

# Polylactic Acid/Empty Fruit Bunch Fiber Biocomposite: Influence of Alkaline and Silane Treatment on the Mechanical Properties

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**Abstract**—Biocomposites were prepared by combining polylactic acid (PLA) with treated or untreated empty fruit bunch (EFB) fiber by melt compounding method to observe the effects of fiber surface treatments on the mechanical properties of the biocomposite. Surface treatment of the fiber was conducted using alkali followed by silane treatment. It was found that treated fibers showed superior mechanical properties of the reinforced biocomposite as compared with untreated fiber reinforced biocomposites due to the enhanced adhesion between the EFB fiber and the PLA matrix.

**Index Terms**—Biocomposites, empty fruit bunch, polylactic acid, surface treatments

## I. INTRODUCTION

Composites have lately received much attention in research, developmental sector as well as industry due to the high demand in applications such as construction, automotive to aerospace industries [1]. Composites used are usually composed of synthetic polymer as the matrix and synthetic fiber such as glass, aramid or carbon fiber as the reinforcement. However, both matrix and reinforcement are the source of rising environmental problems since they do not degrade in landfills or composting environments [2]. Thus, biocomposites have been developed as an alternative to these composites as they offer environmental advantages at the end of their cycle due to their biodegradability [3].

Polylactic acid (PLA) a corn based polymer has recently been introduced commercially for products where biodegradability is required [4]. Currently, PLA is the major thermoplastic polymer made from renewable resources which is produced in large scales with a capacity of 140 000 tons per year [5]. PLA provides good aesthetics, strength and easy processability in most equipment and is thus used in various areas of applications such as fiber/textile, food packaging, film and interior automotive parts [6]. However, PLA has its drawback such as brittleness and fast degradation above its glass transition temperature [7], [8] and thus needs modification for enhanced performance.

Reinforcement can be achieved by using natural fibers in order to improve mechanical and thermal properties of PLA [9]. Natural fibers have reinforcing efficiency which is

related to the nature of their cellulose and crystallinity. They are also biodegradable, renewable, harmless to humans, less abrasive to processing equipments and have comparable mechanical properties with inorganic fibers [10]. A potential natural fiber which has been the subject of studies by many researchers is empty fruit bunch (EFB). EFB is a tough fiber produced as a by-product of palm oil extraction [11].

Interfacial adhesion between matrix and fiber is usually an obstacle due to their different polarities which results in diminished composite properties. However, this can be improved by using chemical modification of the fiber, either by chemical solution [7] or free radical reaction [12], use of modified thermoplastic containing a compound capable of interacting with the fiber [13], or addition of a third component (a compatibiliser) with the ability to interact simultaneously at the interface with the thermoplastic and the fiber [14], [15].

In this paper, two surface treatments methods have been used; EFB fiber treated with alkaline solution followed by silane coupling agent. The main aim of this research is to investigate the influence of alkaline and silane treatments to the mechanical properties of EFB/PLA biocomposites.

## II. EXPERIMENTAL

### A. Fiber Surface Treatment

EFB fibers were collected from Sime Darby Plantation in Labu, Negeri Sembilan and were dried for three days before grinding to obtain a uniform size of 400  $\mu\text{m}$ . The fibers were then soaked in hot distilled water at 70°C for 4 to 6 hours to remove impurities and large particles [7]. Next, the fibers were dried in an air oven at 105°C for 12 hours to remove the moisture [8]. These dried fibers are designated as untreated fibers.

The untreated EFB fibers were initially treated with alkaline solution to obtain cleaner and rougher fiber surface which provides both mechanical interlocking and the bonding reaction due to exposure of the hydroxyl groups to the matrix [16]. Treatment was conducted by soaking the fibers in (5% w/v) sodium hydroxide (NaOH), obtained from Bendosen Laboratory Chemicals, solution for 2 hours at room temperature before washed with distilled water containing a few drops of acetic acid and followed by distilled water until all NaOH was removed by checking the pH. After washing, the fibers were left to dry for 2 days at room temperature. The fibers were then dried in air for 6

hours followed by drying in an oven at 80°C for another 6 hours. Subsequently, the fibers were silane treated to improve the degree of cross-linking between the matrix and

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the fiber. This was done by dissolving the alkaline treated fibers in 5 wt% 3-Aminopropyltriethoxysilane (APS), purchased from Sigma-Aldrich, (weight percentage compared to fiber) for hydrolysis in a mixture of water-ethanol (40:60 w/w). The pH of the solution was adjusted to 4 by adding acetic acid and stirred continuously for 1 hour. The fibers were then soaked in the solution for 3 hours. Next, the fibers were washed and left to dry for 3 days. Finally, the fibers were oven dried at 80°C for 12 hours.

TABLE I: MECHANICAL PROPERTIES OF TREATED AND UNTREATED FIBER BIOCOMPOSITE

	Tensile Strength	Tensile Modulus	Flexural Strength	Flexural Modulus	Impact Strength (Izod)
Untreated fiber/biocomposite	30.2	0.9	72.1	3.1	31.4
Treated fiber/biocomposite	53.0	1.1	84.4	4.4	35.0

### B. Preparation of Biocomposites

PLA, purchased from NatureWorks LLC, was used as the base resin in this biocomposite compound. The melting temperature for this PLA is 150°C to 170°C to prevent EFB fibers degradation during processing. Prior mixing, PLA resins was dried for 2 hours at 90°C while EFB fibers were predried for 12 hours in a hot air oven at 105°C to remove moisture content. The PLA/EFB biocomposites were then prepared using an internal mixer type Haake Rheomixer at 160°C with a rotor speed of 50 rpm. The amount of fiber used was 10wt%. The biocomposites was then compression moulded using a hot press at 180°C with 150 kg/m<sup>2</sup> pressure. Molded sheets than were cut using to obtain specimen for tensile, flexural and impact testing.

### C. Testing

An INSTRON Universal Testing Machine (Model 4301) was used to measure tensile and flexural properties according to ASTM D 638 and ASTM D 790 respectively. The morphology of the fiber surface was observed using a Quanta FEG, 450 Scanning Electron Microscope (SEM) with an accelerating voltage of 10 kV at room temperature. The specimens were vacuum coated with a thin layer of platinum using JEOL JFC-1600.

## III. RESULTS AND DISCUSSION

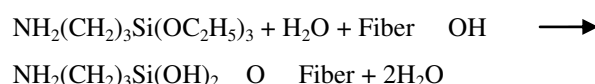
### A. Tensile test

Mechanical properties of composites depend on the adhesion between the matrix and the fiber. Generally, fiber surface modification improves biocomposites properties by decreasing the moisture absorption, increasing wettability and interfacial bond strength with the matrix [16].

The superior tensile strength of the treated biocomposites showed in Table I is due to the treatment method applied. Alkali treatment removes surface impurities and makes fiber surface rougher [7]. This is proved by SEM

picture Fig. 1(b) which shows a rougher fiber surface after NaOH treatment. This is different with Fig. 1(a) where the fiber surface is clean. The rough surfaces increase the additional sites of mechanical interlocking and promote more matrix or fiber interpenetration at the surface [17].

Silane treatment enhances compatibility by linking the fiber surfaces and matrix with its silanol and amino functional groups. The silanol group will react with fiber hydroxyl groups through an ether linkage with removal of water as shown in equation 1 [18]. Whereas, the other functional group of silane, amino group, will react with the hydroxyl groups of the PLA forming a chemical bonding linkage between the matrix and the EFB fiber. It can be concluded, surface modification improve the tensile strength of the biocomposites.



Equation 1: Reaction of silanol with fiber hydroxyl group

The increased in tensile modulus after treatment can be described by uniform dispersion of fibers in the PLA matrix lead to even distribution and transfer of stress from matrix to fiber [19]. Similar results were observed by Rosa et al. [20] and John et al. [17] who reported that alkali and silane treatment resulted in higher value of tensile properties compared with untreated fiber.

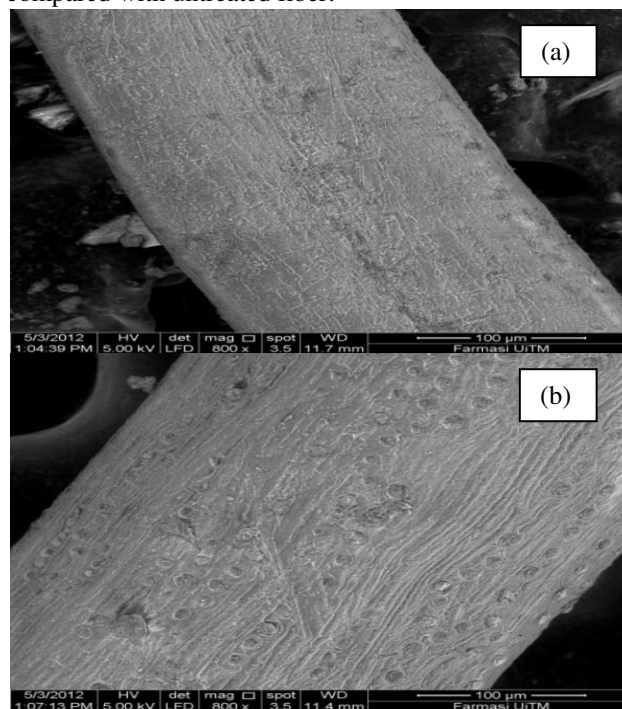


Fig. 1. SEM images of EFB fiber: (a) Untreated fiber; (b) alkali treated

### B. Flexural Test

Fiber treated with alkali and silane showed a 17% increase in flexural strength value compared with untreated fiber/biocomposite. When EFB fiber treated with alkali, the fiber becomes cleaner and rougher surface. During compounding, it facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups [21].

The treated biocomposites exhibited a superior increase in flexural modulus of 42%. There are many factors affecting the modulus of the biocomposites such as filler loading, modulus and aspect ratio [18]. The increasing in flexural modulus value after treatment suggests an efficient stress transfer between the fiber and the matrix after treatment the fiber. Another reason is good dispersion or mixing between the fiber and the matrix [9].

The notch Izod impact strength test measures the energy required for the propagation of the existing crack [22]. Impact strength of the fiber reinforced polymer composites are dependent on the fiber/matrix adhesion, toughness of the matrix and the fiber, crystalline morphology, defects in the packing of fiber/matrix, etc. [23]. Based on table 1, alkali followed by silane treatment improved the impact values of the biocomposites by 11%. This enhance value is due to the improvement of the interfacial adhesion between the fiber and the matrix. During Izod impact test, the improved interfacial adhesion provides higher resistance to crack propagation [7].

#### IV. CONCLUSIONS

In this work, biocomposites were produced from treated and untreated EFB fibers. Treated EFB fibers were surface treated with alkali and silane to increase their compatibility with the PLA matrix. Mechanical tests conducted showed improved values compared with untreated fibers. Alkali treatment is effective in removing impurities producing a rougher fiber surface which promote fiber adhesion as proven by SEM images. Silane treatment acts as bridge between the PLA matrix and the fiber. According to the results, combination alkali and silane is the best surface treatment to obtain optimum results. Further study will be focused on thermal properties of the biocomposite.

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