in composites for various industrial applications.

1. Introduction

Natural fiber was believed to have comparable properties to conventional synthetic fiber such as low density, high stiffness and good mechanical properties. The abundant sources of natural fiber is one of the factors leading to its use in var-

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ious applications [1]. It was reported that different locations and variants of plant displays different chemical composition [2–4]. Studies had shown that PALF planted in Indonesia had 70.51% cellulose content whereas PALF in Johor, Malaysia recorded 78.11% cellulose content [5,6]. This cellulose content contributes to the fiber's strength. Element composition test carried out on PALF from India showed the presence of three elements, C (73.13at%), O (24.17at%) and N (2.7at%) [7]. Analysis conducted on banana leaf fiber showed the existence of C, O, N and H at 44.01 wt%, 24.17 wt%, 1.36 wt% and 6.1 wt% respectively [8]. In different studies, the Furcraea foetida (FF) leaf fiber showed the presence of C and O at a weight percentage of 66.43% and 36% respectively.

Regardless of the strength and other benefits of natural fibers, there is a limitation in the compatibility of natural fiber with polymers in composites [9]. Due to this limitation, researchers had explored the surface modification as one of the solution to improve the natural fiber/polymer compatibility. Silane is one of the commercially available inorganic compound for fiber's surface modification [10–13]. Silane reacts with water to form Silanol and Alcohol. The Silanol group will chemisorbed to fiber hydroxyl group forming stable covalent bonds with the cell wall. Hydrophobicity of the fibers increased on Silynylation. The hydrophobic coupling agent forms a protective monolayer on the proton-bearing surfaces, which then removes the sites for moisture absorption [9]. Studies on the effects of alkali and silane treatment on PALF and kenaf fibers had been reported previously. Results showed that PALF has higher tensile strength compared to kenaf fiber, both treated with silane. In different comparison, the silane-treated fibers showed higher tensile strength compared to alkali-treated fibers [14]. Thermal analysis showed that treated PALF had higher decomposition temperature compared to untreated PALF, suggesting the improvement of thermal stability through chemical treatment on fibers [15].

Pineapple, *Ananas comosus (L.) Merr.*, from Bromeliaceae family, is a tropical plant originated from Southeast America, which has been introduced in Tanah Melayu in 1922. The Malaysian Pineapple Industry Board (MPIB) data on the statistic of pineapple industry showed that the world had almost 1,022,319 Ha of pineapple plantation area in 2014, which produce 25,439,366MT of Pineapple in that year alone. Moreover, it showed that Malaysia had been listed in the top 20 country for pineapple plantation area, which produced 335,725MT of pineapple in that particular year and equivalent to 0.01% of total world pineapple production. This data indirectly showed that there is abundance of pineapple raw material waste available to fulfil the demand of composite manufactures, research purposes and development department in Malaysia along with the import-export activities.

There are many varieties of pineapple in Malaysia, which belongs to different categories such as Queen, Hybrid, Cayenne, Yankee etc. Each one with its specific registration code as listed in Table 1. Similar with other natural fibers, it was important to analyse each variant of pineapple as the properties of each variant differs depending on the chemical composition, fiber type and growth condition [3,16].

Due to its abundance sources, the current study aim to fully utilize the Yankee PALF as potential materials in composites, thus producing a useful and sustainable materials. It was reported [3,4] that plastic reinforced flax fiber had been used as door panels in Mercedes due to its lower density, better vibration dumping and blunt fracture compared to synthetic composite.

Table 1 – Malaysia pineapple varieties.

Pineapple varieties	Registration code	Category
Moris	AC 1	Queen
Sarawak	AC 2	Cayenne
Gandul	AC 3	Spanish
Maspine	AC 4	Hybrid
Josapine	AC 5	Hybrid
Yankee	AC 6	Queen
Moris Gajah	AC 7	Queen
N36	AC 8	Hybrid
MD2	AC 9	Hybrid
Madu Kaca	AC 11	Cayenne

Therefore in this study, comprehensive analyses on the Yankee PALF have been done to investigate specifically on its fiber's morphology, mechanical and thermal properties with the aim to substitute current natural and synthetic fiber in textile and composite manufacturing.

2. Materials and methods

2.1. *Materials*

Pineapple leaves from Yankee variant was obtained from Teluk Panglima Garang, Selangor, Malaysia. The leaves were collected after the pineapple fruits were harvested from 1 to 2 years old pineapple plants. The leaves were processed to obtain pineapple leaf fibers (PALF) used throughout this research. Triethoxy(ethyl)silane with molecular weight of 192.23 g/mol brand Sigma-Aldrich was used to treat the PALF after the fiber extraction process.

2.2. *Preparation of PALF*

The collected fresh pineapple leaves were extracted using fiber's extractor machine model PALF M1, which had been fabricated in Malaysia, at Universiti Tun Hussein Onn, Johor, Malaysia [17]. The extracted pineapple leaf fibers (PALF) were rinsed using tap water and sun dried for 2 days to remove the water content in the fiber. The dried PALF were comb to improve the separation of fibers.

Half of the dried PALF was chemically treated using silane treatment. The PALF was immersed in distilled water containing 2% of Triethoxy(ethyl)silane solution with solution to fiber ratio at 30:1 and constant pH value of 4 [7,10]. The fibers were left immersed at three different treatments hours, which are 1, 3 and 5 h. After the respective time, the fibers were rinsed with distilled water to neutralize the pH level and oven dried at 80 °C for 48 h [14].

The untreated and silane-treated PALF were then separately ground into powders for all the characterization analysis conducted in this study except for the tensile single fiber testing and atomic force microscopy (AFM) analysis. These two analyses required long single fibers. Grinder machine (Wiley® Mill, Thomas®, United States) was used to

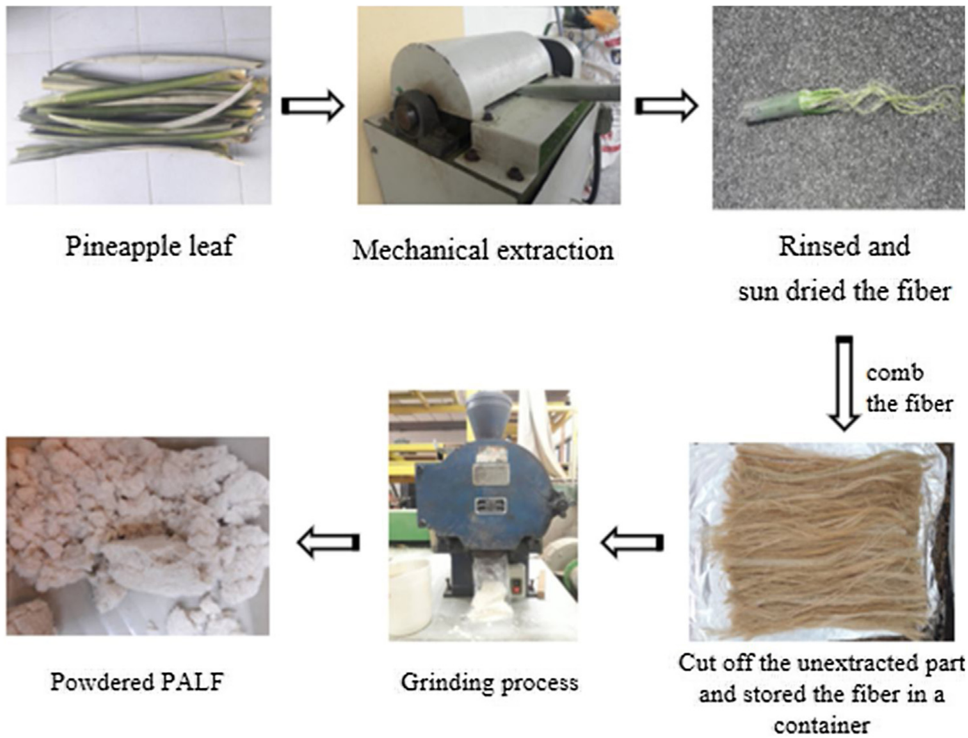

Fig. 1 – Material preparation process.

convert the long PALF into powder. Fig. 1 summarizes the preparation of PALF from the fresh pineapple leaves to the PALF powder.

The chemical composition analysis had been carried out according to a previously reported study [18]. The cellulose, hemi-cellulose and lignin content of the Yankee's PALF are 47.74%, 15.98% and 2.44% respectively.

2.3. *Scanning electron microscopy (SEM)/ energy-dispersive X-ray (EDX)*

The short single PALF was carbon coated using VC-100 Carbon Coater (Vacuum Device, Japan) before the images were scanned using scanning electron microscopy (SEM)/ energy-dispersive X-ray spectroscopy, EDX (JCM-6000, Jeol, Japan) operated at 15 kV accelerating voltage.

2.4. *Fourier transform infrared spectroscopy - attenuated total reflection attenuated total reflection (FTIR-ATR)*

The molecular component and structure of the PALF was analysed using Fourier transform infrared (FTIR) spectroscopy - attenuated total reflection (ATR) (Thermo Fisher Nicolet iS5, United States). Each sample recording consisted of 16 scans recorded from 550 to 4000 cm−1.

2.5. *X-Ray diffraction (XRD)*

The X-Ray diffraction (XRD) was used to determine the crystallographic, structure and physical characteristics of PALF. XRD measurements were performed in a MiniFlex600, Rigaku Co., Japan, at 40 kV and 15 mA at room temperature. CuKα radiation (λ = 0.154 nm) was used as an X-ray source. The operating range was set to be 3-80(2θ) with step size of 0.02. The calculation of the crystal index was done by using peak height ratio (Segal method) as per Eq. (1), where the maximum height peak, I200 at 200 lattice plane and I_{AM} is the height at minimum between two peak which represent the amorphose region.

$$CrystalIndex = \left(\frac{I_{200} - I_{AM}}{I_{200}}\right) \quad (1)$$

The crystal size of the fiber was calculated by:

$$CS200 = \frac{k\,(\lambda)}{\beta_{200} \cos(\theta)} \quad (2)$$

where K is 0.89 the Sherrer's constant, the wavelength, λ of CuKα radiation is 0.154 nm, Beta is the peak's full width at half-maximum in radians and theta is corresponding Bragg angle.

2.6. *Thermogravimetric analysis (TGA)*

The thermal stability of PALF was analysed through Thermogravimetric analysis (TGA) using EXSTAR TG/DTA 6200 (SII Nanotechnology Inc., Japan). The sample was scanned from room temperature to 550 °C with heating rate of 10 °C/min with continuous nitrogen flow at 100 mL/min. Based on literature [11,12], most of the natural fibers possess thermal stability between 300 and 550 °C.

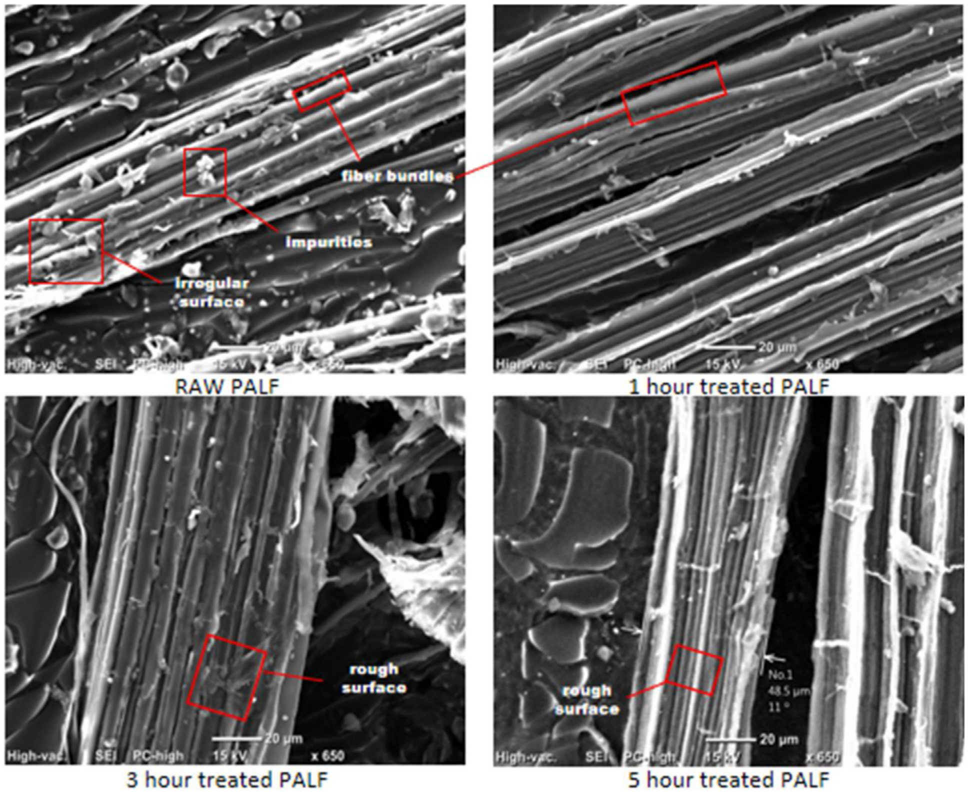

Fig. 2 – SEM image of untreated and treated fiber.

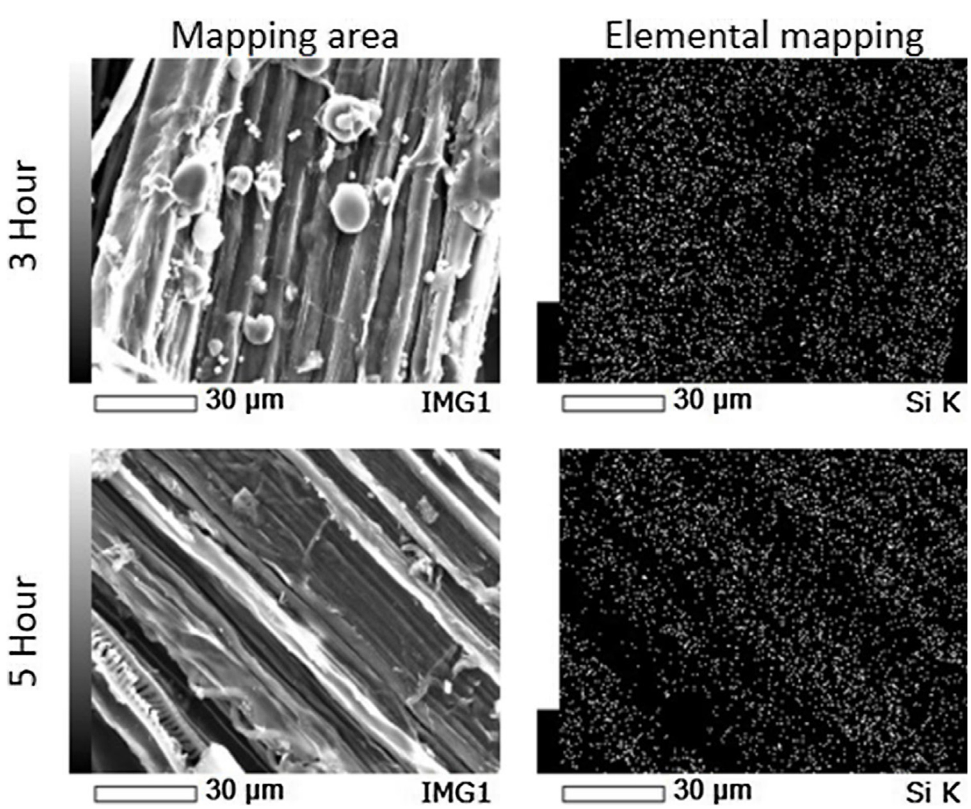

Fig. 3 – Si element mapping through EDX.

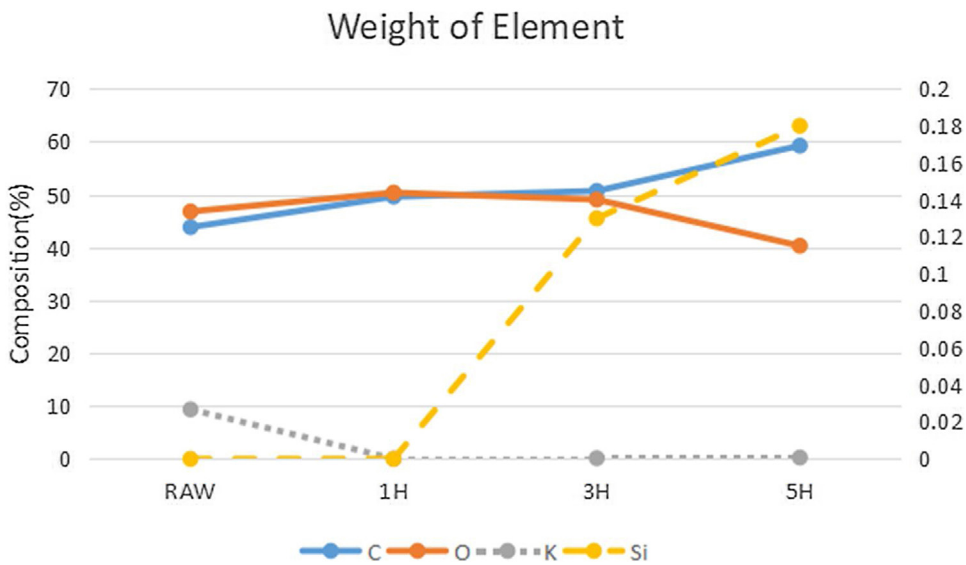

Fig. 4 – Element's weight composition.

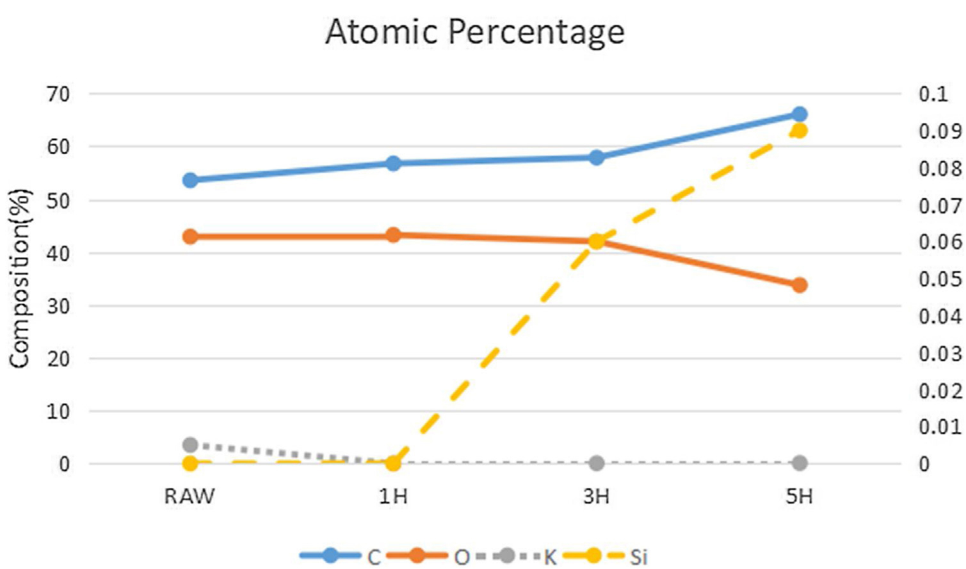

Fig. 5 – Element's atomic composition.

2.7. *Atomic force microscope (AFM)*

Surface three-dimensional nanoscale profile was observed using Atomic Force Microscope (AFM) by measuring forces between a sharp probe and the surface. Commercial silicon AFM tips were used (BrukerNano, United States) with spring constants of 0.4 N/m and resonance frequency of 70 kHz. NanoDrive Dimension Edge software (version 8.06) was used for the image analysis. The measurement was conducted at 24 °C with relative humidity of 40%.

2.8. *Single fiber test*

The tensile single fiber test was conducted according to ASTM D3822 with cross-head speed at 1 mm/min and 5 N max load. The gauge length of fibers varied as 20, 30 and 40 mm. The average result was calculated from 20 sets of sample for each gauge length.

3. Results and discussions

3.1. *Scanning electron microscopy (SEM)/ energy-dispersive X-ray (EDX)*

SEM images of untreated and treated Yankee's PALF were shown in Fig. 2. The untreated PALF bundles were composed of many single fibers bounded to each other. It was observed that the impurities were reduced after the treatment. Fig. 3 shows the presence of the Si element, from silane compounds, in the elemental mapping images. This confirms the successful treatment of silane solution. The amount of Si attachment on the fiber was very low at the beginning of the treatment due

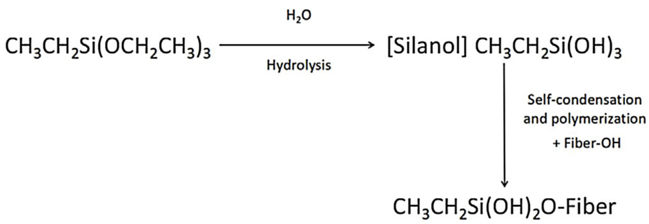

Fig. 6 – Possible Silane reaction with fiber surface.

to the slow condensation process between the silanol group and fiber's surface in an acidic medium [19].

Figs. 4 and 5 show the weight and atomic elemental composition percentage of the fiber captures by EDX. Carbon content increased as the treatment hour increased due to the removal of impurities exposing the fiber surface (cellulose). The oxygen was increased by the 1st hour of treatment due to the removal of impurities on the fiber. However, as the treatment hour increased, the oxygen content decreased, which showed small reaction had taken place between the silane solution and the fiber's surface. Fig. 6 shows the possible reaction taking place between the fiber and silane [7].

3.2. *Fourier transform infrared spectroscopy - attenuated total reflection attenuated total reflection (FTIR-ATR)*

The presence of cellulose, hemicellulose and lignin in the fiber was confirmed from the FTIR (ATR) analysis as shown in Fig. 7. From the FTIR (ATR) The broad band at 3306.05 cm^{-1} revealed the presence of OH group in cellulose [20,21], while the band 2850.20 cm^{-1} the bending of CH_2 hemicellulose component [22]. Yang et al. [23] reported that lignin had C—O—C bond, which the stretching of the functional group was found at 1248.61 cm^{-1}. Besides that, the band 1431.77 cm^{-1} presented methoxyl–O—CH_3, which belongs to one of the lignin's compound [23,24].

The effects of silane treatment can be seen through the changes in bands at 1317.81 and 1100 cm^{-1}. The presence of CH on the treated fiber was represented by the band at 1317.81 cm^{-1}, confirming its link with the fiber's surface. The silane usually have general structure R–Si(OR′)$_3$, in which R is a group that can react with the adhesive of liquid resin and R′ is usually a methyl or ethyl. The general structure of silane hydrolyzed becomes silonal group, which then react with the fiber's surface of OH group. The reaction of silane was illustrated in Fig. 6. The band of CH and Si-O-CH_2 shows self-condensation polymerisation of trisilanol, and its reaction with fiber's surface —OH groups [25–28].

Besides that, the methoxyl–O—CH_3 band of the treated fiber is reduced due to the disruption of some lignin compound during the treatment [23,24]. The reduction of the band was caused by the acidic environment during the treatment process, which affected the lignin's structure [29]. The removal of lignin is one of the objective in doing surface treatment, which increased the hydrophilic property of natural fiber by binding with the cellulose, thus improving its compatibility with the polymer. The crosslinking of polysaccharides of lignin enhanced the water resistant of the plant cell wall.

3.3. *X-ray diffraction*

Fig. 8 showed the crystal index and size for untreated Yankee's PALF with values of 55.22% and 2.17 nm respectively. Previous study reported a lower value of crystallinity index of PALF, which was 13.74% compared to the current study [30]. It was also found that the crystal index of Yankee's PALF fiber was higher compared to jute fiber by approximately 45% [31]. Higher crystal index lead to a higher thermal stability [32].

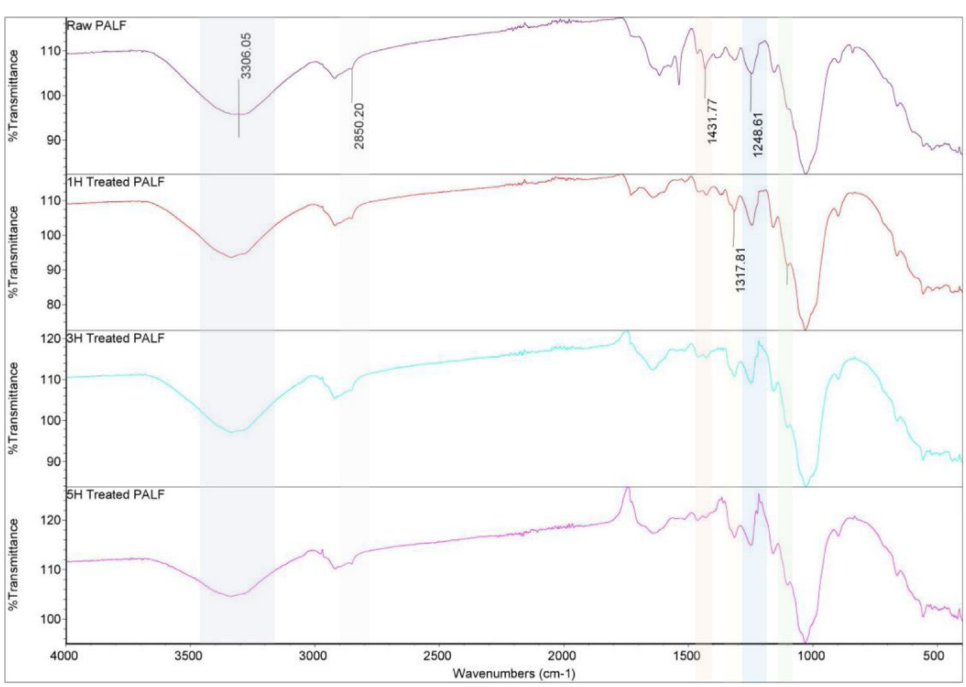

Fig. 7 – The results of FTIR(ATR) on treated and untreated PALF.

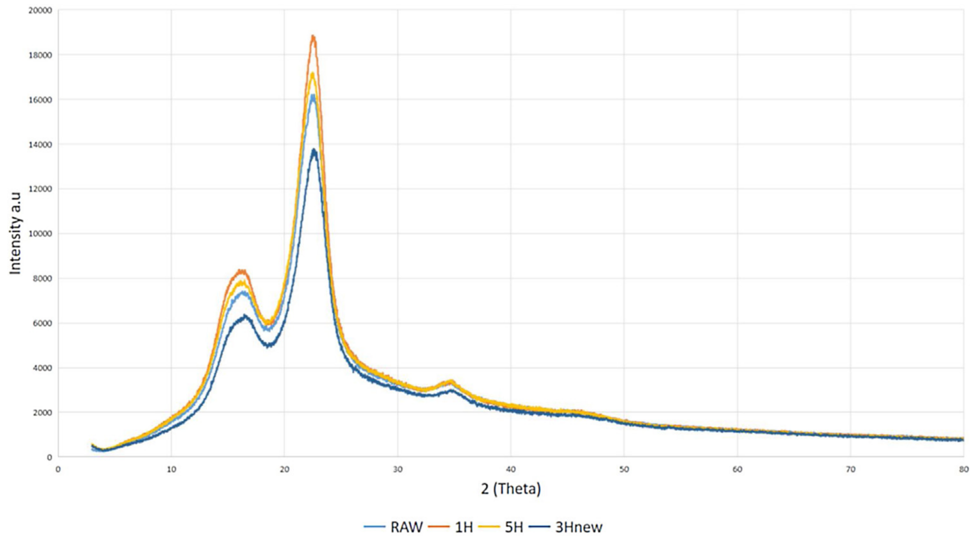

Fig. 8 – XRD pattern of treated and raw PALF.

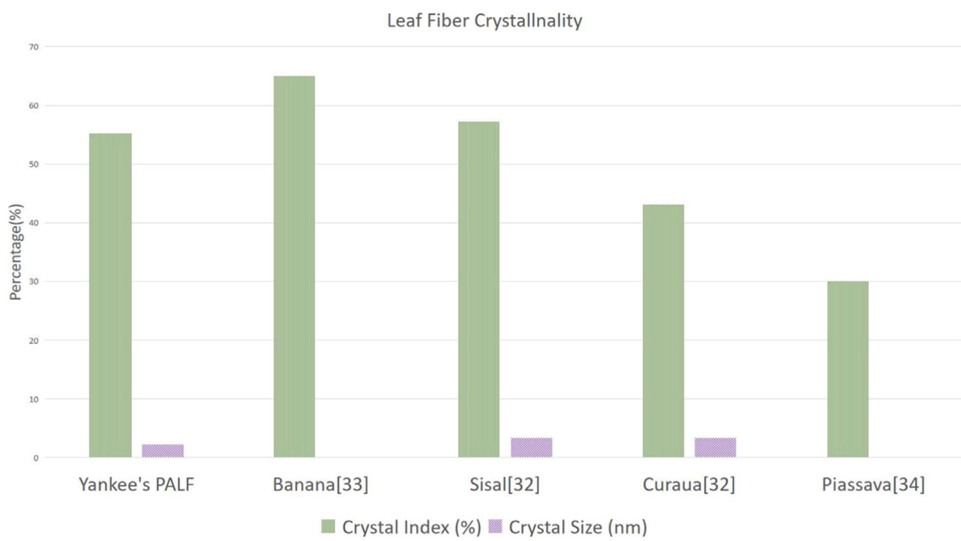

Fig. 9 – Leaf fiber crystallinity.

Table 2 – Crystal size and index of Yankee's PALF.

PALF condition	Crystal size (nm)	Crystal index (%)
Raw	2.17	65.31
1h	2.44	68.61
3h	2.08	63.39
5h	2.19	65.46

On top of that, Yankee's PALF was analyse to have a comparable crystallinity compared to other types of plant leaf's fibers, as shown in Fig. 9 [32–34]. The crystal size of Yankee's PALF was smaller compared to sisal and curaua fiber, which were 3.37 and 3.43 nm, respectively [32]. It was reported that high crystal size arrangement would decreased the chemical reactivity [22]. In comparing the soaking time during the treatment, no significant changes was found on the crystallinity of fiber as shown in Table 2. These results were in parallel with the published results reported previously [35,36].

3.4. *Thermogravimetric analysis (TGA)*

Fig. 10 showed the TGA curves of untreated and treated Yankee's PALF. It was observed that small weight loss occurred between 19-100 °C due to the evaporation of moisture. The next weight loss between 150 and 240 °C was caused by the decomposition of low-molecular weight of lignin component. In Fig. 11, the hemicellulose and lignin degradation peak of treated fiber was decreased at 190–250 °C. These possibilities were due to the removal of some hemicellulose during treatment [37,38]. Hemicellulose is insoluble in water, hence, the acidic condition during the treatment allowed the hydrolyzation to take place [39]. Yankee's PALF had a decomposition temperature approximately at 340 °C, which is similar to that

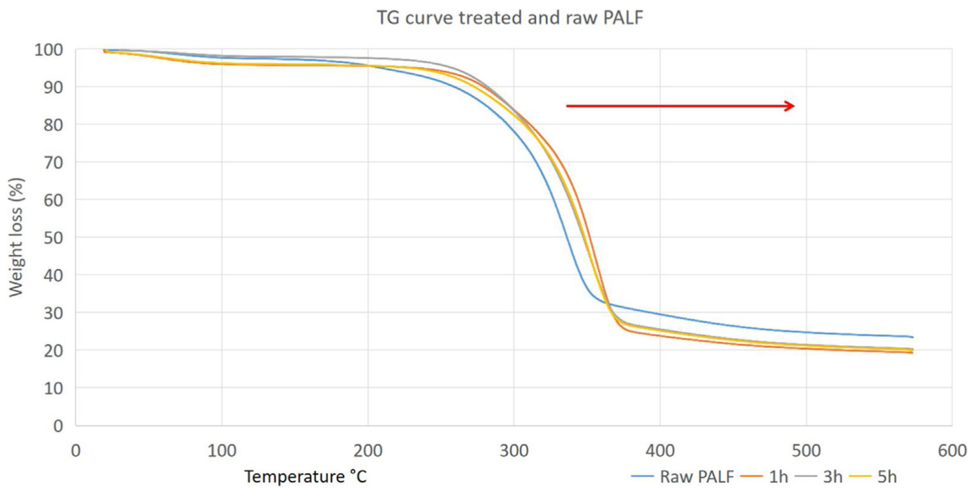

Fig. 10 – TG Curve of Yankee's PALF.

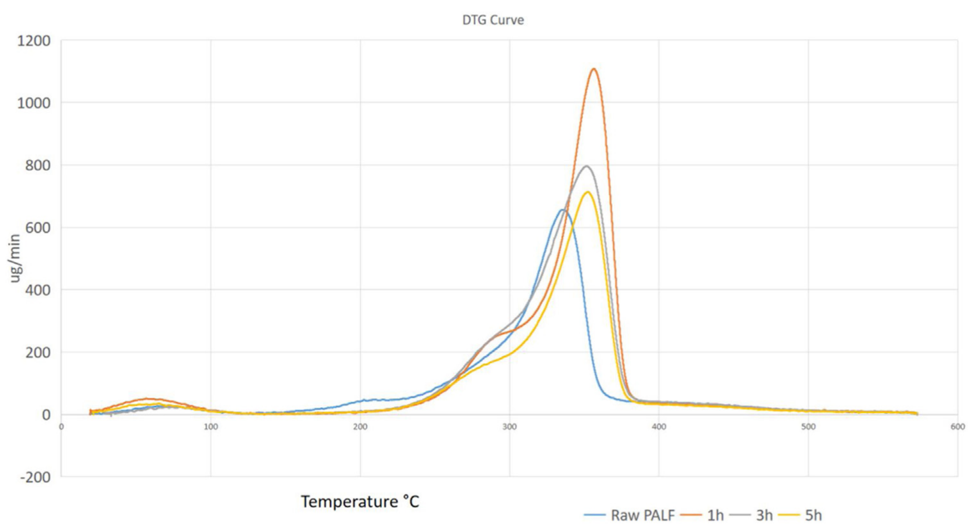

Fig. 11 – DTG Curve of Yankee's PALF.

of Sarawak's PALF but higher compared to Josaphine and Moris's PALF which both decomposed at 320 °C [16]. Overall, the treatment on Yankee's PALF had improved its thermal stability, as shown by the shifted curves to a higher temperature compared to untreated PALF. Different study reported that untreated fiber had lower decomposition temperature compared to fiber treated with 6% NaOH in 1st hour, which were 282.25 and 308.34 °C, respectively [15]. It was also found that the untreated Yankee's PALF had higher yield with value of 34% compared to that of treated PALF of 26%. This proved the removal of some components from the fiber during treatment [7].

3.5. *Atomic force microscope (AFM)*

AFM was conventionally used to investigate the surface morphology and topography of materials. AFM provides a greater level of detail, profiling of three dimensions, as well as the surface roughness [40]. Average roughness (Ra), gives the standard deviation in height, while the root mean square roughness (Rg) represents the standard deviation of surface heights. Skewness (Rsk) is the third moment of profile amplitude probability density function and measures the symmetry of surface data about a mean data profile. Kurtosis (Rku) is the fourth moment of profile amplitude probability function and measures the surface roughness [41].

Table 3 – Yankee's PALF surface roughness.

	RAW	1h	3h	5h
Image Rq (um)	0.00356	0.00781	0.00948	0.00560
Image Ra (um)	0.00286	0.00570	0.00746	0.00449
Skewness	−0.0756	−0.339	−0.362	−0.186
Kurtosis	2.78	4.27	3.57	3.00

Results in Table 3 and Fig. 12 show the surface roughness obtained from image captured at 1.0 × 1.0um. The Ra, Rq, Rku and Rsk were recorded in the table. The Ra and Rq of treated increased as the hour of treatment increased. A high roughness value is favorable for better interlocking between fiber and matrix in polymer composites [42]. The roughness increased at the 1st hour of treatment, which showed the

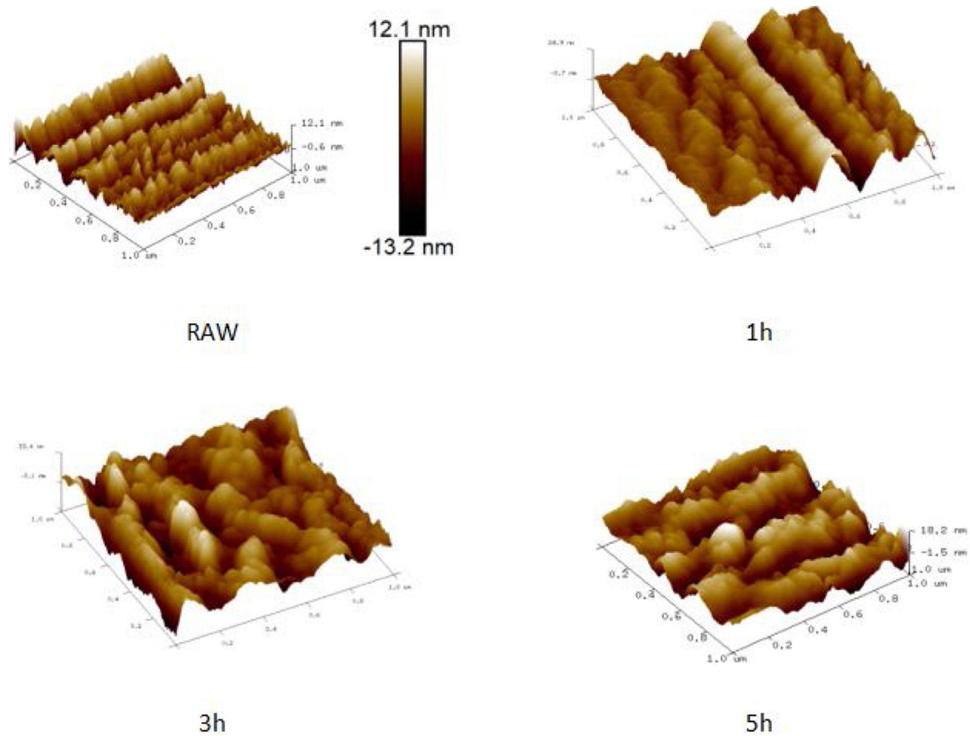

Fig. 12 – 3-Dimensional presentation of the fiber surface texture.

reaction of silane with some lignocellulosic materials of PALF. This observation was supported with the reduction of peak between 190-250 °C in the DTG curve, as shown in Fig. 11, indicating the removal of some lignocellulosic content, which caused the surface to be rougher. A small reduction of roughness was observed at the 5th hour of treatment due to the longer exposure time for fiber in acidic condition, which resulted in smoother fiber's surface.

Data in Table 3 showed negative values of Rsk for untreated and treated PALF respectively. These values indicated that PALF's surface had more peak than valleys, which lead to a good fiber-matrix compatibility in composites. The Rku value larger than 3 for untreated and treated PALF, also reveals that the surface has more peak than valley.

3.6. *Single fiber test*

Tensile strength of PALF single fiber from the current study was compared to other variants of PALF as shown in Fig. 13. It was seen that Yankee's PALF had the highest tensile strength compared to other Malaysian PALF [16].

Fig. 14 showed the fiber with 4 cm gauge length exhibiting the highest tensile strength compared to other gauge length of fibers. This observation was due to a longer fiber having more lignocellulosic contents, which contributed to the fiber strength. The treated fiber showed an improvement on its mechanical strength as the treatment hour increased due to removal of some impurities on the fiber surface. This result was supported by the SEM and DTG analyses discussed previously in Section 3.1 and 3.4. However, the strength of fiber reduced at the 5th hour of treatment due to the reduced amount of lignin in the fiber as shown in the DTG analysis. Lignin has strong intermolecular bond contributing to the strength of fiber. However, the main purpose of the surface treatment is to enhance the matrix-fiber compatibility. The surface treatment caused the fiber's surface to be rougher compared to the untreated as shown in Fig. 12. The rougher fiber's surface is one important indicator to enhance the matrix compatibility with fiber. The composites reinforced with surface-treated fiber showed higher strength compared to untreated fiber [9].

4. Conclusions

The purpose of the current study was to investigate the effects of surface modification on Yankee's PALF using silane solution. The presence of Si element for treated fiber proved the successful of reaction with the fiber. The surface modification did not affected entirely the crystallinity of the fiber. However, the thermal stability of treated fiber improved by 5.9% and the lower yield by 29%. The surface roughness (Ra,Rq) of treated fiber showed an increment. However, at the 5th hour of treatment, it was reduced. Single fiber test showed that after 3 h of treatment it was produced the highest tensile strength compared to other configurations. It can be concluded that different treatment hours have different influences on the fiber's surface. Optimization is required to select appropriate treatment parameters according to the application and desired properties [43]. Overall, the 3rd hour of treatment time proved

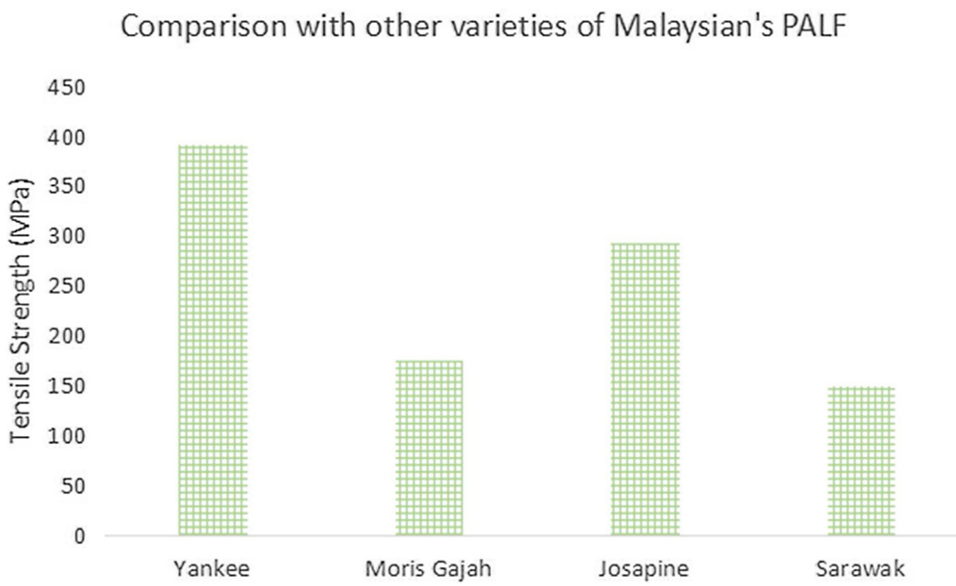

Fig. 13 – Tensile strength of Malaysian PALF varieties.

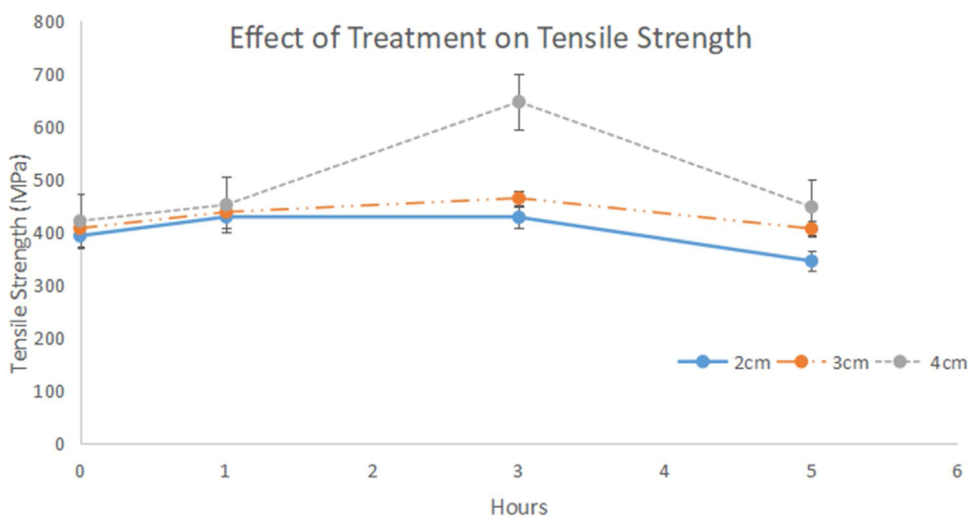

Fig. 14 – Tensile strength of Yankee's PALF.

to be the best condition for surface modification based on the experiment set-up for Yankee's PALF to be used in industrial applications such as in composite and textile industries.

Conflicts of interest

The authors declare no conflicts of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.jmrt.2020.01.058.

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