

Characterization and Tensile Properties of Silane Treated Bamboo Fibre Reinforced Poly (lactic) Acid Composite

Nasyitah Qabsah Mohktar¹, Mazatusziha Ahmad^{1,3*} Shaiful Rizal Masrol^{2,3}

¹Department of Chemical Engineering Technology, Faculty of Engineering Technology,
Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

²Department of Mechanical Engineering Technology, Faculty of Engineering Technology,
Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

³Bamboo Research Centre, Faculty of Engineering Technology,
Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

*Corresponding Author Designation

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Abstract: In the couple of decades, bamboo is getting interested in producing bamboo fiber as a reinforcement agent in polymer composite due to its biodegradable characteristic and mechanical properties which can replace use of synthetic polymer. *Gigantochloa Albociliata* (Bamboo Madu) is the type of bamboo that has been chosen in this study. The main objectives of this study are to characterise the effect of alkali pretreatment and silane treatment on bamboo fibre, and to investigate the effect of silane treated bamboo fibre content on mechanical properties of PLA composite film. The characterisation of bamboo fibre can be accessed via Fourier Transform Infrared Spectroscopy (FTIR) analysis and Particle Size Analyzer (PSA). The pretreatment and treatment of bamboo fibre used in this study were alkali and silane treatment respectively. Untreated and treated bamboo fibre were evaluated tensile strength using tensile test using standard ASTM D882. It can be concluded that at 8% of bamboo fibre content, results the highest tensile strength. Silane treated bamboo fibre depicts higher tensile strength than the untreated bamboo fibre.

Keywords: Bamboo Fibre, Silane treatment, Polymer composite

1. Introduction

Natural fibre-based polymer composites are a composite material consisting of a polymer matrix embedded with high-strength natural fibres, like jute, oil palm, sisal, kenaf, and bamboo. Many researchers have examined and researched the suitability, competitiveness, and capabilities of natural fibres embedded in polymeric matrices. Polymer matrix composites are mostly commercially produced composites in which resin is used as a matrix with different reinforcing materials. Polymer (resin) is classified into two types, which are thermoplastics and thermosets, which reinforce different types of fibre like natural. Polyamide (PA), Polylactic Acid (PLA), HDPE and polystyrene (PS) are the common examples of thermoplastics, whereas epoxy, polyester, and phenol-formaldehyde resin are examples of thermoset polymers. Different types of natural fibre and polymer matrix resulting in different mechanical properties. HDPE, PLA, polypropylene, and epoxy have the tensile strength of 38 MPa, 53 MPa, 31 MPa, and 50 MPa respectively [1].

Thus, in this study, it is our interest to explore the effect of bamboo fibre reinforced PLA composites. The interfacial bonding between bamboo fibre and polymers are improved by applying a coupling agent. The effect of bamboo fibre content and coupling agent on mechanical and thermal properties of composite are investigated and will be further discussed in discussion.

2. Materials and Methods

The materials and methods section, otherwise known as methodology, describes all the necessary information that is required to obtain the results of the study.

2.1 Bamboo Powder and Polylactic Acid (PLA) Pellets

The bamboo fiber (*Gigantochloa Albociliata*) was obtained via a fiber extractor machine. The fiber was dried naturally for 72 hours and was grinded using a ball mill (Planetary Ball Mill) to obtain bamboo powder. The powder was sieved via laboratory sieve to obtain 75 micron particle size. The polylactic acids used are in solid state or in pellets.

2.2 Chemical Treatments

Bamboo fiber was treated via alkali and silane treatment before mixing with PLA. Figure illustrates the process of chemical treatment for bamboo fiber. Bamboo powder was soaked into 6% concentration of sodium hydroxide solution for 24 hours for partial removal of lignin, hemicellulose, and other residues. The bamboo powder is rinsed with distilled water several times to remove excess sodium hydroxide from the surface until the water no longer indicates any alkalinity reaction. Next, the bamboo powder is dried in an oven at 80 °C for another 24 hours. The bamboo powder was soaked in a silane solution with 1% concentration, in a mixture of distilled water and ethanol. The silane solution will be made by diluting 3-aminopropyltriethoxysilane in a 40:60 w/w (weight-to-weight) ratio of ethanol and water. Subsequently, the acetic acid is used to adjust and maintain the pH of the silane solution at 4–5. The bamboo powder was soaked in the silane solution for 3 hours at room temperature with slow stirring via a magnetic stirrer. Then, bamboo powder was separated from the solution by using a Bunchner funnel, and filter paper with a vacuum pump and dried in a drying oven at 50 °C for another 24 hours.

2.3 Preparation of Bamboo Fiber Reinforced Polylactic Acid Composite

The bamboo fiber reinforced polylactic acid composite was prepared by using three equipment, which are an internal mixer (Brabender Plastograph), Plastic Granulator and hotpress (GOTECH Testing Machine) as shown in Figure. The bamboo powder and PLA were mixed manually before being fed into an internal mixer at 190°C and 25 rpm for 10 minutes. The molten-like composite was crushed into a small size at approximately 50 mm diameter via Plastic Granulator. The granulated

composite was pressed via hot press at 190°C and at pressure of 25 tons for 10 minutes to obtain a sample within 1 mm thickness.

2.3 Characterization and Testing

2.3.1 Particle Size Analysis (PSA)

The size and distribution of the particles that make up the bamboo powder is determined analytically using a particle size analyzer, or PSA. By measuring the intensity of light scattered when a laser beam travels through the dispersed particulate cellulose sample, the Mastersizer 3000 will employ the concept of laser diffraction to determine the particle size and particle size distribution of cellulose [2].

2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy is used to study the chemical structures and the presence of functional groups in the samples after silane treatment. FTIR used to analyse the effectiveness of silane treatment which can be used to examine the presence of the Si-O-Si C-H,, C=O, and C-O bonds. The device FT-IR with a maximum resolution of 0.5 wavenumber. The range of analysis is around 400 to 4000 wavenumber [3].

2.3.1 Tensile Test

A tensile testing machine (UTM) that is used to perform the tensile test of the PLA/BF composites according to ASTM D882 Standard Test Methods for Tensile Properties of Plastics with a 10kN load cell. The specimens were cut into rectangular shape with dimension of 12.7 cm × 2.5 cm (length width) as shown in Figure 1 below.

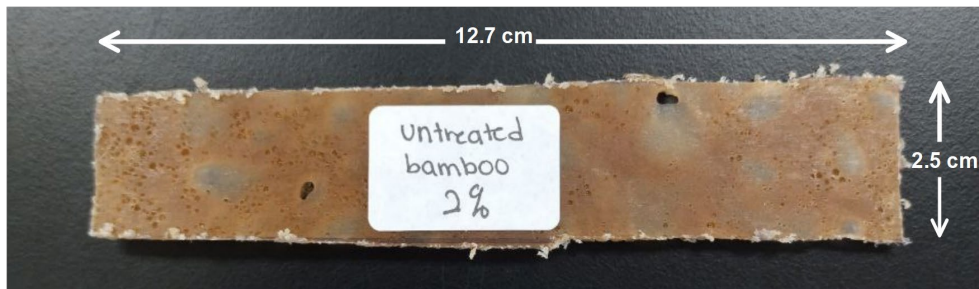


Figure 1: Dimension of specimen for tensile test

3. Results and Discussion

The result and discussion were divided into two sections: characterization of silane treated bamboo fiber via Particle Size Analysis (PSA) and Fourier Transform Infrared Spectroscopy (FTIR) analysis, and the mechanical properties of bamboo fibre reinforcement PLA composite.

3.1 Particle Size Analysis

Particle size analysis is used to characterize the size distribution of particles in a given sample. The median particle size of bamboo powder used in this work was 33.4 m. Particle size of bamboo powder with size 9.05 m was less than 10% and 69.6 m was the 90% size proportion of the bamboo powder. Table 1 illustrates the summary of the result obtained on particle size analysis.

Table 1: Particle size of bamboo powder

Sample	Size distribution	Size Classes (μm)		
Bamboo Powder	Intensity	D10	D50	D90
		9.05	33.4	69.6

Figure 2 illustrates the particle size distribution of bamboo powder. 3320m²/kg a specific surface area of bamboo powder was used in particle size analysis. The uniformity of the bamboo powder was 0.561 shows that the bamboo powder was uniform in size as the polydispersity index obtained less than 1.

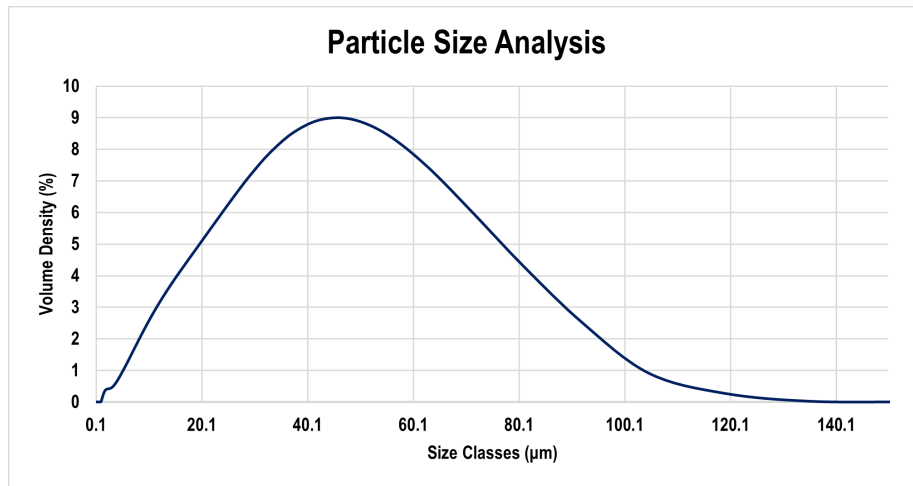


Figure 2: Particle size distribution of bamboo powder

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

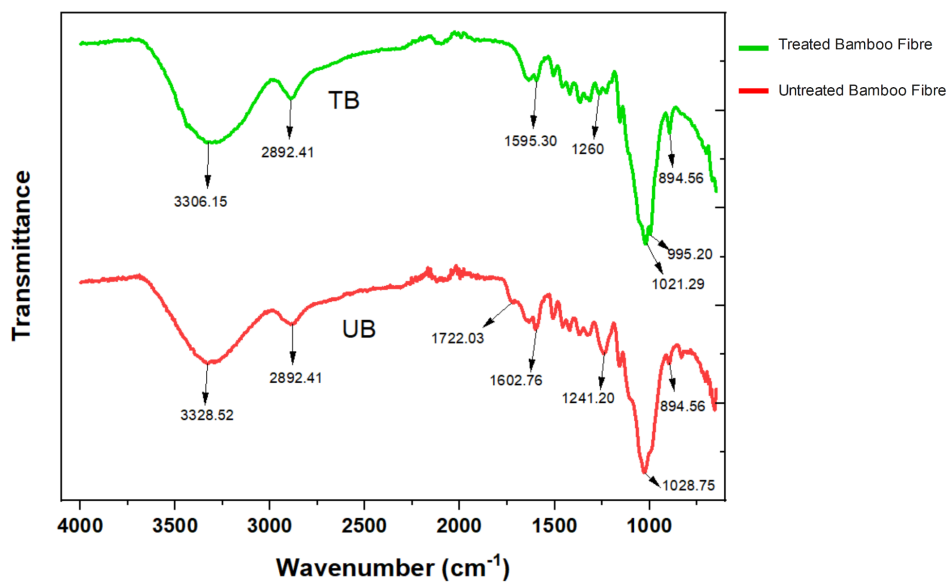


Figure 3: FTIR of treated and untreated bamboo powder with alkali pretreatment and silane treatment

The components of the bamboo fibre which are lignin, hemicellulose, carbonyl and hydroxyl group were identified in the FTIR spectrum. From the findings, the lignin stretching, C-O for untreated bamboo fibre, has a peak of 1028 cm^{-1} , while the alkali pretreated bamboo fibre illustrates a peak of 1021 cm^{-1} . The untreated bamboo fibre showed a peak at 2892 cm^{-1} , 1722 cm^{-1} and 3328 cm^{-1} for C-H stretching, C=O stretching and -OH stretching, respectively. The band at 1722 cm^{-1} was attributed to C=O stretching vibration in hemicellulose presenting in bamboo fibres [9]. The alkali pretreated bamboo fibre showed a peak at 2882 cm^{-1} , and 3306 cm^{-1} for C-H stretching and O-H stretching. From the result obtained, there was no hemicellulose in bamboo fibre treated alkali as there was no peak indicating the presence of hemicellulose. The peak of 1028 cm^{-1} and 1021 cm^{-1} indicate the presence of lignin in alkali untreated and pretreated bamboo fibre respectively. The decomposition of lignin in alkali pretreated bamboo shows that the pretreatment of alkali solution was successful. A similar finding was investigated the characteristic bands for a functional group of untreated and alkali-treated highland bamboo fibre, reported that the -OH and C=O stretching for untreated and treated alkali bamboo fibre were at range of $3400\text{-}3425\text{ cm}^{-1}$ and $1617\text{-}1620\text{ cm}^{-1}$ respectively [4]. The removal of hemicellulose and lignin from alkali-treated fibres could explain the decrease in certain vibrational bands of the infrared spectra. The reaction between the hydrolyzed silane and the bamboo powder is confirmed. The peak 995 cm^{-1} indicates the presence of spectra of 3 aminopropyltriethoxysilane. It is also interesting to note that absorption at 894 cm^{-1} corresponding to the -Si-OH bond reveals the presence of residual hydrolyzed silane in bamboo powder. From FTIR spectrum data, we can conclude that a silane coupling agent has been attached into bamboo powder.

3.3 Tensile Test

Tensile test is known as tension test in measuring the maximum strength of material. The specimen geometry follows the ASTM standard of plastic which is ASTM D882. The tensile tests of specimens were conducted at common test speed at a rate of 500 mm/min and 50 mm gauge length in accordance with prior findings. Seven samples with different compositions of bamboo fiber and PLA were used in this testing. Figure 4 indicated the mechanical properties of pure PLA, both untreated and treated bamboo fiber reinforced PLA composite film. Tensile strength can be identified as the maximum stress that a film can withstand while being stretched and pulled before breaking.

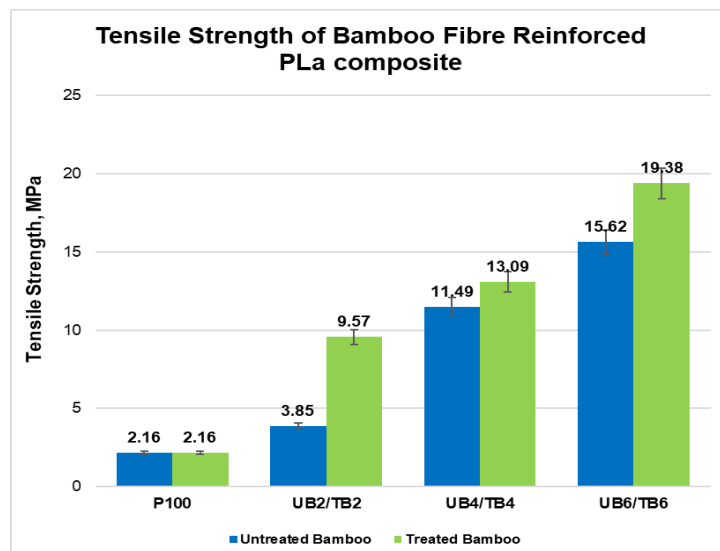


Figure 4: Tensile strength of bamboo fibre reinforced PLA composite with different bamboo fibre content for both untreated and treated bamboo fibre.

The untreated bamboo fibre reinforced PLA has higher tensile strength than pure PLA as the combination of bamboo as a reinforcement in a composite and PLA. The fibre content with 6wt% in UB/PLA has shown great mechanical properties in terms of tensile strength, Youngs’ modulus and

elongation percent with 15.62 MPa, 195.25 MPa and 3.036% respectively. According to the results obtained, addition of different content of treated bamboo also affects the mechanical properties of treated bamboo fibre reinforced PLA composite. With 6 wt% of treated bamboo fibre, the mechanical properties in terms of tensile strength, Youngs' Modulus and elongation percent are higher than pure PLA as the values are 19.38 MPa, 193.8 MPa and 3.863% respectively.

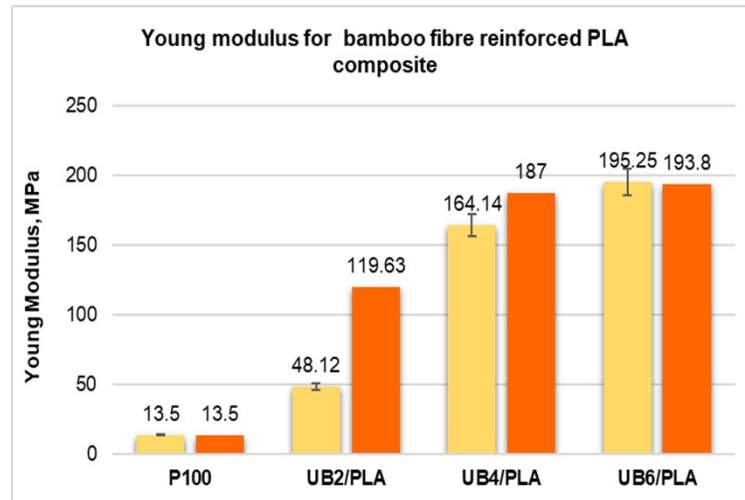


Figure 5: Young modulus of untreated bamboo fibre reinforced PLA composite

From the Figure 5, the incorporation of untreated bamboo fibre increased the Young modulus of PLA composite from 48.12 MPa to 195.25 MPa. The effect of bamboo fibre content resulted in the increase of Young modulus of bamboo fibre reinforced PLA composite for untreated bamboo fibre in which the bamboo fibre content increased from 2 to 6 wt%. Basically, the bamboo fibre content in a composite affects the mechanical properties of untreated and treated bamboo reinforced PLA composite. The relationship between the fibre content and the mechanical properties is directly proportional. When the bamboo fibre content increases, the mechanical properties also increase. Basically, higher fibre content results in better tensile properties in composites, as fibres have higher tensile strength than matrices [6]. The dependence of strength on fibre content was also studied by A similar findings who found that increasing the percentage of bamboo fibre in bamboo fibre reinforced polypropylene (BFRP) only slightly increased the tensile strength of the composites [5]. Furthermore, high fibre content enhanced the tensile strength as more reinforcement could hold the load applied, but poor adhesion between the fibre and matrix reduced the tensile strength. Without proper handling during fabrication, higher fibre content could result in more, and probably larger, void formations in the composites, which then promote micro crack formation under loading and reduce tensile strength [6]. Modulus of composites was enhanced with the addition of bamboo fibre content due to increasing restrictions on the movement of polymer molecules.

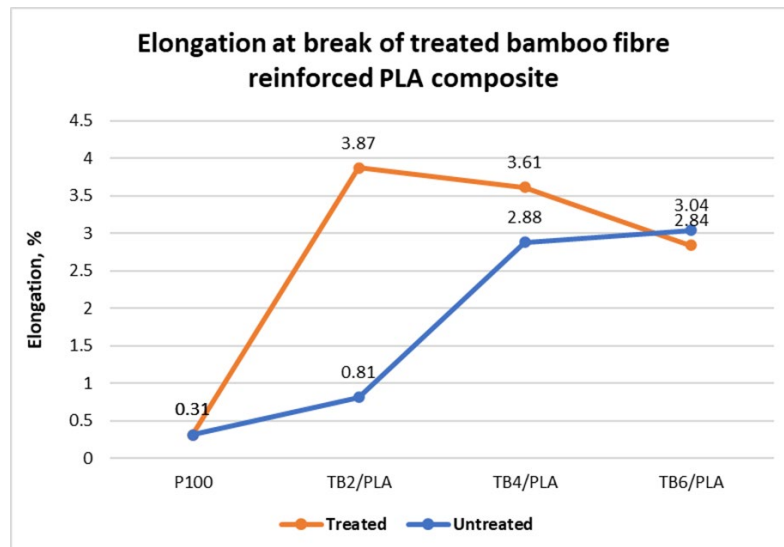


Figure 6: The elongation at break of untreated and treated bamboo fibre reinforced PLA composite

Based on the Figure 6 above, the elongation at break of untreated treated bamboo fibre reinforced PLA composite is increasing as the bamboo fibre content is increasing. For untreated bamboo fibre, the highest elongation at break is UB/PLA at 6 wt% bamboo fibre content and the lowest is UB/PLA at 2 wt% bamboo fibre are 3.04 and 0.81 respectively. The increasing of fibre content resulting in the increasing of elongation at break of untreated bamboo fibre reinforced PLA composite. Meanwhile, for the treated bamboo fibre, it shows the decreasing of elongation at break as the bamboo fibre content is increasing. For 2 wt% of bamboo fibre content in TB/PLA has the highest elongation at break and 6 wt% has the lowest value of elongation at break in TB/PLA which are 3.87 and 2.84 respectively. Overall, the bamboo fibre content in a composite affects the mechanical properties of untreated and treated bamboo reinforced PLA composite. The relationship between the fibre content and the mechanical properties is directly proportional. When the bamboo fibre content increases, the mechanical properties also increase. Basically, higher fibre content results in better tensile properties in composites, as fibres have higher tensile strength than matrices [6]. The dependence of strength on fibre content was also studied by Thwe and Liao [5], who found that increasing the percentage of bamboo fibre in bamboo fibre reinforced polypropylene (BFRP) only slightly increased the tensile strength of the composites.

Furthermore, high fibre content enhanced the tensile strength as more reinforcement could hold the load applied, but poor adhesion between the fibre and matrix reduced the tensile strength. Without proper handling during fabrication, higher fibre content could result in more, and probably larger, void formations in the composites, which then promote micro crack formation under loading and reduce tensile strength [6]. Shah et al., [6] stated that the tensile strength for bamboo fibre composite fabrication with PLA is between 25 to 30 MPa. From the result obtained in the tensile test, the tensile strength of the pure PLA was 2.16 MPa. Emily [7] stated that the value of the tensile strength does not only depend on the size of the test specimen, however, it is dependent on other factors such as the preparation of the specimen, temperature, and the presence of the surface defects. The specimen of pure PLA for tensile test was obtained after the film production via hot press. The surface of the pure PLA specimen as shown in Figure below.

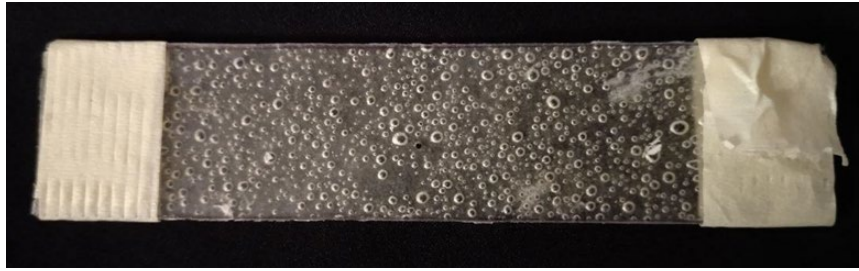


Figure 7: Specimen of pure PLA for tensile test

Based on the Figure 7 above, it is shown that the pure PLA film has bubbles within the specimen. A similar finding regarding moisture content was reported by Titone et al., [8] where it stated that the tensile strength decreases with increasing the content of absorbed moisture in both the pretreatments. The elongation at break is strongly dependent on both the pretreatments, but, in this case, the elongation at break increases with increasing the amount of the absorbed moisture on both the pretreatments. Tensile strength decreased as anticipated with increasing moisture content in the specimen, which was the same as the PLA pellets' drying prior to processing.

It also decreased with increasing moisture absorbed prior to hot pressing into thin film, which was the same as the PLA film pretreatment prior to testing. Therefore, both the conditioning of the pellets prior to processing and the conditioning of the specimens prior to mechanical testing affect the mechanical properties. The hydrolytic breakdown with a decrease in molecular weight is to blame for the impact of moisture content during compression moulding.

4. Conclusion

The results obtained from this study proved that the objectives were successfully achieved. Firstly, the characterisation of bamboo fibre via Particle Size Analyzer (PSA) and Fourier Transform Infrared Spectroscopy (FTIR) analysis were conducted. The bamboo powder was successfully obtained from bamboo fibre via a ball mill. The targeted the size of the bamboo fibre was 75 micron. From the particle analysis, the particle size of the bamboo fibre was found finer than the targeted size. The median particle size of bamboo fibre was 33.4 . Particle size of bamboo powder with size 9.05 was less than 10% and 69.6 was the 90% size proportion of the bamboo powder. From the FTIR results obtained, the alkali and silane treatments were successful as alkali treatment shows the removal of hemicellulose and the decreasing of lignin, also the presence of siloxane bond appear in the FTIR spectra. The preparation of bamboo fibre reinforced PLA composites films with thickness of 1mm were successfully produced via hot press machine. From the result obtained in tensile test, the tensile properties of the bamboo fibre reinforced PLA composites were successfully identified via Universal Testing Machine (UTM) in terms of tensile strength, Young modulus and elongation at break.

Based on the results obtained in this study, several recommendations can be made for further improvement in future studies. The recommendations are as follows:

1. It is recommended that to conduct thermal analysis of the polymer composite for instance Differential Scanning Calorimetric (DSC) analysis and Thermogravimetric Analysis (TGA) between untreated and treated fibre.
2. It is recommende to choose the suitable species of bamboo.
3. It is recommended that to conduct the investigation on the morphology of the polymer composite.

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