STUDY ON CHANGES IN MORPHOLOGY AND PHYSICOHEMICAL PROPERTIES OF ARECA NUT FIBRE BY DIFFERENT SURFACE TREATMENT METHODS

Thwe Thwe Soe[1,2], Htin Zaw Myint[3], Saw Hla Myint[4]

Abstract

Areca nut husk is abundantly available in Mone, Kyauk-kyi Township, Bago Region as byproduct from the areca nut farm, and finding a way to convert it into a value added material to produce a useful material could be of national interest. The fibre was extracted from the areca nut husk, washed and treated with 5 % NaOH, some of the product was further treated separately with permanganate and benzoyl chloride to modify the fibre surface. As a result, considerable change in surface morphology of fibres was observed by SEM using an Evol 18 Zeiss scanning electron microscope (SEM). Lignin and hemicellulose contents were reduced by the alkali treatment as shown by the FT IR spectra recorded on a Tracer 100 Shimadzu spectrophometer. The thermal characteristics of the untreated and treated natural fibre was studied by TG TDA. The surface tension of the untreated and treated fibre (in the order given above) were found to be, 24.86, 25.86, 26.95 and 26.10 mN m^{-1}. Thus by the surface treatment, the fibre surface tension, was found to increase, which is favorable for a better fibre – matrix binding in a composite. Fibre diameter (by micrometer), fibre length (by vernier calliper) and aspect ratio of the untreated and treated fibre were found to be, respectively, 0.36, 0.38, 0.35 and 0.38 mm; 53.34, 56.61, 52.19 and 54.86 mm; 148.16, 148.97, 149.11 and 144.37. Water absorption property by soaking-squeezing method showed the water absorption % of the untreated and treated fibres at 6, 12 and 24 h, respectively, to be 277.64 , 292.17, 219.39 and 288.05 %; 268.45, 290.70 , 205.70 and 251.50 %; 250.95, 254.19 , 204.66 and 222.23 %, showing increase in water absorption of the fibre after alkaline treatment, but significant decrease by the permanganate treatment, which is good for resistance of the composite to moisture. The present study suggests improvement of fibre morphology and composition for the preparation of a useful composite material.

Keywords: Areca nut fibre, surface modification, alkali treatment, permanganate treatment, benzoyl chloride treatment, cellulose, lignin

Introduction

Nowadays natural fibres have become superior alternatives to synthetic fibres for polymeric composites due to their advantages *i.e*. cheap, lightweight, renewable, biodegradable, flexible in usage and naturally recyclable. Moreover the application of natural fibre reinforced polymeric composites are found in house construction materials, aerospace, panels, and automobile parts.

Many attempts were made by scientists to utilize the natural fibres in the fabrication of composites. Their efforts to introduce the natural fibre composites are because of the following reasons;

1. These fibres, despite their low strength can lead to composite with specific strengths because of their low density;
2. Dried natural fibres are nontoxic and eco-friendly and biodegradable and are cheap;
3. Natural fibres are abundantly available renewable resources.

[1] Assistant Lecturer, Department of Chemistry, Yangon University of Education
[2] PhD Student, Department of Chemistry, University of Yangon
[3] Dr, Associate Professor, Department of Chemistry, Taungoo University
[4] Dr, Part-Time Professor, Department of Chemistry, University of Yangon

With growing environmental awareness, new rules and legislations, scientists and engineers are forced to seek new materials which are more eco-friendly in nature.

Hence, the attention of the research community is focused toward finding an eco-friendly material which can give high performance at affordable costs.

Natural Fibre Surface Modification

Natural fibres are amenable to modification as they bear hydroxyl groups from cellulose and lignin. The hydroxyl groups may be involved in the hydrogen bonding within the cellulose molecules thereby reducing the activity towards the matrix. Chemical modifications may activate these groups or can introduce new moieties that can effectively interlock with the matrix. Interfaces play an important role in the physical and mechanical properties of composite. Simple chemical treatments can be applied to the fibres with the aim of changing surface tension and polarity of fibre surface.

Natural fibres have a good potential for chemical treatment due to presence of hydroxyl groups in lignin and cellulose. Reaction of hydroxyl groups can change the surface energy and the polarity of the natural fibres. Many studies have been undertaken to modify the performance of natural fibres.

The different surface chemical modifications of natural fibres have achieved various levels of success in improving fibre strength, fibre fitness and fibre-matrix adhesion in natural fibre composites.

Different surface treatment methods such as alkali treatment, isocyanate treatment , acrylation, benzoylation, latex coating, permanganate treatment, acetylation, silane and peroxide treatment (Baiardo *et al*., 2002) have been applied on the fibre to improve its strength, size and its shape and the fibre-matrix adhesion. Generally, mechanism of the performance of these methods is different and is depended on the chemical structure of the reagent.

Consequently, the aim of the present work is surface modification of areca nut fibres, which is abundantly available as waste material, by different chemical treatments with alkali treatment, permanganate and benzoyl chloride, and to study the changes in morphology, chemical constitution and physiochemical properties, with the final aim to prepare useful composite materials with natural rubber.

Botanical Description of *Areca catechu* L.

Botanical name : *Areca catechu* L.
Family name : Arecaceae
Order : Arecales
Genus : *Areca*
Species : *catechu*
Common name : Areca nut, Betel nut
Myanmar name : Kun-thee
Part used : Fruits

Materials And Methods

This research consists of two main parts. The first part deals with the raw material collection, isolation of areca nut fibre and its surface modification. The second part is the determination of morphology, chemical constitution and physiochemical properties, the areca nut fibre.

Preparation of Areca Nut Fibre

Sample collection

The areca nut husks were collected from the local area, Mone, Kyauk-kyi Township, Bago Region (Figure 1).

Extraction of areca nut fibre

The areca nut husks were taken directly from the areca nut fields containing a lot of dirt and dust. The dirt, dust, individual fibre and coarse fibre were removed by washing with distilled water. The selected areca fruit husks were soaked in deionized water for about five days. This process is called retting; allowing the fibre to be removed from the husk easily. Then areca nut fibre was removed from the husk and separated into different grades of purity. The selected grade of fibre was dried under direct sunlight (temperature 30 °C) for five days before the alkali treatment. The dried fibre was designated as untreated fibre.

Figure 1 Areca nut husk

Alkali treatment or mercerization

The clean and dried areca nut fibre was soaked in a stainless steel vessel containing 5 % NaOH solution. The alkali treated fibre was immersed in the distilled water for 24 hours in order to remove the residual NaOH. Final washing was done with distilled water containing a small amount of acetic acid for neutralization. Subsequently, the fibre was dewatered and dried under sun light for five days (Figure 2).

Potassium permanganate treatment

Some of the fiber, pre-treated with 5 % alkali were immersed in a 0.5 % $KMnO_4$ solution in acetone for 30 min. The permanganate treated fibers were then decanted and dried in air.

Benzoyl chloride treatment

Some of the fiber, pre-treated with 5 % alkali was immersed in acetone solution and added sodium chloride, same ratio of benzoyl chloride with acetone (0.1: 0.1), and then agitated with sodium bicarbonate solution for 15 min. This solution was filtered. Then the treated areca fiber was

soaked in ethanol solution for 1 h to remove benzoyl chloride that adhered to the fiber surface, washed thoroughly using distilled water and dried in air.

Comparative Study on the Surface Morphology of the Untreated and Treated Areca Nut Fibres

The morphological changes by the fibre surface modification was studied by Evol 18 Zeiss scanning electron microscope (SEM) at West Yangon University (Figures 3 (a), (b), (c) and (d)).

Comparative Study on the Infrared Absorption Spectra of the Untreated and Treated Areca Nut Fibres

The changes of chemical constitution of the fibre by surface chemical treatment was studied by infrared absorption spectra recorded on a Tracer 100 Shimadza, Japan spectrometer at West Yangon University (Figures 4 (a), (b), (c) and (d)).

Comparative Study on the Thermal Properties of the Untreated and Treated Areca Nut Fibres

The effect of the untreated and treated fibre thermal properties was studied using a TG DTA instrument (Hi-TGA 2950 model). The temperature range between 0 ºC and 600 ºC under nitrogen (at a rate of 50 mL/min) at Universities' Research Center, Yangon University (Figures 5 (a), (b), (c), (d) and Table 2).

Comparative Study on the Surface Tension of the Untreated and Chemically Treated Areca Nut Fibres

Approximately 20 mL each of ten aqueous solutions of methanol (various mole ratios from 1.000, 0.910, 0.840, 0.775, 0.675, 0.600, 0.530, 0.480, 0.435 and 0.395 corresponding to surface tension at 25 ºC respectively, 22.33, 23.00, 23.50, 24.00, 25.00, 26.00, 27.00, 28.00, 29.00 and 30.00 (Ghahremani et al., 2011)) was poured into a clean

50 mL beaker and the temperature adjusted at 25 ºC. Twenty pieces of each type of fibre were carefully placed on top of the liquid surface in each beaker and within for 20 s, and the number of fibres that remained floating on the liquid was counted and the percent floating fibres calculated. The surface tension of the liquid in which 50 % of the fibres floated was taken as approximate surface tension of the fibres tested (Hazendonk *et al.*, 1993) at Taungoo University (Table 3).

Determination of Water Absorption Property of Fibre

Water absorption was determined after immersion of the samples in water at room temperature for 6, 12 and 24 h (Table 5). Each sample (3 g) was weighed before and after immersion of fibre. Water absorption was determined by using the following equation.

$$\text{Water absorption}(\%) = \frac{m_2 - m_1}{m_1} \times 100$$

Where, m_1 = mass of fibre before immersion

m_2 = mass of fibre after immersion

Results and Discussion

Extraction of Areca Nut Fibre

After soaking and drying, 1.4 viss of fibre was extracted from 5 viss of raw areca husk. This is the mixture of coarse and fine fibres (Figure 2).

Figure 2 Extracted areca nut fibre

Surface Modification of the Areca Nut Fibre

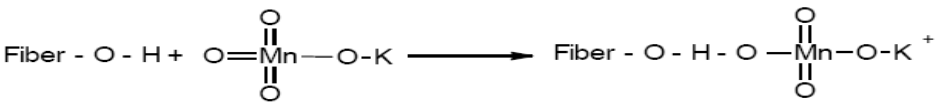
Alkali treatment

$$\text{Fiber-OH} + \text{NaOH} \rightarrow \text{Fiber-ONa} + H_2O$$

Alkali treatment of areca fibers result in the formation of Fiber–cell–O–Na groups between the cellulose molecular chains. Alkali treatment leads to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose and there is a change in the surface topography of the areca fibers (Dhanalakshmi *et al*., 2015). During alkali treatment the removal of hydrogen bonding takes place in the fibre network structure. Due to this, hydrophilic hydroxyl groups are reduced and the fibres moisture resistance property is increased. It also takes out certain portion of hemicelluloses, lignin, pectin, wax and oil covering materials. As a result, the fiber surface becomes cleaner. In addition to this it increases the fibre aspect ratio (Table 4). This could result in better fiber-matrix interfacial adhesion. Mechanical and thermal behaviors of the composites are improved significantly by this treatment. Alkali treated fibre have lower lignin content than untreated fibres.

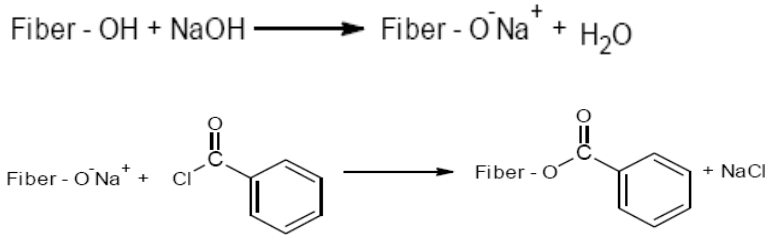
In the present work, the extracted areca nut fibre was treated with 5% NaOH solution. The resulting fibre have yellowish brown colour. The untreated fibre was paler than alkali treated one.

Potassium permanganate treatment



Highly reactive permanganate ions react with cellulose hydroxyl groups and form cellulose manganate. Permanganate ions also react with the lignin constituents and carve the areca fiber surface. As a result, areca fiber surface becomes darker, physically rough, bristly and this reduces hydrophilic nature of the areca fibers (Table 5). This could improve chemical interlocking at the interface and provides better adhesion with the polymeric matrix

Benzoyl chloride treatment



When benzoyl chloride reacts with areca fibers, an ester linkage is formed. This benzoyl chloride treatment results in the reduction of hydrophilicity of areca fibers and hence made the areca fibers to become more compatible with polymer matrix. Benzoylation treatment also enhances thermal stability of the fibers. These could improve chemical interlocking at the interface and provides effective fiber surface area for good adhesion with the matrix (Dhanalakshmi *et al*., 2015).

Morphological Study of the Areca Nut Fibres

Different chemical treatments for the fibre surface modification have effects on the fibre surface morphology (Figures 3 (a), (b), (c) and (d)).

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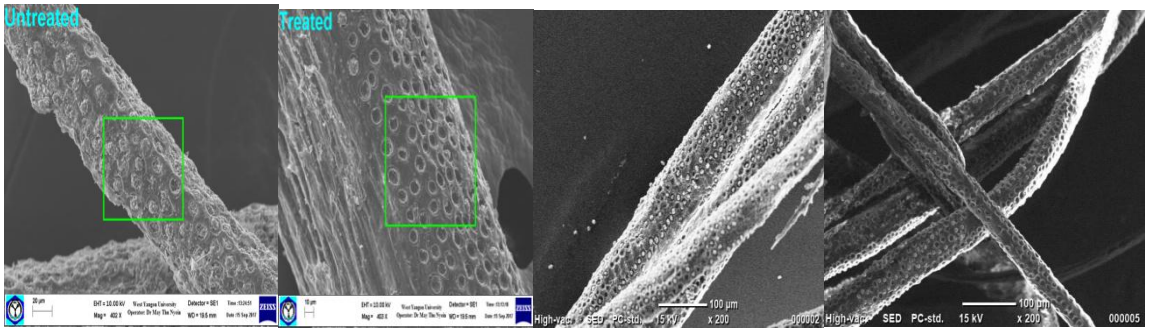

(a) **(b)** **(c)** **(d)**

Figure 3 SEM micrographs of the areca nut fibre (a) untreated (b) alkali treated (c) permanganate treated and (d) benzoyl chloride treated

The untreated fibre presents a network structure in which the fibrils are bound together by hemicelluloses and lignin. Longitudinally oriented unit cells with almost parallel orientations are present (Dhanalakshmi *et al*., 2015).

The alkali treated fibre clearly shows large number of pinholes or pits on the surface, which are due to the removal of fatty deposits from the fibre. Alkali treatment removes waxy epidermal tissue, adhesive pectin and hemicelluloses. Topographical changes because of the removal of low molecular weight compounds result in the formation of a rough surface.

The permanganate treatment (Binoj *et al*., 2016), highly reactive permanganate ions react with the cellulose hydroxyl groups and forms cellulose–manganate. This ion reacts with the lignin and carve the fiber surface and thus reduces moisture absorption. Areca fiber surface becomes physically rougher on permanganate treatment which can be evidenced from the SEM micrograph (Figure 3 (c)) and enhances effective fiber surface area for good adhesion with the matrix.

The SEM image of benzoyl chloride treated areca nut fibre clearly shows a large number of pinholes and rough surface (Figure 3 (d)). It forms an ester linkage to the areca fibers and thus

reducing hydrophilicity making it more compatible with polymer matrix. Hence, this benzoyl chloride treatment provides effective fiber surface area for good adhesion with the matrix.

Chemical Composition of the Untreated and Treated Areca Nut Fibre

In the FT IR spectra, the peak at 1737 cm^{-1} is referred to ester and ether crosslinks between cellulose and lignin or cellulose and hemicellulose (Table 1). The peaks observed between 1100-1600 cm^{-1} shows the presence of hemicellulose in the fibre (Dhanalakshmi *et al*., 2015).

Table 1 FT IR Spectral Assignment of Areca Nut Fibre

Wave number (cm^{-1})	Band Assignments
1000- 1300	Alcohols, Ether, C-O stretching vibrations
1100- 1600	Hemicellulose (ester, benzene ring, alcohol, ether)
1377	Alcohol group of cellulose
1400- 1600	Aromatic rings (C=C stretching)
1737	Ester and ether crosslinks between cellulose and lignin or cellulose and hemicellulose

(Dhanalakshmi *et al*., 2015)

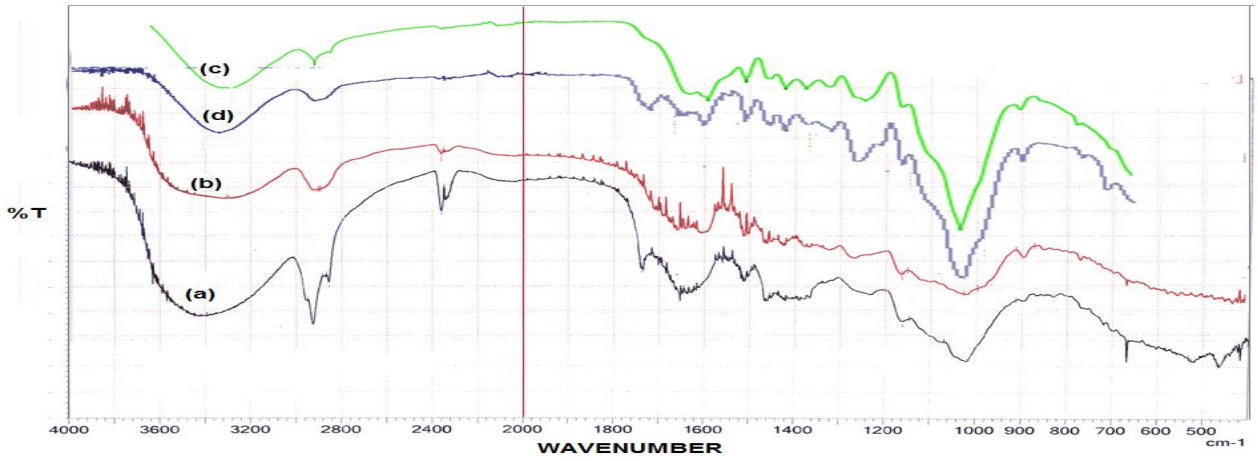

Figure 4 FT IR spectra of areca nut fibre (a) untreated (b) alkali treated (c) permanganate treated and (d) benzoyl chloride treated

After the alkali treatments, hydrolysis occurs which breaks down the ester bond or ether bond, resulting in the absence of 1737 cm^{-1} peak in alkali treated areca fibres (Figure 4 (b)). The peaks observed between 1100-1600 cm^{-1} shows the presence of hemicelluloses in untreated areca fibre and the reduced intensity of these peaks in alkali treated areca fiber indicates the slight removal of hemicelluloses from the fibre surface.

After the potassium permanganate treatments, hydrolysis occurs which breaks down the ester bond or ether bond, resulting in the further reduction of the band at 1737 cm^{-1} in potassium permanganate treated areca nut fibre (Figure 4 (c)). Further reduction of band intensity between 1100-1600 cm^{-1} shows further removal of hemicelluloses from the fiber surface in a permanganate treated areca.

The IR spectrum of benzoyl chloride treated areca nut fiber indicates a carbonyl stretching band at 1718 cm^{-1} and the C=C stretching bands of aromatic ring between 1400-1600 cm^{-1}, indicating benzoylation at the fibre surface. Further reduction of band intensity between 1100

-1600 cm^{-1} showing further removal of hemicelluloses from the fiber surface is also observed (Figure 4 (d)) (Silverstein *et al*., 2003).

Thermal Properties of the Untreated and Treated Areca Nut Fibres

In general, natural fibers have different chemical compositions of cellulose, hemicelluloses, lignin and pectin and so their flammability varies from fiber to fiber. Higher cellulose content results in higher flammability while higher lignin content results in greater char formation with lower degradation temperature (Dhanalakshmi *et al*., 2015). Hence, the cellulose and lignin content present in these natural fibers decides the thermal stability of natural fibers reinforced polymer composites. So, it is very important to promote surface modification of natural fibers with various chemical treatments to decrease hydrophilicity (Table 5) and to improve the thermal stability of natural fibers (Table 2).

Table 2 Thermal Decomposition of the Untreated and Treated Areca Nut Fibres

Fibre	Temperature (°C)	Temperature during mass loss (°C)	Weight loss (%)	Nature of peak	TG/DTA Remark
Untrated Fibre	67.04	38.56-100	6.70	Endothermic	Loss of moisture and highly volatile extractives
	349.06	100-350	65.08	Exothermic	Decomposition of cellulose and hemicelluloses
	466.58	350-470	89.62	Exothermic	Degradation of the lignin
Alkali Treated fibre	80.50	38.94-100	4.82	Endothermic	Loss of moisture and highly volatile extractives
	351.92	100-360	64.90	Exothermic	Decomposition of cellulose and hemicelluloses
	450.62	360-600	87.98	Exothermic	Degradation of the lignin
$KMnO_4$ treated fibre	57.52	38.70-100	6.08	Endothermic	Loss of moisture and highly volatile extractives
	360.05	100-370	43.76	Endothermic	Decomposition of cellulose and hemicelluloses
	423.85	370-600	76.05	Exothermic	Degradation of the lignin
C_6H_5COCl treated fibre	65.04	38.59-100	6.95	Endothermic	Loss of moisture and highly volatile extractives
	381.98	100-390	73.5	Endothermic	Decomposition of cellulose and hemicelluloses
	428.60	390-600	87.56	Exothermic	Degradation of the lignin

The TG DTA curves show the three main degradation phases (Figure 5). The initial degradation phase was due the loss of moisture and highly volatile extractives. In this degradation phase, the volatile hydrocarbons were released from the natural fibre as a result of thermal decomposition of cellulose and hemicellulose.

The second phase of degradation is due to the decomposition of cellulose and hemicellulose.

The third degradation points in the TG DTA curves represent heavy fractions mainly from lignin degradation.

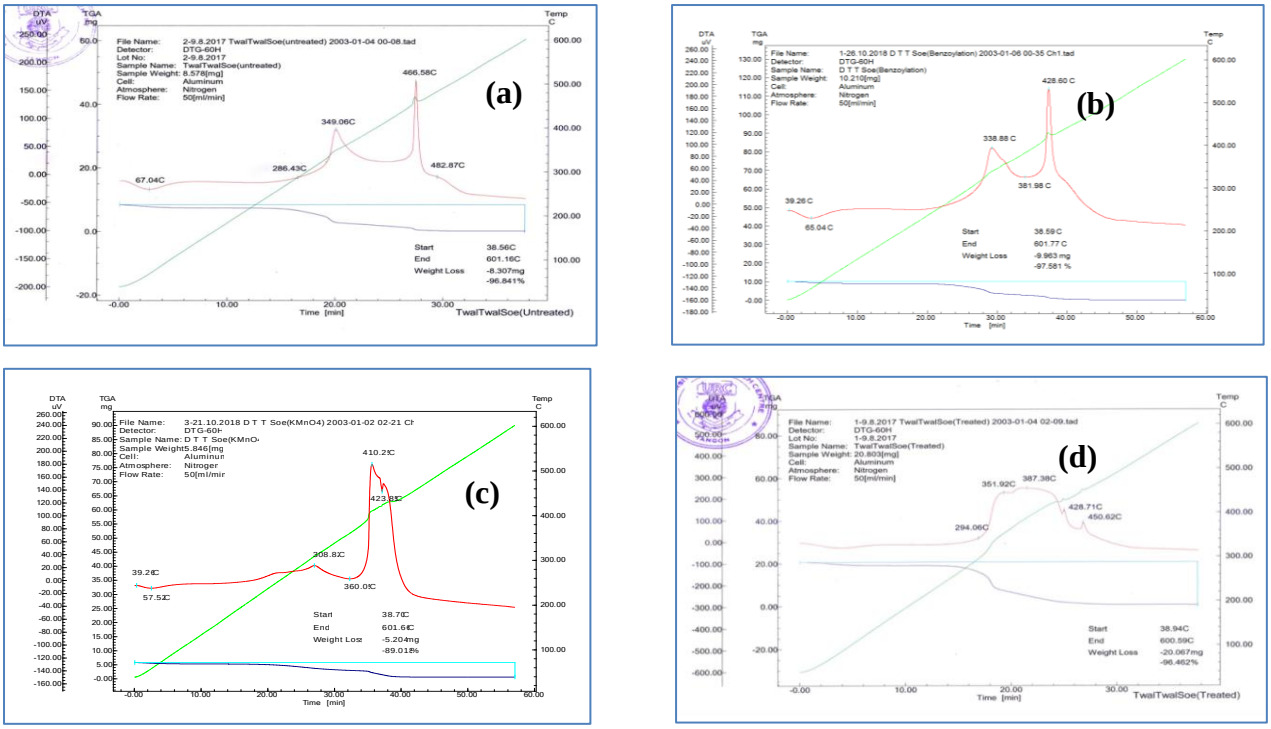

Figure 5 TG TDA thermograms of (a) untreated (b) alkali treated (c) permanganate treated and (d) benzoyl chloride treated areca nut fibres

The TG DTA results of untreated and treated areca fibres from (Table 2) revealed that the maximum decomposition for the untreated, alkali treated, permanganate treated and benzoyl chloride treated fibres were 349.06, 351.92, 360.05 and 381.98 ºC, respectively. According to the data, the main second peak decomposition temperature of benzoyl chloride treated, permanganate treated and alkali treated areca fibers in DTG curve were higher than that of untreated areca fibers. These results confirmed the improved thermal stability for treated areca nut fibers.

Surface Tension of the Untreated and Treated Areca Nut Fibre

The determination of the Surface tension at which all fibres just float proved to be difficult, because fibre heterogeneity there is a range of γ_L values in which some fibres float and other sink. So, γ_F was chosen as the surface tension where 50 % of the fibres float on the liquid surface (Ghahremani *et al.*, 2011). This surface tension was determined graphically (Figure 6). According to the data, the standard deviation is below 1 mN m^{-1}. It can be concluded that permanganate treated fibre were more hydrophobic than other untreated and treated fibres (Table 3).

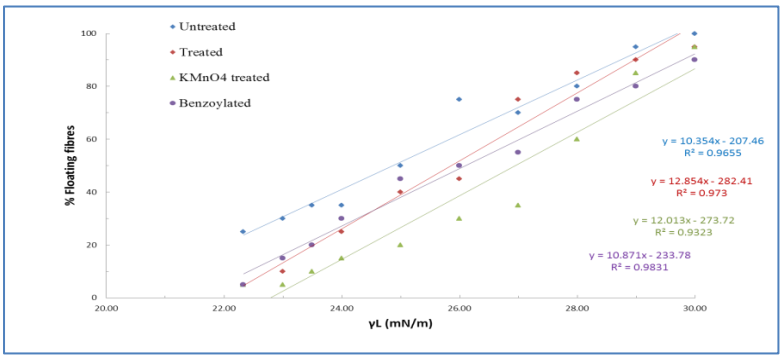

Figure 6 Plot of percent of floating fibres as a function of surface tension of water-methanol mixtures

Table 3 Surface Tensions of Water-methanol Mixtures Giving Zero Contact Angles (γ_F) for Different Fibres

Surface Tension (mN m^{-1})			
Untreated fibre	**Alkali treated fibre**	**Permanganate treated fibre**	**Benzoyl chloride treated fibre**
24.86	25.86	26.95	26.10

Comparative Study on the Physicochemical Properties of the Untreated and Treated Areca Nut Fibres

The size of lumen in natural fibre is proportional to the diameter of the untreated and treated fibre, where the lumen size increases with the increase in fibre diameter. Alkali treated fibre were observed with bigger lumen size, whereas the untreated fibre exhibits a slightly smaller and elongated lumen. By relating the lumen size of areca nut fibre in (Figure 3) with the results of areca nut fibre length shown (Table 5). Moreover, the good aspect ratio and lightweight characteristic of (permanganate treated fibre, alkali treated fibre and untreated fibre) well-suits the fabrication of lightweight composite than other treated fibre.

Table 5 Dimensional Characteristics of the Untreated and Treated Areca Nut Fibres

Measurement	Untreated Fibre	Alkali treated Fibre	Permanganate treated Fibre	Benzoyl chloride treated Fibre
Fibre diameter (mm)	0.36	0.38	0.35	0.38
Fibre length (mm)	53.34	56.61	52. 19	54.86
Aspect ratio	148.61	148.97	149. 11	144.37

Water Absorption Property of the Untreated and Treated Fibre

Alkaline treatment fibre more water absorption properties than other treated fibre, but significant decrease by the permanganate treatment, which is good for resistance of the composite to moisture (Table 6).

Table 6 Variation of Water Absorption with Soaking Time for Untreated and Treated Areca Nut Fibres

	Water absorption (%)			
Time (h)	**Untreated Fibre**	**Alkali treated Fibre**	**Permanganate treated Fibre**	**Benzoyl chloride treated Fibre**
6	277.64	292.17	219.39	288.05
12	268.45	290.70	205.70	251.50
24	250.95	254.19	204.66	222.23

Conclusion

The unmanaged disposal areca husk was collected from the local area, Mone, Kyauk-kyi Township, Bago Region. The collected husk were cleaned and soaked in distilled water for 5 days to extract the fibre. The extracted fibre was treated with 5 % of sodium hydroxide solution, and the alkali treated fibre was surface modified by 0.5 % potassium permanganate solution and benzoyl chloride solution. From SEM study, the surface morphology of the areca nut fibre was observed to change favorably for the preparation of composite materials with a matrix material by the treatment with 5 % NaOH solution, 0.5 % potassium permanganate solution and benzoyl chloride. Alkali treatment removes waxy epidermal tissue, adhesive pectin and hemicelluloses. Topographical changes because of the removal of low molecular weight compounds result in the formation of a rough surface. FT IR spectra showed that the alkali treatment, potassium permanganate treatment and benzoyl chloride treatment were also found to reduce unwanted lignins and hemicelluloses. The thermal stability of benzoyl chloride, potassium permanganate and alkali treated areca nut fibres were higher than the untreated fibre as characterized by

TG TDA method. The results from TG TDA data have also showed that the concentration of cellulose is higher in benzoyl chloride treated fibre than in potassium permanganate treated fibre. The surface treated fibres were found to possess higher fibre surface tensions than the untreated fibre. Alkali treated fibre has the highest water absorption property, followed by untreated fibre; potassium permanganate treated fibre has the lowest water absorption property. However, benzoylation shows irregular pattern of water absorption. In addition, the aspect ratio of the treated fibres, except the benzoyl chloride treated fibre, is higher than the untreated fibre. From the results of the present study, the treated fibres from the unmanaged disposal areca nut fibre has good potential for the fabrication of a useful value added composite material product. Hence, the areca fiber reinforced natural rubber composites can be considered as a very promising material for fabrication of lightweight materials and can be effectively used in industrial sectors like automobile body building, office furniture, packaging industry, partition panels.

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