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STUDY OF IMMERSION BEHAVIOR AND THERMAL STABILITY OF GREEN COMPOSITE PLA/BAMBOO

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Abstract. One way to address environmental problems caused by the use of synthetic materials is by exploring the potential of new materials that are more environmentally friendly. This research aims to develop PLA/bamboo green composites by investigating the effects of fiber composition and alkali treatment on the thermal stability and immersion behavior of the composites. PLA/bamboo composites were produced using the hot press method with bamboo fibers at 10, 20, and 30 wt.% concentrations. Alkaline treatment was conducted using a 5% NaOH concentration for 24 hours. The tests that have been carried out include TGA analysis, density and immersion test. The PLA/bamboo composite exhibited weight gain due to water absorption during the soaking process. The inclusion of bamboo composition increases the water absorption of the composite, whereas alkali treatment decreases the water absorption of the composite. Furthermore, the introduction of fibers also reduces the degradation temperature of the composite. This research is expected to provide valuable insights for the broader utilization of green composite materials.

Keywords : composite, polylactic acid, bamboo fiber, stability

1. INTRODUCTION

The escalating growth of the global population has led to an increasing demand for food and tableware. Tableware, especially single-use containers that are difficult to recycle, is a major contributor to waste and global warming. Consequently, researchers are increasingly focusing in revealing the environmental effects of various types of materials popularly used as food containers such as aluminum, expanded polystyrene (EPS) and polypropylene (PP) [1]. Due to the huge environmental consequences, an imperative to investigate the creation of eco-friendlier food containers has arisen. The preferred method is to reuse natural materials to replace synthetic materials because they are bio-renewable and environmentally friendly.

Natural fibers, intrinsic components of the human experience, assume a pivotal role. The natural fiber studied in this research is a cellulose-type fiber, specifically bamboo. In terms of its mechanical properties, bamboo exhibits a high level of strength while maintaining a relatively low density of about 640 kg/m^3. In addition, bamboo is one of the fastest-growing renewable plants, which only takes 2-5 years to reach full maturity [2]. One of the drawbacks of natural fibers is that they are susceptible to variations in properties that occur naturally in nature, such as fiber type, fiber length, and fiber chemical composition. This predicament, along with the hydrophilic traits and stability concerns inherent to natural fibers, has spurred investigations into chemical treatments aimed at modifying these attributes.

Mitigation of the limitations inherent to natural fibers finds manifestation in composite materials that amalgamate distinct components to engender novel and enhanced materials. Composites are materials that consist of two components with different properties, so they are combined in such a way as to produce a new and improved material. Currently, the commonly used matrix is a polymer matrix derived from petroleum processing, which is non-renewable and non-biodegradable.

With the various needs of the properties previously described, he realm of composite materials is currently advancing towards the innovation of materials termed "Green Composites". Green composite is a type of material whose constituents can be naturally found and degraded in nature. Consequently, such composites integrate a matrix fabricated from bio-thermoplastics, encompassing polylactic acid (PLA), polyhydroxy butyrate (PHB) and polysaccharides from plants [3]. PLA is a long chain of lactic acid monomers produced from the fermentation process of corn and potato starch [4].

In the present study, PLA is employed as the matrix, reinforced with bamboo fibers, thereby yielding a "Green Composite" material designated as PLA/bamboo. In general, the composition of natural fibers and matrix greatly affects the mechanical properties of the resulting composite [5]-[7]. Research with bamboo micro-fibrils (BMF) shows that tensile strength and modulus of elasticity are influenced by the composition of reinforcing fibers, where the maximum tensile strength is obtained in specimens with 40% BMF [8]. Other research on polymer composites with bamboo reinforcement shows that 30% fiber composition produces optimum composite mechanical properties [9, 10]. Another facet demanding consideration in the utilization of green composites pertains to their thermal stability and behavior upon immersion. Alkali treatment is one of the methods used to modify the properties of cellulose fibers by dissolving hemicellulose compounds and other impurities in the fiber, to improve the bond between the fiber and the matrix in the composite [11]. Therefore, this research was conducted to study the effect of fiber composition and alkali treatment on the stability and immersion behavior of PLA/bamboo composites. Given its propensity for degradation and water absorption, a comprehensive examination of this trait bears profound relevance in propelling the application of green composite materials [12].

2. METHODS

The research methodology in this study unfolds across three pivotal stages: material preparation, composite manufacturing, and testing. Material preparation begins with selecting and cutting bamboo into short fibers with an average length of 1 cm. The bamboo fibers were then soaked in a 5% NaOH solution for 24 hours. Composites were made by mixing bamboo fibers and PLA matrix with variations in fiber composition of 10%, 20%, and 30%. Composite molding was carried out through a hot press process to form test specimens. This method is relatively cheaper and simpler because it does not use sophisticated control equipment. Initial characterization was carried out to see the effect of variations on the density and mechanical properties of the composite. Immersion testing was carried out in a container of water for 1, 2, 3, and 4 weeks. The thermal stability of the composite was analyzed through Thermal Gravimetry Analysis (TGA) testing.

The materials used in this research include betung bamboo (Dendrocalmus asper), PLA, NaOH and distilled water. Betung bamboo fibers with short size are used as reinforcement in PLA matrix. NaOH powder was used to make a 5% NaOH solution by dissolving it in distilled water. Aquadest was also used in the bamboo rinsing process. The research apparatus encompasses an oven and a hot press instrument. The oven is used in the drying process of raw materials before mixing. Hot press is used in the process of molding bamboo/PLA composites to produce test specimens according to the sample mold.

The bamboo fiber employed originates from betung bamboo (Dendrocalmus asper) sourced from Belega Village, Blahbatuh District, Gianyar Regency. The betung bamboo used has an outer diameter of about 20 cm with a thickness of 1-2 cm. The bamboo used was harvested when it was 3-4 years old. The bamboo obtained is then cut using a wood saw to a size of 20 cm. Then the bamboo is split into bamboo slats with a width of 4 cm to facilitate the soaking process using an alkaline solution. After the solution is homogeneous, the bamboo slats are then soaked with a soaking duration of 24 hours. After the soaking process is complete, the bamboo is then rinsed repeatedly using distilled water. Rinsing is done until all NaOH is completely removed from the bamboo. Then the bamboo is dried in the sun for 3 days until the bamboo is dry. The next process is the collection of bamboo fibers from the two bamboo variations. The collection is done by manual mechanical means, namely by beating the bamboo until it is flat, so that the bamboo fiber will separate by itself. The fibers were then cut with scissors into short fibers measuring 1 cm.

In this study, the variations of bamboo fiber used were 10%, 20% and 30% of the weight of the PLA/bamboo composite. Before the bamboo fiber and PLA were weighed, both materials were preheated in an oven using a stainless steel container at 90˚C for 15 minutes. Heating is done to remove moisture content from both materials which can cause pores in the resulting composite. Both raw materials were then weighed with digital scales with an accuracy of 0.1 gram. In one mixing process, the total weight of the resulting composite is 300 grams, so the weight of bamboo fiber and PLA used can be calculated by multiplying the weight percentage by the total weight of 300 grams. The mixture of the two materials was then put into the oven to be heated at 220˚C for 40 minutes. The heating process will cause the fusion of PLA which acts as a matrix with bamboo fiber as reinforcement. The hot pressing process was then carried out to increase the density and reduce the pores of the composite. Pressing is done at a pressure of 10 KPa at a mold temperature of 120˚C for 10 minutes. The finished composite was then cut to the required specimen size for the testing process.

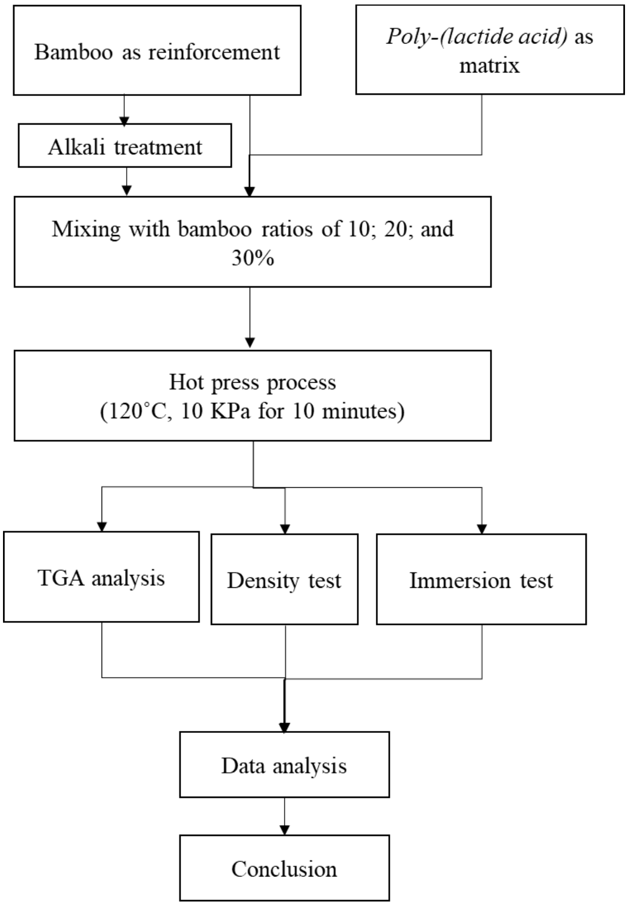

Figure 1. Experimental procedure

The research has been done following Figure 1 and included several characterizations:

a. Density Measurement
One method of measuring density without the need to measure the volume of an object is to compare the weight of an object in air and in a liquid whose density is known, according to the ASTM D792 standard. The weight of the object is measured with an analytical balance with an accuracy of 0.001 g.

b. Immersion Testing
Immersion testing was carried out for 4 weeks in distilled water. The immersion specimens used had an average size of 20x20x5 mm. Before immersion, the composite specimens were dried using an oven at 100˚C for 1 hour. The specimens were then weighed using analytical scales with an accuracy of 0.001 gr.

c. TGA Analysis
Thermal Gravimetry Analysis (TGA) testing is carried out to see the process of changes that occur in the composite when it is heated. TGA testing was conducted at the Material Analysis Laboratory, Department

of Mechanical Engineering, Udayana University. Specimens were prepared weighing approximately 1 gram and then analyzed.

Table 1. Specimen code of PLA/bamboo composite

No	Specimen code	Composition
1	PLA	100% PLA
2	10A	90% PLA + 10% bamboo fiber with alkali treatment
3	20A	80% PLA + 20% bamboo fiber with alkali treatment
4	30A	70% PLA + 30% bamboo fiber with alkali treatment
5	10NA	90% PLA + 10% bamboo fiber without alkali treatment
6	20NA	80% PLA + 20% bamboo fiber without alkali treatment
7	30NA	70% PLA + 30% bamboo fiber without alkali treatment

2. RESULTS AND DISCUSSION

a. Density

Density stands as a pivotal physical parameter influencing the potential applications of materials. Notably, composites, characterized by their light density, emerge as promising candidates for applications prioritizing efficiency, such as transportation. Bamboo fibers are known for their strength and lightweight, so they can be used as an alternative to steel in some applications [13]. Despite its relatively low density of 600-800 kg/m^3, bamboo has a tensile strength of about 200 MPa, with an elongation of 10.2% and a flexural strength of about 230 MPa [14]. The density of the composite material is presented below.

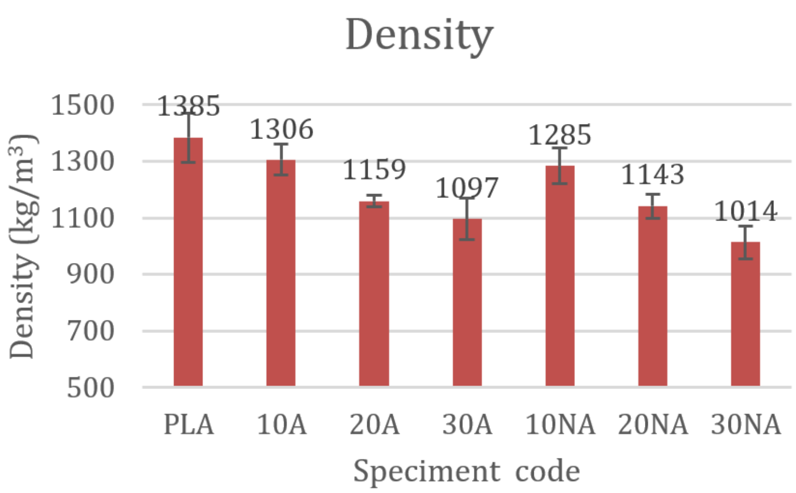

Figure 2. The density of sample composite

Based on the measurements taken, the density of PLA is 1385 kg/m^3. Composite is a type of material with physical properties that are influenced by its constituent materials. Bamboo fiber has a lighter density than PLA, so in theory, mixing PLA with bamboo fiber will produce a material with a lower density. This is consistent with the measurement results, where the higher the fiber composition, the lower the density of the resulting composite. The same trend is obtained for both alkali-treated and untreated fibers. The lower density of alkali-treated specimens is expected because alkali treatment dissolves light components of the fiber such as wax and oil content [15]. The lowest composite density was obtained at 30% fiber composition without alkali treatment, which was 1014 kg/m^3, while the highest density was obtained in the composite with 10% fiber composition with alkali treatment, which was 1306 kg/m^3.

b. Immersion Testing

he escalating interest in PLA arises from its notable biodegradability. One method to explore PLA's biodegradability involves the execution of immersion testing. In this study, the following immersion curve was obtained.

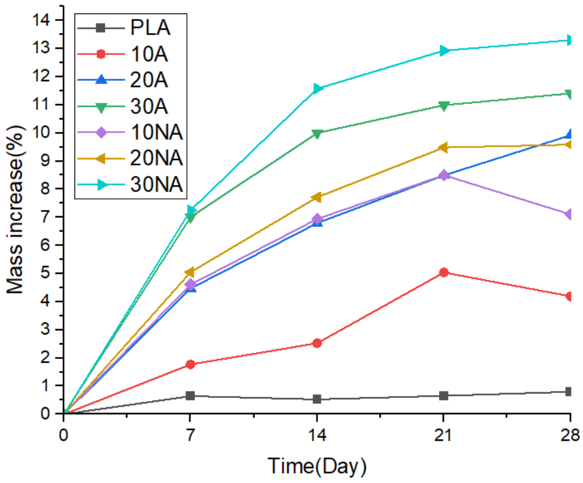

Figure 3. Mass increases of specimens composite

During the immersion test conducted on PLA/bamboo composite specimens, a consistent phenomenon observed across all samples was weight gain. The weight gain exhibited its highest magnitude within the initial 7 days of immersion, after which it gradually subsided over the subsequent weeks, indicative of the composite attaining saturation. Similar results were obtained in another study, where weight loss occurred during the first 7 days of immersion and remained relatively constant until 28 days later [16]. In general, the higher the bamboo fiber composition, the greater the water absorption by the composite. This aligns with the inherent hydrophilicity of natural fibers, rendering them prone to water absorption, a phenomenon evidenced by the observed weight gain. At identical composition, composites with bamboo fibers without alkali treatment experienced a greater increase in weight than those with alkali treatment. These results support previous research where the water absorption content of biocomposites without alkalization is higher than alkalization at 5% NaOH [17]. The largest weight increase occurred in the composite with a composition of 30% bamboo fiber without alkali treatment, which was 13.3%. The composite with the smallest weight increase occurred in the composite with 10% bamboo fiber composition with alkali treatment, which was 4.2%. The pure PLA specimens showed very low water absorption (<1%), so it can be concluded that most of the absorbed water is due to the addition of bamboo fibers. Although PLA is theoretically biodegradable, this immersion test yielded no discernible degradation of the material. This observation implies that the immersion conditions failed to stimulate any degradation process within the material. However, further analysis of the soluble compounds in the immersion solution needs to be done to substantiate this finding. The rate of degradation is influenced by several environmental factors such as water content, type of carbon source, temperature, and pH. The degradation of PLA begins with the hydrolysis of ester groups on its main chain [4].Several factors can cause degradation, such as hydrolysis reaction, thermal activation, photolysis, micro-organism activity and oxidation [18].

c. TGA Analysis

Thermogravimetric Analysis (TGA) stands as a pivotal assessment method for ascertaining the heat stability of materials. TGA curve of the composite specimen is shown as Figure 4. In this study, the composite specimens were heated to temperatures above 900˚C. From several specimens tested, a relatively similar TGA curve was obtained, where there was a decrease in weight up to a temperature of 150˚C, followed by a sloping curve up to temperatures above 250˚C. This weight change occurs due to the evaporation of moisture in the composite specimens. Then there is a drastic decrease until less than 20% of the specimen mass remains at temperatures around 400˚C. In this temperature range, the specimen degradation process occurs. This was attributed to the degradation of the ester linkages involving cyclic oligomers, lactide, and acetaldehyde molecules, along with the emission of gaseous carbon monoxide[19]. The same trend in the composites with fiber addition, which occurs in a single-step process, shows that the degradation of PLA and bamboo fiber occurs simultaneously in the composite.

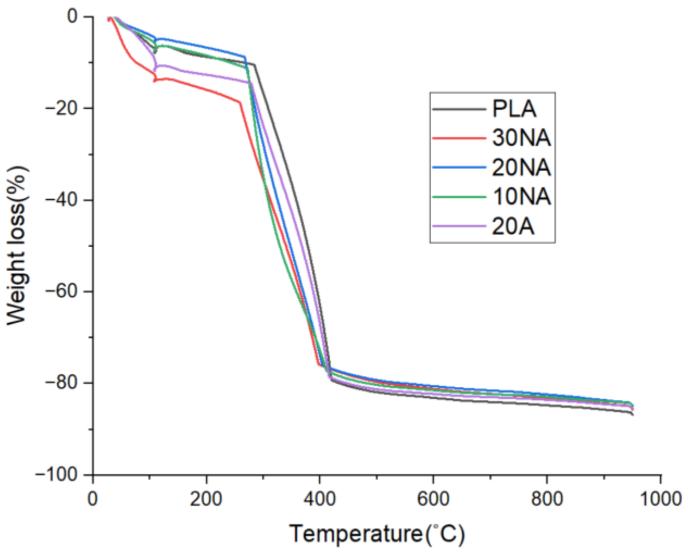

Figure 4. TGA curve of composite specimens

The highest degradation temperature was the pure PLA sample, where the degradation started at 284.1˚C. The addition of bamboo fiber decreased the stability of the composite, where the lowest degradation temperature occurred in the 30% composition of bamboo fiber without alkali treatment, at a temperature of 259.1˚C. This finding contradicts the previous study, which found that the degradation temperature of PLA increased with the addition of coconut fiber [20]. However, other studies show similar results, where the thermal stability of PLA is better than that of its biocomposites [19, 21]. This shows that the thermal stability of PLA biocomposites is influenced by the type of fiber used. In this case, it means that bamboo has a lower thermal stability, causing the PLA/bamboo biocomposite to also have a lower thermal stability. Changes in heat stability are associated with changes in interfacial compatibility and crystal structure of PLA composite [22]. Thus, further explanation of the mechanism of fiber influence on heat stability requires an in-depth analysis of composite crystallinity using X-ray diffraction (XRD).

Table 2. Decomposition temperature of specimens composite

No	Specimen Code	Decomposition temperature (˚C)
1	PLA	284.1
2	20A	278.8
3	10NA	272.2
4	20NA	267.0
5	30NA	259.1

4. CONCLUSION

This research has discussed the development of a bamboo fiber-based "Green Composite" using polylactic acid (PLA) as a matrix. The outcomes of this research underscore that the incorporation of bamboo fiber induces alterations in the properties of composite materials with a PLA matrix. The addition of bamboo fiber reduces the density of the composite because the fiber is lighter. However, the addition of bamboo fibers needs to be controlled due to the hydrophilic nature of the fibers, making the resulting composite more susceptible to changes in properties due to water immersion. The PLA/bamboo composite experienced weight gain due to water absorption in the immersion process. The addition of bamboo composition increases the water absorption of the composite, while the alkali treatment decreases the water absorption of the composite. In addition, the addition of fiber also reduces the degradation temperature of the composite. However, this temperature is still far above the operating temperature of various plastic materials, so this composite still has potential as an alternative material that is more environmentally friendly. Therefore, similar research using natural materials should be further promoted with the help of all stakeholders so that the waste problem can be resolved more quickly.

5. ACKNOWLEDGEMENT
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