**Properties of Borassus Fruit Fiber** 

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Abstract

Borassus fruit fiber is a waste material from Borassus flour production, which is popular in

Phetchaburi province, Thailand. Natural fiber developments for textile industry create value added

benefits of agricultural waste material and increase farmers' income. This paper focused on Borassus

fruit fibers by studying their important properties such as physical, chemical, mechanical and structural

aspects when they were scoured and bleached. They were determined according to ASTM standard and

SEM, XRD and FTIR instruments were used. Highlight properties of fibers indicated that fibers consisted

of three main components namely,  $\alpha$ -cellulose, hemicellulose and lignin. When they were scoured and

bleached, results showed that the percentage of  $\alpha$ -cellulose was increased on scoured and bleached

fibers but the percentage of hemicellulose and lignin was decreased. The crystallinity of untreated fiber

was 67.16% and it was increased to 68.97% and 71.50% when the fibers were scoured and bleached

respectively. Fiber diameter, length and linear density after scouring and bleaching were reduced. Tensile

strength property was increased on scoured fiber but it was decreased on bleached fiber. For fiber

surface morphology, we found that scoured and bleached fibers were cleaner than untreated fiber and

numerous pores or pits were revealed on bleached fiber surface.

Keywords: Borassus fruit fiber, Cellulose fiber properties, FTIR, SEM and XRD

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#### 1. Introduction

Thailand is an agricultural country. Thai government has encouraged production of various economic crops for exports such as rice, corn, sugarcane, cassava, oil palm, and rubber tree [1]. Harvesting or transformation into instant products, results in a large amount of crop residues/waste. This issue has increased an awareness of the Thai textile industry about environmental friendliness, valued added textiles, and eco-innovative textiles, and led to a greater focus on recycling and use of waste as raw materials, eco-design, renewable material, waste management, and novel technology. Many textile companies have tried to develop upstream production by using waste materials from such crops as pineapple, banana, and coconut fibers to serve the textile [2].

Borassus (Borassus flabellifer L.) is a plant in the Arecaceae family. Other common names of Borassus are Palmyrah or Toddy palm. Borassus is one of the most important economic plants. It has a range of utilization especially in Cambodia and India. Its fruit is used to make food products. The ripe fruit shell is used as a fiber source for textile fiber, "cellulose fiber" [3-4]. In Thailand also, Borassus is an economic crop in many provinces such as Sukhothai, Suphan Buri, Phetchaburi and Songkhla. Phetchaburi province has the largest quantity of Borassus. There are about 336.027 rai of land used for growing the Borassus trees. The harvesting areas are about 296,125 rai and about 101,156 tonnes a year of Borassus product are produced [5]. Our study of Borassus fruit fiber properties in Thailand is a guideline for future development in the textile industry. In addition, it is creating value added function for waste material and increasing the farmers' income.

#### 2. Materials and methods

#### 2.1 Materials

Materials used this study were matured Borassus fruit fiber, collected from the Borassus flour production in Phetchaburi province, Thailand, and commercial grade chemicals such as detergent, amylase enzyme, hydrogen peroxide, sodium hydroxide, and sodium silicate.

#### 2.2 Methods

## 2.2.1 Samples preparation

Borassus fruit fibers, were rinsed with water to remove dirt, mud and other water-soluble impurities until clean, and then dried in sunlight for two days. Then they were scoured and bleached [6].

### 2.2.1.1. Scouring

The 100 g of dried Borassus fruit fibers were scoured in a solution (1:20 fiber to liquor ratio) containing 0.5% detergent, 2% sodium hydroxide at 100  $^{\circ}$ C for 1 h. The fibers were rinsed in water until clean.

# 2.2.1.2 Bleaching

Scoured fibers were bleached in a solution (1:20 fiber to liquor ratio) containing 3% hydrogen peroxide, 0.25% sodium hydroxide, 0.5% sodium silicate, 1% sodium carbonate at 100 °C for 20 minutes. The fibers were rinsed in water until clean.

# 2.2.2 Fiber properties testing

## 2.2.2.1 Fiber composition

The percentages of  $\alpha$ -cellulose, hemicellulose, and lignin were analyzed using the Van Soest detergent procedure (acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL)) [7-8].

#### 2.2.2.2 Fiber length

Fiber length was tested according to the ASTM D 5103 – 01 Standard test method for length and length Distribution of Manufactured Staple Fibers (Single-Fiber Test) [9]. Each sample was taken for measurement by measuring scale and an average length was reported.

### 2.2.2.3 Linear density

Linear density was measured according to the ASTM D1577-01 Standard test method for linear density of textile fiber [10] at standard atmospheric conditions. Sample was measured for weight (W) and length (L) and fiber denier was calculated using equation (1).

Denier = 
$$\frac{(W \times 9000)}{(N \times L)}$$
 (1)

Where N is number of fibers in each sample

## 2.2.2.4 Tensile properties

Tensile strength was determined according to the ASTM D3822-01 Standard test method for tensile properties of single textile fibers [11] using Hounsfield Universal Testing Machine-TX0121- Model H50KS at a cross head speed of 5 mm/min, maintaining at a gauge length of 50 mm. An average of 10 test results was reported.

# 2.2.2.5 Scanning electron microscope (SEM)

Fiber diameter and surface morphology were investigated using a Scanning electron microscope (SEM). Samples were coated with gold using a JEOL- model JFC-1600 AUTO fine Coater. SEM images of the fiber samples were recorded using a JEOL- model JSM-6510 Scanning electron microscope with accelerating voltage of 5 KV.

### 2.2.2.6 X-ray diffraction (XRD)

Fiber crystallinity was determined using X-ray diffraction (XRD). The wide-angle X-ray diffraction spectra of the fibers were recorded on an X' Pert Pro, PANalytical model. The system has a rotating anode generator with a copper target and wide-angle powder goniometer. The samples were scanned between the angles (2 $\Theta$ ) from 5 to 50° to obtain the equatorial reflections. Percentage crystallinity was determined using the wide-angle X-ray diffraction counts at  $2\theta$  angle close to  $22^\circ$ and 18°. Among these, the low angle reflection (18°) is of low intensity, representing I18 of amorphous region and the other reflection (22°) has higher intensity, and it represented I22 of crystalline region in cellulosic fiber [12]. Percentage of crystalline (%crystalline) was calculated using equations (2).

% Crystalline = 
$$\frac{I_{22}}{(I_{22}+I_{18})} \times 100$$
 (2)

Where I22 and I18 are crystalline and amorphous intensities at  $2\theta$  scale close to 22° and 18°, respectively.

2.2.2.7 Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier Transform Infrared Spectroscopy (FT-IR) spectra of all samples run on Perkin Elmer, frontier model spectrophotometer using KBr pellets. Samples were powdered with KBr and pellets were used for recording the spectra in transmission mode in the 4000-500 cm-1 region with 32 scans in case at a resolution of 4 cm-1.

#### 3. Results and Discussion

#### 3.1 Fiber composition

Borassus fruit fiber is lignocellulosic fiber containing three main components;  $\alpha$ -cellulose, hemicellulose and lignin. Table 1 shows that untreated Borassus fruit fiber consists of 43.81% α-cellulose, 35.36% hemicellulose and 13.54% lignin. These results were slightly different to the Borassus fruit fiber from India 45.67%  $\alpha$ -cellulose. 32.76% hemicellulose and 21.53% lignin [13]. Before dyeing, fibers were scoured and bleached for improving fiber properties, cleaning and whitening. Scouring and bleaching changed the fiber composition ( $\alpha$ -cellulose, hemicellulose and lignin). Scoured fiber contained 51.77%  $\alpha$ -cellulose, 27.01% hemicellulose and 13.48% lignin and bleached fiber contained 56.25%  $\alpha$ -cellulose, 17.58% hemicellulose and 12.93% lignin. It was noteworthy that the percentages of lpha-cellulose increased because hemicellulose and lignin decreased. It might be that the hemicellulose and lignin were easily damaged by alkaline. The lpha-cellulose component of raw Borassus fruit fiber was similar to coir fiber that had 43.44%  $\alpha$ -cellulose [14], probably due to both of them being extracted from husks that cover its seed.

Table 1 Borassus fruit fiber composition

Fiber type	Lignin (%)	Hemicellulose (%)	α-cellulose (%)
Raw fiber	13.54	35.36	43.81
Scoured fiber	13.48	27.01	51.77
Bleached fiber	12.93	17.58	56.25

### 3.2 Fiber length

Table 2 shows that the average length of raw Borassus fruit fiber was 12.50 cm for crude fiber and 6.20 cm for fine fiber. The fiber length after scouring and bleaching, its average length was 11.06 cm for crude fiber and 5.69 cm for fine fiber and the bleached fiber was 10.60 cm for crude fiber and 5.14 cm for fine fiber. It was noticed that the fiber lengths reduced after scouring and bleaching due to losing the hemicellulose and lignin. Bleaching with hydrogen peroxide decreased cellulose fiber length and this method made the fiber more flexible [15].

Table 2 Fiber length

Fiber Samples	Max (cm)	Min (cm)	Average $(\overline{X})$	SD
Raw fiber (Crude)	27	1	12.50	12.93
Raw fiber (Fine)	21	1	6.20	6.06
Scoured fiber (Crude)	25	1	11.06	14.82
Scoured fiber (Fine)	20	1	5.69	5.02
Bleached fiber (Crude)	22	1	10.60	9.01
Bleached fiber (Fine)	18	1	5.14	5.65

# 3.3 Linear density and tenacity properties of Borassus fruit fiber

Borassus fruit contains 30-40% of crude fiber and 60-70% of fine fiber. Linear density of Borassus fruit fiber shows in Table 3 to be 337 and 85 denier for raw crude and raw fine fibers, respectively. They were reduced to 274 and 62 denier after scouring and further reduced to 228 and 55 denier after bleaching. It was observed that the size of scoured and bleached fibers were smaller than raw fiber because fiber linear density reduced after scouring and bleaching. This is consistent with Boopathi's research result that alkali treatment decrease the fiber diameter; fiber diameter was not uniform throughout the length and fiber diameter was getting reduced with respect to the increase in alkali treatment percentage [16].

For tensile properties of fibers, data supported that the tenacity at break of raw fiber were 223gf/ den for crude fiber, and 6gf/den for fine fiber. Their strength increased on scouring treatment to 262gf/ den for crude fiber and 7gf/den for fine fiber, on the other hand, after bleaching the fiber strength was decreased to 204gf/den for crude fiber and 5gf/den for fine fiber. The percentages of fiber elongation were reduced when they were scoured and bleached. Crude fibers elongation percentage was reduced from 56 to 47 and 46 and fine fibers elongation percentage was reduced from 42 to 40 and 39 respectively (Table 3). It is noticed that the fiber strength increased with scouring but decreased with bleaching. It might be due to alkali concentration of bleaching was stronger than scouring because of the higher concentration percentage and the longer soaking time, the more fiber strength reduced [17-18].

Table 3 Linear density and tenacity properties of the Borassus fruit fiber

Fiber Samples	Linear density (denier)	Tenacity (gf/den)	Elongation (%)
Raw fiber (Crude)	337	337 223	
Raw fiber (Fine)	85	6	42
Scoured fiber (Crude)	274	262	47
Scoured fiber (Fine)	62	7	40
Bleached fiber (Crude)	228	204	46
Bleached fiber (Fine)	55	5	39

# 3.4 Scanning electron microscope (SEM)

SEM technique was used to investigate the cross section and surface morphology of Burassus fruit fiber. Figure 1 shows SEM micrographs for cross sections of (a) raw fiber, (c) scoured fiber and (e) bleached fiber. The fiber cross section outline reveals that most fibers have an oval shape and they have small air cavities all over their structure. When fibers were scoured and bleached, their cross sectional structures were not different in appearance. For fiber diameters, from the SEM images it was noticed that the diameters of raw, scoured and bleached fibers were almost similar. The diameter range of crude fibers was between 200-425 µm and between 25-150 µm for fine fiber. It was noted that the dimensions started much smaller than Borassus fruit fiber from India, which has dimensions starting from 90 µm [19]. This difference in size and quality of fiber might be due to the different research method and different

species, whether, soil or planting of Borassus.

Fiber surface morphologies are shown in Figure 1 as (b) raw fiber, (d) scoured fiber and (f) bleached fiber. Figure 1(b) indicates impurities on raw fiber. Scoured fiber's surface seems to be smooth in comparison with raw fiber (see Figure 1d). On the other hand, micrograph of bleached fiber in Figure 1(f) reveals a lot of micro pores or pits on its surface. Moreover bleached fiber surface become rougher than raw and scoured fibers and also showed some scrapes along the surface.

In this case, it was probably due to some hemicellulose, lignin and impurities being removed from fiber. Consequently, in this study, the results of bleaching treatment were very important for modification of fiber surface morphology to enhance properties such as whiteness, flexibility, heat transfer, sound absorption and composites material.

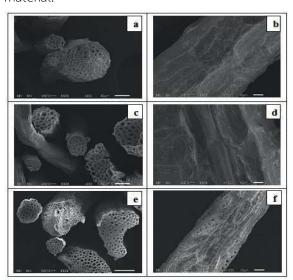


Figure 1 Scanning electron micrographs of (a) cross section of raw fiber,

- (b) surface of raw fiber,
- (c) cross section of scoured fiber.
- (d) surface of scoured fiber,
- (e) cross section of bleached fiber, and (f) surface of bleached fiber

# 3.5 X-ray diffraction (XRD)

Cellulose fiber contains crystalline and amorphous regions in different quantities. In this research X-ray diffraction (XRD) was used to investigate the crystalline percentage and crystalline index in Borassus fruit fiber. From Figure 2, results showed that peaks of around 22° and 16° 2 $\Theta$  reflection were assigned to the crystalline phase and peak of 18°  $2\theta$  reflection was assigned to amorphous phase. This information was used to calculate for crystallinity percentage shown in Table 4. Results indicated that the percentage of crystalline of raw fiber was 67.16%, of scoured fiber was 68.97% and of bleached fiber was 71.50%, respectively. It was observed that the crystalline percentage shown in scoured and bleached fibers were higher than in raw fiber due to the removal of hemicellulose and lignin from the amorphous area in the fiber structure [20-21].

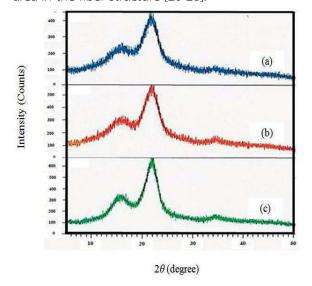


Figure 2 XRD analysis

(a) Untreated fiber,

(b) Scoured fiber,

and (c) Bleached fiber

Table 4 Percentage crystallinity (% Crystalline) of raw fiber, scoured fiber, and bleached fiber

<u> </u>	At 2 $ heta$ (degree)		0/ 6 1 11:
Samples	l <sub>22</sub>	    18	% Crystalline
Raw fiber	450	220	67.16
Scoured fiber	600	270	68.97
Bleached fiber	680	270	71.50

# 3.6 Fourier transforms infrared spectroscopy (FT-IR)

FT-IR analysis was done to identify chemical structure of fiber. Figure 3 shows evidence of structural modification of raw, scoured and bleached Borassus fruit fibers. The peak area within region 4000-2995 cm<sup>-1</sup> showed OH stretching vibration of cellulose [22]. In this case, the peaks were located at 3321 and 2995 cm<sup>-1</sup> for all samples, there are no differences or changes. It indicated that the cellulose was not decomposed from scouring and bleaching treatments. The absorption bands around 1765-1715 cm<sup>-1</sup> corresponded to C=O stretch of carbonyl group of hemicellulose of the fiber [22]. In this research, results showed that the peak at 1725 cm<sup>-1</sup> of raw and scoured fibers still appeared but it disappeared in bleached fiber. This phenomenon was an indication of the removal of hemicellulose from Borassus fruit fibers during bleaching treatment. The characteristic peak located at about 1632 cm<sup>-1</sup> corresponded to C=C stretch of benzene and the 1613 cm<sup>-1</sup> peak corresponded to C=C stretch of Aromatic skeletal of lignin [22]. These peaks were seen in raw fiber but became weaker, or were reduced with the scoured and bleached fibers. Lignin was also seen at peak 1247 cm<sup>-1</sup> corresponding

to C-O stretching vibration of acetyl group [23]. This peak appeared in both raw and scoured fiber but was reduced with the bleached fiber. We can be observed that lignin was removed by bleaching treatment. From the research results we can be interpreted that the hemicellulose and lignin components were the most damaged with bleaching treatment. The scouring treatment was able to eliminate just some hemicellulose, lignin and impurities on the fiber surface because this treatment involved a dilute alkaline solution. These results were consistent with the composition analysis and SEM results.

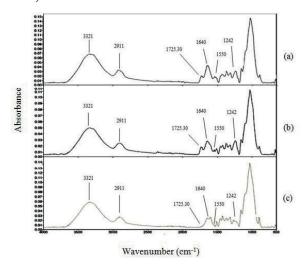


Figure 3 FTIR analysis

(a) Raw fiber,

(b) Scoured fiber,

and (c) Bleached fiber

### 4. Conclusions

Borassus fruit fibers used in this study were waste material from Borassus flour production in Phetchaburi province, Thailand. They were scoured and bleached and their properties were investigated. Results indicated that the fiber composition contained three main components, which were

 $\alpha$ -cellulose, hemicellulose and lignin. After scouring and bleaching,  $\alpha$ -cellulose percentage increased because the hemicellulose and lignin percentages decreased. Fiber diameter, length and linear density were reduced after treatments. Tensile strength of scoured fiber increased from that of raw fiber but it decreased after bleaching. For the fiber surface morphology, we found that scoured and bleached fibers were cleaner than raw fiber and numerous pores or pits appeared on bleached fiber surface. These results were supported by XRD, FT-IR and SEM analysis of raw, scoured and bleached fibers. From research results, Borassus fruit fibers properties are good for products development for example; textile products, green composite material, thermal protection material and sound absorption material etc.

# 5. Acknowledgements

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## 6. References

- [1] Farmthailand. 2013. **The export of agricultural products of Thailand.** AvailableSource: http://www.farmthailand.com/399, April 16, 2015.
- [2] Sirikasemlert, C. 2009. Eco textile, pp. 86-91 In Thailand Textile Institute., eds. Innovative Textiles. P2 design and print co., Ltd. Bangkok, Thailand.

- [3] Palmpedia. n.d. **Borassus flabellifer.** Available Source: http://www.palmpedia.net/wiki/Borassus flabellifer, June 3, 2015.
- [4] Philippine medicinal plants. n.d. Palmira.

  AvailableSource: http://stuartxchange.com/
  Palmira.html, June 3, 2015.
- [5] Phetchaburi Provincial Agricultural Extension Office. 2016. Agricultural areas (2015). Available Source: www.phetchaburi.doae. go.th, May 3, 2016.
- [6] Mongkholrattanasit, R., J. Klayjoy, K. Arayaklua, K. Mahain, S. Jankeaw, S. Chonsakorn and N. Panrattanasin. 2014. Development of Borassus Fruit Fiber for Heat Insulation and Application for Technical Textile. Thailand Textile Institute.
- [7] Goering, HK and PJ. Van Soest. 1970. Forage fiber analyses (apparatus, reagent, procedures, and some applications).

  Agriculture Handbook No. 379, Agric Res Serv, USDA, Washington, DC, USA.
- [8] Van soest PJ, JB Robertson, and BA Lewis. 1991. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. J Dairy Zci 74: 3583-3597.
- [9] ASTM International. 2005a. ASTM D 5103 01 Standard test method for length and length Distribution of Manufactured Staple Fibers (Single-Fiber Test). ASTM Standards Book of Standard, vol. 07.01: D 76-D 4391.
- [10] ASTM International. 2005b. ASTM D1577-01 Standard test method for linear density of textile fiber. **ASTM Standards Book of Standard,** vol. 07.01: D 76-D 4391.

- [11] ASTM International. 2005c. ASTM D3822-01 Standard test method for tensile properties of single textile fiber. **ASTM Standards Book of Standard,** vol. 07.0: D 76-D 4391.
- [12] Susheel, K. and B.S. Kaith. 2009. Synthesis of flax-g-copolymers under pressure for use in phenolic composites as reinforcement. J. Chil. Chem. Soc., 54, No 2.
- [13] Obi Reddy, K., B. R. Guduri and A. Varada Rajulu. 2009. Structural Characterization and Tensile Properties of Borassus Fruit Fibers. Journal of Applied Polymer Science 114: 603–611.
- [14] Mathai, M P. 2005. Coir. **Bast and other plant fibers.** Woodhead Publishing Limited. Cambridge, England.
- [15] Chen, Y., J. Wan, Y. Ma, X. Dong, Y. Wang and M. Huang. 2015. Fiber properties of de-inked old newspaper plum after bleaching with hydrogen peroxide. Bioresources 10(1): 1857-1868.
- [16] Boopathi, L., P.S. Sampath and K. Mylsamy. 2012. Investigation of Physical, Chemical and Mechanical Properties of Raw and Alkali Treated Borassus Fruit Fiber. **Composites:** Part B 43: 3044–3052.
- [17] Bachtiar, D., S. M. Sapuan and M. M. Hamdan. 2008. The effect of alkaline treatment on tensile properties of sugar palm fibre reinforced epoxy composites. Materials & Design, 29: 1285-1290.

- [18] Obi Reddy, M. Shukla, C.U. Maheswari and A. V. Rajulu. 2012. Mechanical and physical characterization of sodium hydroxide treated Borassus fruit fibers. Journal of Forestry Research 23(4): 667-674
- [19] Saravanan, D., N. Pallavi, R. Balaji and R. Parthiban. 2008. Investigations into structural aspects of Borassus flabellifer L (palmyrah palm) fruit fibres. Journal of the Textile Institute 99 (2): 133-140
- [20] Rayung, M., N. A. Ibrahim, N. Zainuddin, W. Z. Saad, N. I. A. Razak, and B. W. Chieng. 2014.

  The effect of fiber bleaching treatment on the properties of poly(lactic acid)/oil palm empty fruit bunch fiber composites.

  Int. J. Mol. Sci., 15: 14728-14742
- [21] Sgriccia, N., M.C. Hawley, and M. Misra. 2008.

  Characterization of natural fiber surface and natural fiber composites. Composites: Part A. 1632-1637
- [22] Moran, J.I., V.A. Alvarez, V.P. Cyras and A. Va´zquez. 2008. Extraction of cellulose and Preparation of nanocellulose from sisal fiber. **Cellulose**. 15: 149-159
- [23] Zhang, X., F. Wang and L. M. Keer. 2015.

  Influence of surface modification on the microstructure and thermos-mechanical properties of bamboo fiber. Materials, 8, 6597-6608.