

## Effect of Acid Hydrolysis Treated Pineapple Fiber in Plasticized Polylactic Acid Composite

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### ARTICLE INFO

#### Article history:

Received 5 June 2018

Received in revised form 24 October 2018

Accepted 2 November 2018

Available online 6 November 2018

#### Keywords:

Pineapple leaf fiber, surface treatment,  
acid hydrolysis, polylactic acid, tensile  
strength

### ABSTRACT

Pineapple leaf is a residue mainly from pineapple food industry which is causing environmental problems by its lack of proper waste disposal method. Therefore, it can be utilized as filler for the reinforcement of PLA which is brittle in nature. Treated fiber from pineapple leaf fiber (PALF) was derived through acid hydrolysis method. The morphological properties and chemical structure of treated PALF were determined using scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). Plasticized PLA/treated PALF was prepared by solvent casting method at 1wt%, 3wt%, 5wt% and 7wt% loading rates of acid hydrolysis treated PALF. Mechanical properties of composite were characterized through tensile test. FTIR analysis shows significant changes in the chemical properties of the raw PALF and treated PALF. SEM image reveals that acid hydrolysis treated PALF displays a rod like structure and smooth surface as the effect of hemicellulose removal. The findings of this research is expected to decrease the amount of waste accumulated in the environment due to non-degradable materials and the pineapple industry residues.

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## 1. Introduction

Natural resources such as plant fiber is widely utilized as an alternative raw material in the industries to replace fossil-fuel based products. Cellulose is one of the most abundant natural polymer present in the world. Being renewable, biodegradable and non-toxic, it could be the source of environmental friendly and biocompatible product. It is obtained from various resources such as wood, cotton, grass, blast fibers, tunicates and bacteria, with wood being the major contributor to the cellulose amount. Pineapple leaf is one of the many sources of cellulose. Pineapple fruit is a popular fruit crop grown in Malaysia for pineapple based food products such as canned pineapple, juice, concentrate and jams. However, the production of processed food also formed its byproducts, which is residual pulp, peels, stems, and leaves. The large amount of unusable material may cause serious environmental problems as it is left in agro industrial yards without any significance and

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commercial value. A proper disposal of this waste is expensive and not cost effective leading to eliminating of this waste by burning and decomposing [1]. Therefore, utilization of pineapple waste residue has become more necessary and can be done through various approaches such as extracting cellulose from pineapple leaf fiber to be used as reinforcement material in biodegradable polymer composites.

## 2. Methodology

### 2.1 Pretreated PALF

Processed PALF were pretreated by cutting PALF to 3-4 mm long and subjected to chemical treatment using mild sulfuric acid ( $\text{H}_2\text{SO}_4$ ) 4 wt% at  $80^\circ\text{C}$  for 2 hours. It was followed by washing with water to remove the acid and dried in oven for 15 hours.

### 2.2 Acid Hydrolysis Treated PALF

Subsequently the PALF was subjected to another treatment using sodium hydroxide ( $\text{NaOH}$ ) at  $80^\circ\text{C}$  for 2 hours before washed and dried in oven for another 15 hours [2]. The pretreated PALF was immersed in 64wt%  $\text{H}_2\text{SO}_4$  at  $45^\circ\text{C}$  for 45 minutes then diluted 10 fold using water to stop hydrolysis process. The solution was centrifuged at 7000 rpm for 10 minute to separate acid from the treated fiber. It was washed with water until reaches neutral pH before dried in oven at  $60^\circ\text{C}$  for 15 hours [3].

## 2. Characterization

A portion of pretreated PALF was taken out from the total pretreated PALF prior to acid hydrolysis treatment. All the treated fibers were dried in oven to powder form and analyzed using FTIR as it is in the range of  $4000\text{ cm}^{-1} - 380\text{ cm}^{-1}$  [3]. Morphology of the treated PALFs is investigated using Scanning Electron Microscopy (JEOL, JSM-5600, UK). Samples are observed at 100 x, 500 x and 1000 x with 15 kV accelerating voltage. All the samples are sputter coated with palladium.

## 3. Results

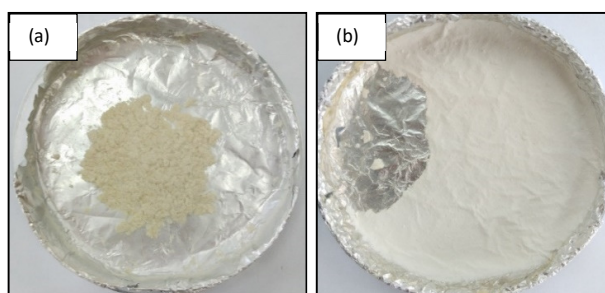
### 3.1 Visual Observation

Figure 1 shows optical image of pretreated PALF and acid hydrolysis treated PALF. Fig.1.(a) shows the pretreated PALF is in a light brown color which signifies that there is a change in the surface structure of the fiber after mild acid and mild alkali treatments. From Fig.1.(b), acid hydrolysis treated PALF are of white color, which shows that it is cellulosic. Acid hydrolysis treatment further removed the leftover hemicellulose and lignin from the fiber after pretreatment process.

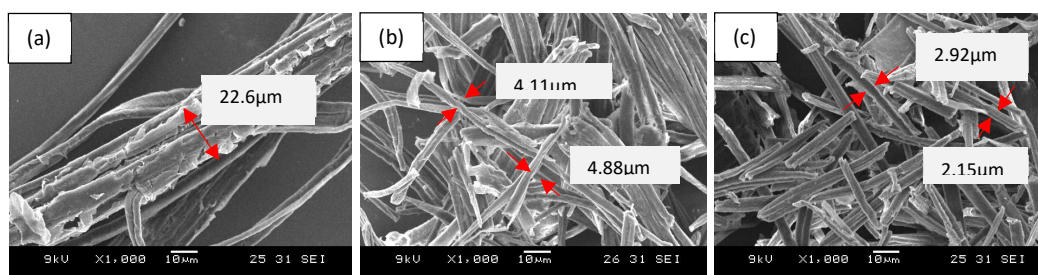
### 3.2 Surface Morphology of Fibers

SEM micrographs of pretreated PALF and acid hydrolysis treated PALF in comparison with untreated PALF were taken. Diameter of untreated PALF was measured to be  $22.6\text{ }\mu\text{m}$ . The fiber surface was rough for the presence of impurities and lignocellulosic components like hemicellulose and lignin. From Fig.2, the effect of pretreatment and sulfuric acid hydrolysis on the surface of PALF are observed. After pretreatment by mild acid and mild alkali, the diameter of fibers significantly reduced to an average of  $4.5\text{ }\mu\text{m}$ . Mild Acid dissolved hemicellulose from the fiber bunch followed by

alkali treatment to disrupt the lignin structure of PALF [2]. The fibers are not fully fibrillated from the main fiber as observed in Fig.2.(b). According to Tanpichai and Witayakran [4], the fibrillated fiber obtained was due to the lignin adhesion removed by alkaline treatment. Fig.2.(c) reveals that after treatment with 64 wt%  $H_2SO_4$ , PALF is well isolated with an average diameter of 2.5  $\mu m$  and exhibit a rod like structure with a smooth surface contributed by the removal of hemicellulose and lignin. Reduction in the diameter of fiber after acid hydrolysis treatment can be seen which might be contributed by the removal of amorphous region from cellulose of PALF. Upon acid hydrolysis, the amorphous region disintegrates, leaving single strands of crystalline cellulose as in Fig.2.(c) [2].



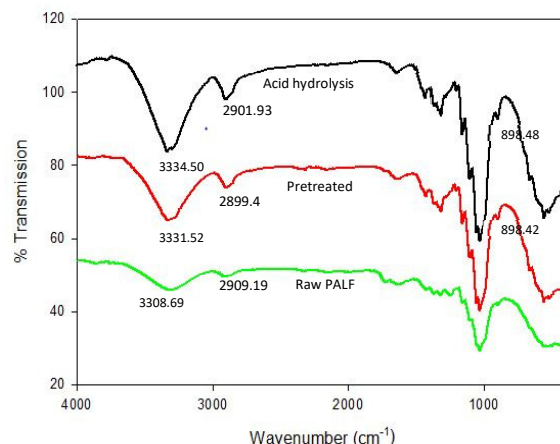
**Fig. 1.** (a) Pretreated PALF, (b) Acid hydrolysis treated PALF



**Fig. 2.** SEM micrographs of PALF before and after treatment (a) PALF (b) Pretreated PALF (c) Acid hydrolysis treated PALF

### 3.3 Chemical Properties of Fibers

FTIR was used to analyze the effect of pretreatment followed by acid hydrolysis on the constituent of the fibers. The spectra of raw PALF, pretreated PALF and acid hydrolysed PALF is shown in Fig.3 below. A broad peak in  $3308.69\text{ cm}^{-1}$  region of untreated PALF is due to O-H stretching. For pretreated PALF and acid hydrolysis treated PALF, spectrum shifted to  $3331.52\text{ cm}^{-1}$  and  $3334.50\text{ cm}^{-1}$  as the result of increase of free hydroxyl group which indicates that cellulose group remained throughout the treatments. This in line with the work of B. M. Lee *et al.*, [5]. The wavenumber of  $2909.19\text{ cm}^{-1}$  region in untreated PALF is attributed to C-H stretching vibration of cellulose material. The peak is more distinct after pretreatment and acid hydrolysis of PALF [6]. The peak between the wavenumbers of  $1210\text{ cm}^{-1}$  and  $900\text{ cm}^{-1}$  can be related to cellulose and hemicellulose structure.  $1029.89\text{ cm}^{-1}$  is the C-O-H stretching of alcohol group in cellulose, and the peak at  $898.4\text{ cm}^{-1}$  is the  $\beta$  – 1,4 – glycosidic bond that makes the repeating unit in cellulose and is also signifying the amorphous absorption band. After the two treatments, the peak is visible and stronger than in the untreated PALF which can mean the removal of amorphous region from cellulose of PALF [7].



**Fig. 3.** FTIR spectra of raw PALF, Pretreated PALF and Acid hydrolysis treated PALF

#### 4. Conclusions

The objective of this research was to prepare treated fiber from PALF using acid hydrolysis. It was achieved by pretreatment of PALF followed by 64wt% sulfuric acid hydrolysis at 45°C for 45 minute. The second objective was to characterize the morphological properties and chemical structure of the fiber. This was carried out through SEM and FTIR analysis. Significant changes in the chemical properties of the raw PALF and treated PALF have been observed. The acid hydrolysis treated PALF displays a rod like structure and smooth surface as the effect of hemicellulose removal.

#### References

- [1] Upadhyay, Atul, Jeewan Prava Lama, and Shinkichi Tawata. "Utilization of pineapple waste: a review." *Journal of Food Science and Technology Nepal* 6 (2010): 10-18.
- [2] Lee, H. V., S. B. A. Hamid, and S. K. Zain. "Conversion of lignocellulosic biomass to nanocellulose: structure and chemical process." *The Scientific World Journal* 2014 (2014).
- [3] dos Santos, Roni Marcos, Wilson Pires Flauzino Neto, Hudson Alves Silvério, Douglas Ferreira Martins, Noélcio Oliveira Dantas, and Daniel Pasquini. "Cellulose nanocrystals from pineapple leaf, a new approach for the reuse of this agro-waste." *Industrial Crops and Products* 50 (2013): 707-714.
- [4] Tanpichai, Supachok, and Suteera Witayakran. "All-cellulose composite laminates prepared from pineapple leaf fibers treated with steam explosion and alkaline treatment." *Journal of Reinforced Plastics and Composites* 36, no. 16 (2017): 1146-1155.
- [5] Lee, Byoung-Min, Jin-Young Lee, Phil-Hyun Kang, Sung-Kwon Hong, and Joon-Pyo Jeun. "Improved pretreatment process using an electron beam for optimization of glucose yield with high selectivity." *Applied biochemistry and biotechnology* 174, no. 4 (2014): 1548-1557.
- [6] Kumar, Rajeev, and Charles E. Wyman. "Cellulase adsorption and relationship to features of corn stover solids produced by leading pretreatments." *Biotechnology and Bioengineering* 103, no. 2 (2009): 252-267.
- [7] Ciolacu, Diana, Florin Ciolacu, and Valentin I. Popa. "Amorphous cellulose—structure and characterization." *Cellulose chemistry and technology* 45, no. 1 (2011): 13.