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Influence of alkali treatment on thermal stability of polyester composites reinforced with *Bambusa maculat* fibers

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Abstract

The various species of bamboo plants cultivated in Indonesian people's plantations are apus bamboo (*Gigantochloa apus*) (GA), wulung bamboo (*Gigantochloa atroviolacea*) (GV), and tutul bamboo (*Bambusa maculat*) (BM). Apart from being used as a craft material and household utensils, bamboo can be made into natural fiber as a reinforcement for polyester composites. In this research, *Bambusa maculat* fiber was used. In this work, BM fibers were treated with Alkali with NaOH solutions with concentrations of 2, 5, and 8% by weight respectively at room temperature. The next procedure is that the BM fibers are used as composite reinforcing fibers, arranged unidirectional the polyester composites. The manufacturing technique is using hot pressing molding technique. The effect of alkali concentration on the thermal performance of polyester composites was examined using differential scanning calorimetry (DSC) and thermogravimetric (TG) analysis. As the result, the alkaline treatment of BM bamboo fiber with a NaOH concentration of 5%, causes the BM bamboo fiber as reinforcement and matrix to have a tight interface and strong interfacial bonding, allowing the composite to have the best thermal stability among other composites.

Keywords: Polyester composites; *Bambusa maculat* fibers; Thermal stability; Interfacial bonding

1. Introduction

Researchers are now focusing on natural fibers to strengthen polymer matrix composite materials. By considering good mechanical characteristics, low cost, biodegradability, durability, environmental friendliness, sustainability and corrosion resistance [1]. Polyester composites with reinforced natural fiber as a class of environmentally friendly materials possess excellent mechanical properties being in favor of engineering plastics, and have attracted increasing attention in the fields of construction, transportation, aerospace, and new energy vehicles [2]. Bamboo plant is one of the potential plant species as a producer of bamboo fiber which is cultivated in smallholder plantations in Indonesia. Types of bamboo commonly planted include: Bamboo fiber is the main type of reinforcement that can be considered for the synthesis of bamboo laminate composite materials or commonly called bamboo laminate boards.

In the taxonomy, bamboo is included in the grass family (Poaceae, Bambusoideae). Nevertheless, no one would argue that bamboo is one of the most important economic grasses and has a myriad of potential benefits. A total of 1,439 bamboo species throughout the world [3]. Specifically in Indonesia there are around 176 species of bamboo, including 140 native species and 105 endemic species which are only found in Indonesia [3]. Indonesian people have for generations used around 81 species of bamboo for their traditional daily needs, and also for industrial needs [4], especially the species apus bamboo (*Gigantochloa apus*), wulung bamboo (*Gigantochloa atroviolacea*) and spotted bamboo (*Bambusa maculat*).

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Bamboo fiber is a natural fiber that is easy to obtain, has many extraordinary inherent mechanical properties. The tensile strength, flexural strength and fracture statistics of fiber-reinforced composites are superior to other natural fibers, palm fiber [5]. Bamboo fiber is usually taken from bamboo stems after extracting the vascular sheath by several physical methods. As a matrix and adhesive material for making composites, polyester is usually used. Considering its high modulus and high temperature performance, polyester has been widely used in various industrial engineering applications. But polyester also has many disadvantages, such as apparent brittleness due to its interconnected network structure [6]. Therefore polyester composites have not completely replaced conventional composite materials in high load applications.

Natural fiber reinforced polyester composite materials always show weak interfacial compatibility. The explanation is that the hydroxyl groups on the fiber surface absorb water molecules and form hydrogen bonds, preventing close intermolecular contact with the hydrophobic polymer (polyester) matrix [7]. As a result, it is difficult for bamboo fibers to penetrate into the matrix during the composite fabrication process, resulting in non-uniform fiber dispersion in the matrix [8]. The weak interaction between the fibers and the matrix reduces the compatibility of the composite interface, thereby causing less effective load transfer from the matrix to the fibers in the composite material, resulting in negative effects on the mechanical properties of the composite [9]. Increasing interfacial adhesion is an important issue in the development of biocomposites in general.

The study [10] found that the water absorption of bamboo fibers decreased after chemical modification, due to the effective removal of hemicellulose and lignin from the surface of the fibers, indicating that their hydrophilicity was weakened. The effect of alkali treatment on the morphological and thermomechanical properties of bamboo fiber has been carried out by [11]. The conclusion is that the appearance of a fibrillary structure after treatment causes an increase in the available contact area resulting in a stronger bond with the matrix, thereby increasing the interfacial wettability. Additionally, [11] investigated the effect of alkali treatment of bamboo fibers on the properties of PLA-based composites. The results show that the fiber surface treatment provides a stronger mechanical interlocking effect with the matrix, which is beneficial for superior interfacial performance. It could be said that the adhesion between the fiber and the matrix greatly influences the overall properties composite materials [12].

Although many researchers have carried out substantial investigations into the mechanical properties of chemically modified bamboo fiber composites, efforts made on the thermal stability of interfacial bonds and their correlative mechanisms are still very limited. Therefore, evaluating the thermal stability performance of the interface between epoxy resin and bamboo fiber is very important in the design of safe composite structures. In this study, the effect of fiber surface treatment on the thermal stability performance of bamboo fiber composite polyester material reinforced with bamboo fiber type BM was examined through experimental investigations using. Differential scanning calorimetry (DSC), and thermogravimetric (TG) analysis, on single fibers were performed

2. Material and methods

The specifications for the bamboo used as potester composite are: *Gigantochloa apus*, which is locally called Wulung bamboo, the local name for Wulung bamboo *Bambusa maculat*, which is locally called Tutul bamboo. Bamboo plants with an average age of three years from the bamboo plantation of Gangga Village, Gangga District, North Lombok Regency, West Nusa Tenggara Province, Indonesia. Bamboo has an average density of 725 kg/m^3 to 730 kg/m^3. The modulus of rupture and modulus of elasticity of the former are 120 N/mm^2 and 11.239 N/mm^2. The types of bamboo are medium sized with an average stem diameter of 6–8 cm and wall thickness of 10–11 mm, with an estimated height and length of 15–20 m each and the length of this bamboo segment measuring 40 cm, as shown in Figure 1. The thermal stability of fiber, epoxy resin, and composites were tested by TG using a synchronousdifferential thermal analyzer (STA409PC, NETZSCH, Selb, Germany) under nitrogen atmosphere with a flow of 50 mL/min. About 3–5 mg samples were operated in a continuous heating mode from 200 ∘C to 600 ∘C at a heating rate of 10 ∘C/min. The weight loss of various samples at 600 ∘C was determined by % weight loss using the following equation:

$$\text{Weight loss (\%)} = (W1 - W2)/W1 \times 100,$$

where W1 is the sample weight prior to test and W2 is the one at any given temperature.

Figure 1 a. *Bambusa maculat* (BM), b. *Bambusa maculat* (BM) fibers

It should be emphasized that the length of BM bamboo fiber used as reinforcement for polyester composites must be the same length, 150 mm. In addition, the fibers are selected visually to ensure that there are no defects on the surface, as they are vegetable fibers with geometric irregularities. For simplicity, an abbreviation has been created for composite polyester with BM bamboo fiber treated with alkali concentrations of 2%, 5%, and 8%, respectively.

Research specimens, BM bamboo fibers were treated with alkali by soaking them in NaOH solutions with varying concentrations 2, 5, and 8% for 6 hours at room temperature. Next, it is removed from the solution and washed with water, repeated 3-4 times to remove the remaining solution on the fiber surface. The washed fibers were dried in an oven at 80 ∘C for 8 h to reduce the water content and then stored in a desiccator (ZKF040, Shanghai Experimental Instrument Factory Co., Ltd., Shanghai, China) in a sealed polyethylene bag.

Furthermore, composite polyester made with a fiber volume fraction of 30% (Vf = 30%) was produced by hot pressing preforms with a press molding machine (Y/TD71-45A, Tianduan Press Co., Ltd., Tianjin, China). The mold size for the composite plate is 175 × 170 × 2 mm^3, as show in the Figure 2. To make it easier to remove the composite from the mold, the inner surfaces of the top and bottom plates were coated with polystyrene stripper BM bamboo fiber is mixed evenly with polyester in the mold. Hot pressing was carried out on the samples for 3 hours at a temperature of 100 ∘C. After that, the samples were dried at room temperature for 12 hours and stored in a printing machine at 75 ∘C for 6 hours.

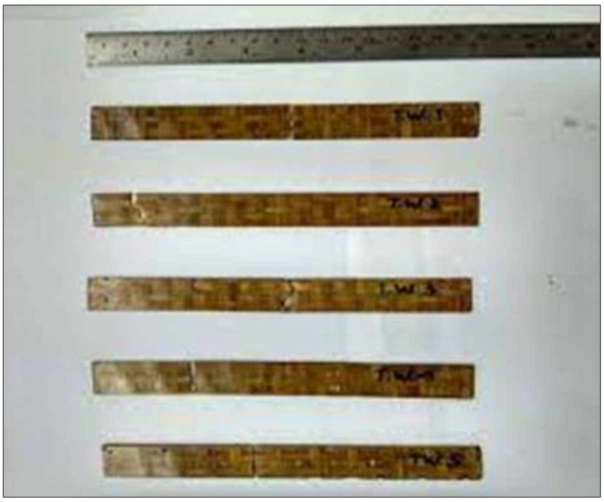

Figure 2 The polyester composites with reinforced *Bambusa maculat* fibers

3. Results and discussion

3.1. Differential scanning calorimeter analysis.

The glass transition behavior which is a characteristic of the thermal stability of the specimen was evaluated with a DSC Q20 analyzer with instrument specifications (TA Instruments, New Castle, DE, USA) in a nitrogen atmosphere (80 mL/min). Masses of about 3–5 mg samples were tested based on the following three steps: heating from 30 ∘C to 200 ∘C and holding for 5 min to eliminate the effects of thermal and mechanical history, then cooling to 30 ∘C and holding for 5 min. In the third (final) step, the sample is heated to 200 ∘C. All the above processes were carried out at a temperature rate of 10 ∘C/min. The results of the test DSC are shown in Figure 3.

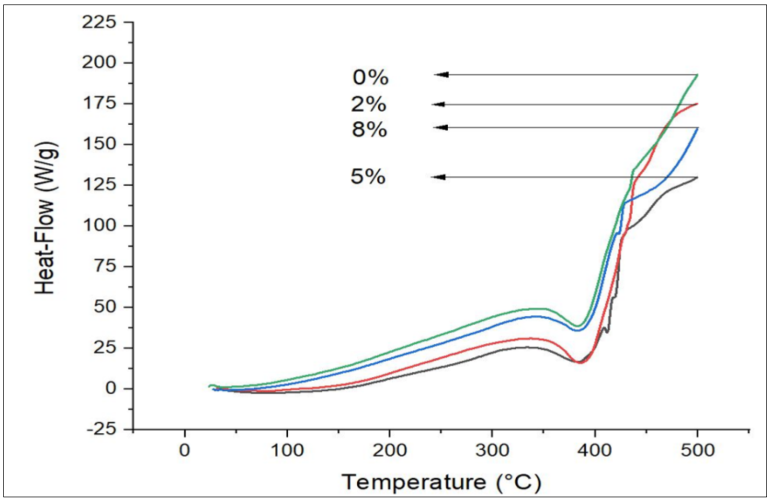

Figure 3 Differential scanning calorimeter analysis of the polyester composites with reinforced *Bambusa maculat* fibers

Figure 3 shows that due to differences in alkali treatment of BM bamboo fiber, not all polyester composites reinforced with BM bamboo fiber have more than 1 peak melting point in the exothermic phase or crystallization point in the endothermic phase. However, all specimens have something in common, namely that they are all monotonous in the exothermic direction because the heat flow in the composite is higher than the heat flow in the reference sample. The peak point of each melting and crystallization point in polyester composite reinforced with BM bamboo fiber with variations in alkali treatment of 0%, 2%, 5% and 8% NaOH concentration is shown in table 1.

Tabel 1 Peak Point of Melting and Crystallization of BM Bamboo Fiber Reinforced Polyester Composite

No	NaOH Concentration (%)	Crystal 1		Melting 1		Crystal 2		Melting 2	
		T (°C)	Heat-Flow (mJ/s)	T (°C)	Heat-Flow (mJ/s)	T (°C)	Heat-Flow (mJ/s)	T (°C)	Heat-Flow (mJ/s)
1	0%	347	63.04	388	39.84	428	133.14	429	131.81
2	2%	354	57.02	386	53.21	-	-	-	-
3	5%	331	30.78	391	15.53	412	43.68	414	42.95
4	8%	346	41.44	383	36.29	412	84.52	413	84.45

In table 1. it can be seen that the treatment of BM bamboo fiber reinforced polyester composite material with alkali treatment using 5% NaOH concentration has the best thermal stability. the first crystallization point has a heat-flow of 30.78 ml/s and the first melting point has a heat flow of 15.53 mJ/s. Furthermore, the composite with the worst thermal stability was obtained by polyester composite reinforced with BM bamboo fiber without alkali treatment (0% NaOH concentration) which had a heat-flow at the first crystallization point of 63.04 mJ/s, and the first melting point had a heat flow of 39.84 ml /s.

3.2. Thermogravimetric analysis polyester composites with reinforced *Bambusa maculat* fibers

The thermal stability of BM bamboo fiber reinforced polyester composite specimens was tested with a TG device using a synchronous differential thermal analyzer (STA409PC, NETZSCH, Selb, Germany) in a nitrogen atmosphere with a flow of 50 mL/min. Approximately 3–5 mg samples were operated in continuous heating mode from 200 ∘C to 600 ∘C at a heating rate of 10 ∘C/min. The weight loss of various samples at a temperature of 600 ∘C is determined by the % weight loss using the following equation:

$$\text{Weight loss (\%)} = (W1 - W2)/W1 \times 100$$

W1 is the initial sample weight (before testing) and W2 is the sample weight after TG testing at a certain temperature. The results of thermogravimetric analysis are shown in Figure 4. TGA or Trigravimetric Analysis testing was carried out to determine the effect of temperature on Weight-Loss or mass reduction of BM bamboo fiber reinforced polyester composites with variations in fiber alkali treatment, soaking with 0%, 2%, 5% and 8% NaOH.

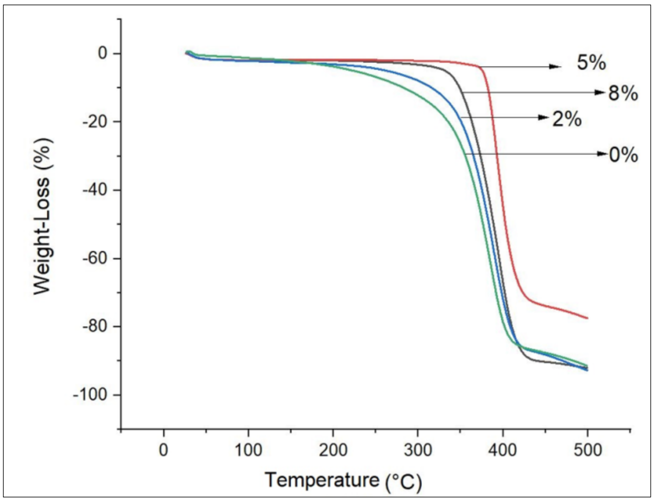

Figure 4 Thermogravimetric analysis of the polyester composites with reinforced *Bambusa maculat* fibers

In Figure 4. it is shown that there is a first stage mass decrease starting from the initial temperature to 200°C which is caused by the evaporation of the water contained in the composite, then the second stage of decomposition or mass reduction occurs at a temperature of 250°C - 400°C, and the final stage of decomposition.The third which still continues above 400°C

Tabel 2 Weight loss of polyester composites with reinforced *Bambusa maculat* fibers

NaOH (%)	**0%**	**2%**	**5%**	**8%**
Stage 1 Temperature (°C)	- 275	- 291	- 371	– 334
Stage 1Weight Loss (%)	9.32	7.19	4.32	5.51
Stage 2 Temperature (°C)	344 - 414	307 - 419	250 - 427	244 – 425
Stage 2 Weight Loss (%)	75.55	78.41	67	82.69
Stage 3 Temperature (°C)	414 - 500	419 - 500	427 - 500	425 – 500
Stage 3 Weight Loss (%)	6.54	7.6	6.13	3.82

Based on Figure 4 and Table 2. it is known that the percentage of NaOH in the alkali treatment influences decomposition or weight loss, where the percentages with the highest to smallest decomposition are obtained from the percentages of 5%, 8%, 2% and 0% NaOH. Alkaline treatment with 5% NaOH experienced water evaporation decomposition the fastest, and 0% NaOH (without treatment) experienced water evaporation decomposition the longest. Therefore, the best percentage of NaOH that has thermal resistance to decomposition is alkali treatment of BM bamboo fiber with 5% NaOH. This is caused by the very strong bond between fiber and polyester. and has a dense matrix interface with reinforcement compared to the bonds and interfaces in other composites. In other words, composites with this percentage have the best thermal resistance.

4. Conclusion

Reinforced polyester composite materials and BM bamboo with alkali treatment with a NaOH concentration of 5% have the best thermal resistance because they experience peak decomposition at the highest temperature among composites with other percentages. On the other hand, reinforced polyester composite materials and BM bamboo without alkali treatment (0% NaOH concentration) had the worst resistance. This is because the alkaline treatment of BM bamboo fiber with a NaOH concentration of 5%, causes the BM bamboo fiber as reinforcement and matrix to have a tight interface and strong bond, allowing the composite to have the best thermal resistance among other composites.

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest to be disclosed.

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