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Effect of Chemical Treatments on Morphological, Physical and Mechanical Properties of Bamboo/ Glass Fibers Hybrid Laminated Composite

Ferriawan Yudhanto[1*], Andika Wisnujati[1], Venditias Yudha[2], Putri Rachmawati[1], Kadek Rihendra Dantes[3]

[1]Department of Mechanical Technology, Vocational Program,
Universitas Muhammadiyah Yogyakarta, Yogyakarta, 55183, INDONESIA

[2]Department of Mechanical Engineering,
Institut Sains & Teknologi Akprind Yogyakarta, Yogyakarta, 55222, INDONESIA

[3]Department of Mechanical Engineering Education, Faculty of Engineering & Vocation,
Universitas Pendidikan Ganesha, Singaraja, Bali, 80223, INDONESIA

*Corresponding Author

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Abstract: Bamboo Apus (*Gigantochloa apus*) is a long wood fiber plant with a short planting period of 4-5 years that can be used as an alternative to reduce synthetic fibers in manufacturing composite products. The bamboo strips used to reinforce was prepared by alkali treatment in 5 wt.% NaOH (sodium hydroxide) at a temperature of 80°C for 2 hours, followed with a delignification (bleaching) of 3 wt.% H_2O_2, at a temperature of 60°C, PH=10 for 1 hour. The manufacturing of laminated composite products was carried out by the vacuum infusion (VI) method with a 0.8 bar pressure with a stacking sequence of 1 ply of the woven bamboo strip and 3 plies of chopped strand mat glass fiber. The tensile test of bamboo/glass fibers hybrid laminated composite treated (HLC-T) was increased by 78%, while the flexural test was increased by 73.6%. It shows that the removal of lignin and hemicellulose in the bamboo fibers due to chemical treatment could improve the compatibility between the fiber and polyester.

Keywords: Hybrid laminated composite, bamboo fibers, chemical treatment, polyester

1. Introduction

The development of composite technology currently used a lot of natural fiber-reinforced composite that is more environmentally and low cost. The natural fiber-reinforced composite also has high strength and elasticity modulus, low density, and lightweight material. Natural fibers as a composite material still have some disadvantages, such as hydrophilic properties and a lot of content impurities on the surface. It causes the voids and weak bonding between the natural fiber and polyester composite. This condition causes the mechanical strength of natural composite products to decreases. The Apus bamboo (*Gigantochloa apus*) is a type of sympodial bamboo (the stems collect in one clump), tight and upright. Rural communities, especially on the islands of Java and Bali, cultivate these plants a lot. The primary purpose of apus bamboo stem used to build construction materials, the material of bridges, household appliances, furniture crafts, roofs, and traditional musical instruments (angklung). The Apus bamboo has a diameter of 30-70 mm with a height that varies between about 4-12 meters. It grows well at an ambient temperature ranging from 15-32°C and rainfall around 600 mm/year in soil conditions with proper drainages, such as on river banks [1]. Base

**Corresponding author: ferriawan@umy.ac.id*

properties of fibers from woody plants such as bamboo are hydrophilic and incompatible with a hydrophobic polyester matrix. These weaknesses could be decreased by soaking fiber in a chemical solution to clean the fiber surface and reduce hemicellulose and lignin content. The chemical treatment also increases the surface roughness of bamboo fibers. Physical and morphological properties compatibility was resulted in the excellent interfacial bonding between the natural fiber and the matrix [2], [3], [4], [5], [6]. Improvement of natural fiber composite is obtained from combined with synthetic fiber such as glass fiber. It combines two or more types, and the structure of laminate fibers is a hybrid laminate composite (HLC).

2. Materials and Method

2.1 Bamboo Fiber

The Apus bamboo material was obtained from Sleman Regency, Yogyakarta, Indonesia. The use of stem Apus bamboo which harvested at age around 4-5 years old. The stem cuts using a knife to a thin strip with a width of 5 mm and 1.5 mm thick. The bamboo strips were then woven by hand to create a woven fabric bamboo strip with an area of 300 x 300 mm (Fig.1).

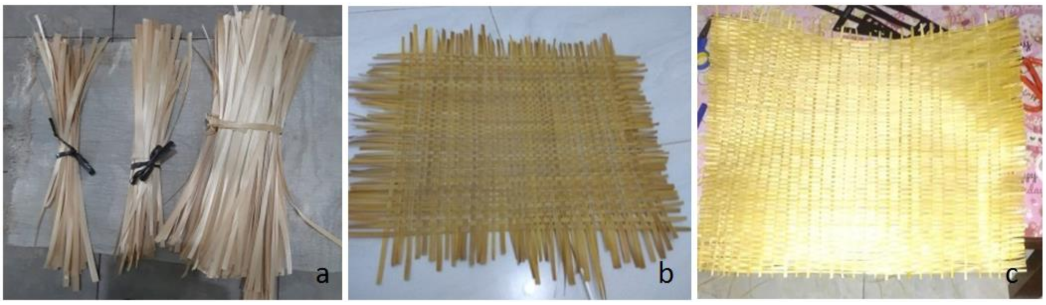


Fig. 1 – Apus Bamboo : (a) raw fibers, (b) before chemical treatment, (c) after chemical treatment

2.2 Materials

Glass fiber chopped strand mat (CSM 200 gr/m^2) used as a layer of another synthetic material. Unsaturated Polyester (UP) used is Yukalac BQTN 157 resin. Hardener uses MEKPO (Methyl Ethyl Ketone Peroxide) type as curing agent. It was obtained from PT. Justus Kimia Raya, Semarang, Indonesia. The chemical solution used for soaking fiber is NaOH (sodium hydroxide) has a purity of 98%, H_2O_2 (hydrogen peroxide) has a purity of 50%. All chemical solutions were obtained from CV. Multi Indo Medika, Yogyakarta, Indonesia.

2.3 Specimen Preparation

The first step of chemical treatment is alkalization which is the soaking of the bamboo strips in 5 wt.% NaOH solution at temperature of 80˚C for 2 hours, and the second step by bleaching process is soaking of the bamboo strips in 3 wt.% H_2O_2 solution at temperature 60ºC, pH=10, for 1 hour [7], [8]. Two step chemical treatment to remove the amorphous region in the surfaces of apus bamboo fiber.

2.4 Vacuum Infusion Method of Hybrid Laminated Composite (HLC)

Fig. 2 shows the schematic of vacuum infusion set-up to produce the hybrid laminated composite (HLC). The T-tube connected the flow tube and spiral tube. The UP enters from the hose in through the flow tube and it is distributed by the spiral tube to flow media into the laminates.

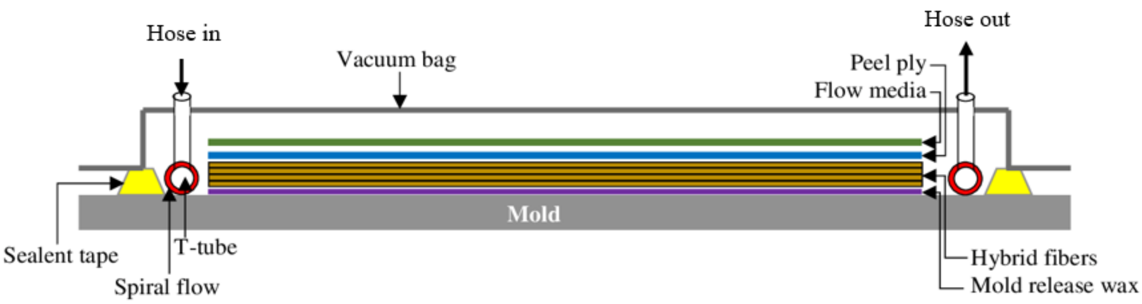
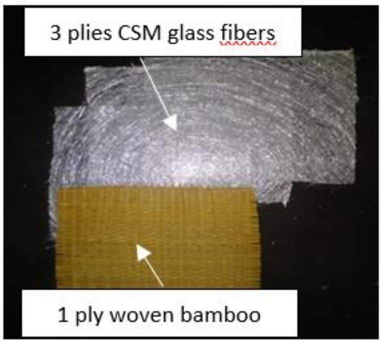
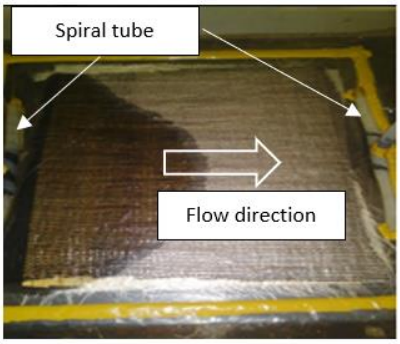


Fig. 2 - Schematic of vacuum infusion set-up

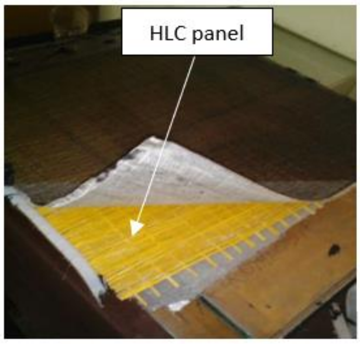
The Fig. 3 shows the laminate stacking sequence of HLC which is consist of 1 ply woven bamboo, 3 plies of chopped strand mat fiberglass (CSM-200) and the flow direction of UPR entering into the hybrid laminate last for 10 minutes. The peel ply material used to separate the HLC and flow media, after curing. Resin trap equipment aims to suck the resin and air buble in the vacuum bag. Pressure gauge in the resin trap aims to control pressure in the vacuum bag and manage of resin flow rate. The pressure gauge in the resin trap set on 0.8 bar and ratio of mix between UPR and hardener is 100 : 1 [3].



Stacking sequence of HLC



The flow direction of UP



Product of HLC panel

Fig. 3 –Vacuum infusion process of hybrid laminate fibers to produce HLC panel

2.5 Physical Test

The density of bamboo strip could be calculated by comparing the mass of a bamboo strip and its volume, using equation 1.

$$\rho = \frac{M}{V} \quad (1)$$

Where, ρ is density (g/cm^3), M is the mass (g) of bamboo strips, and V is the volume (cm^3) of bamboo strips. The weight is calculated using a digital scale with a precision level of 3 decimal. The dimensions of the bamboo strip were calculated using a digital veneer caliper (Mitutoyo). Moisture content calculation aims to determine the percentage of the moisture content contained in the fiber. Count of moisture content refers to ASTM D 629 (Standard Test Methods of Quantitative Analysis of Textiles), which is the ratio of the mass of bamboo strips before and after drying using an oven at 110^{o}C for 1 hour [9]. The difference between the results of the comparison is the amount of moisture vapor contained in the fiber. The count was shown in equation 2.

$$Mc = \frac{W_a - W_b}{W_a} \quad (2)$$

Where, Mc is moisture content (%), Wa is the weight (g) before it was put into the oven, and Wb is the weight (g) after being heated in the oven. The result of density and moisture content test were obtained from 10 test specimens.

2.6 Scanning Electron Microscopy (SEM)

The fiber's morphology during pretreatment could be observed using the SEM model Flex-SEM 1000 II-Hitachi, which was operated in 5 kV conditions. Test specimens coated with Au and using sputtering techniques. This observation was carried out aimed at seeing the surface of the bamboo strip before and after chemical treatment.

2.7 Fourier Transform Infrared (FTIR)

FTIR spectrum as supporting data aims to determine the increase in the amount of cellulose and the loss of hemicellulose and lignin contained in natural fibers by the changes in functional groups. The FTIR model is Shimadzu operate in the wave number range of 500-4000 cm^{-1}.

2.8 Mechanical Test

The specimens for tensile test referred to the American Society for Testing and Materials (ASTM) standard. Tensile and bending test using a universal testing machine (UTM). Fig. 4 shows the dimensions of a test specimen that refer to ASTM D 638 [5], [10]. The width (A) and radius (R) of the grip are 19 mm and 76 mm, respectively. The thickness of the test specimen (t) ranges from 1.85-2.00 mm. The tensile strength, the elongation break, and elasticity modulus of composite material are expressed in equations 3 to 5.

$$\sigma = \frac{F}{A_o} \quad (3)$$

$$\varepsilon = \frac{Li - Lo}{Lo} \quad (4)$$

$$E = \frac{\sigma}{\varepsilon} \quad (5)$$

Where, σ is tensile strength (N/mm^2), F is load (N), A_o is initial area (thickness (t) x width (b)) of specimen test (mm^2), ΔL is increase length after test (mm), and L_o is initial length (mm).

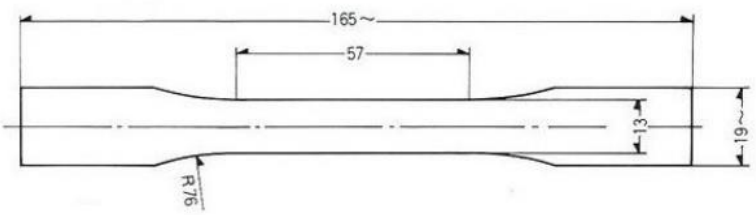


Fig. 4 - Specimen of tensile strength test [10]

Bending testing uses three-point bending, two points as support within 32 mm called the support span length, and compressive load in the middle of the test specimen. The standard refers to the ASTM D790, the span length for material with a thickness of 1.85-2.00 mm is 32 mm [11]. Fig. 5 shows the overall length of the bending test specimen on a hybrid laminate composite is 75 mm. The flexural strength of composite material isexpressed in equation 6.

$$\sigma_f = \frac{3.P.L}{2.b.t^2} \quad (6)$$

Where, σ_f is flexural strength (N/mm^2), P is the load at a given point on the load-deflection (N), L is support span length (32 mm), b is the width of beam tested (15 mm), and t is the thickness of beam tested (mm). The crosshead speed rate for tensile test set on 2 mm/minutes and flexural test set on 1 mm/minutes [5].

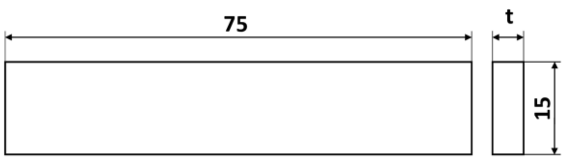


Fig 5 - Specimen of flexural strength test [11]

3. Results and Discussion

3.1 Morphology of Bamboo Strips

Chemical treatment such as alkali and bleaching aims to separate cellulose from hemicellulose and lignin that adhere to the fiber. The previous study by Sugesty et al. 2015 that the chemical composition of raw bamboo fiber such as cellulose, hemicellulose, lignin, and other extractive substances could be seen in Table 1. Hemicellulose content has been obtained from a reduced amount of holocellulose and alfa (α) cellulose (15.67 wt.%). Hemicellulose and lignin in the fibers from stem plants like bamboo have functioned as a constituent of plant walls that are very hydrophilic [13].

Table 1 - Chemical composition of apus bamboo (G*igantochloa apus*) [12]

Cellulose (%)	Holocellulose (%)	Lignin (%)	Pectin (%)	Ash (%)	Extractive (%)
47.56	63.23	22.41	3.38	6.09	4.89

Fig. 6 (a) and (b) shows that the non-cellulosic content, such as hemicellulose and lignin, still covered the cellulose of the bamboo strip. Alkali treatment at high temperatures effectively reduces hemicellulose content in bamboo strips, but it could not be removed the lignin in the fibers because lignin is thermoplastic [14], [15]. Delignification or bleaching process at high temperatures effectively removes the lignin content, could be seen in Fig. 6 (c) and (d). The combines alkali and bleaching process in the fiber effects reduce the hydrophilicity of native fiber and it was improved interface bonding between fiber and matrix [19]. Furthermore, the moisture content and bamboo's density in bamboo strips lessens from 10.7% to 4.2 % (Table 2) and from 1.58 to 1.34 g cm^{-3}, respectively (Table 2), increasing the stiffness of bamboo strip fibers increases.

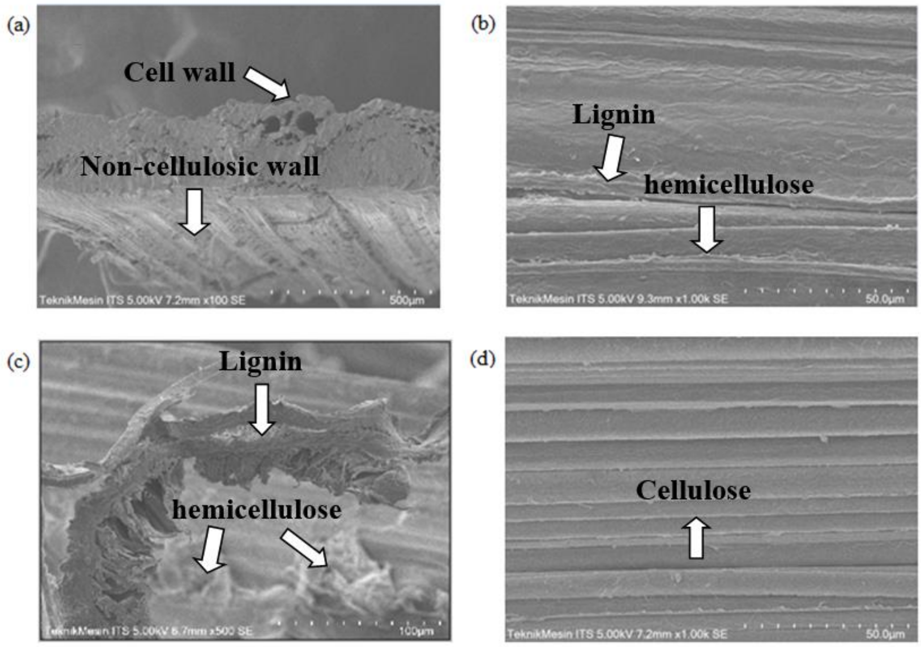


Fig. 6 - SEM image of bamboo strip (a) untreated (front cross section), (b) untreated (side section), (c) removal hemicellulose and lignin, (d) after chemical treated (side section)

Table 2 - Density and moisture content of bamboo strip fiber

Spesimen	Density (g cm^{-3})	Moisture content (%)
Raw material (untreatment)	1.58	10.7
After chemical treatment	1.34	4.2

3.2 FTIR and Chemical Composition Analysis

FTIR spectra graph in Fig.7 shows the functional groups of cellulose, hemicellulose, and lignin. Three dominant wide and high peak shows at a wavenumber of 3443 cm^{-1}, 2900-2924 cm^{-1}, and 1056 cm^{-1} indicates a molecular functional group of OH-stretching, CH-symmetrical stretching, and CO-stretching. These absorbance bands indicate the cellulose group's methyl and glycosidic linkage [20], [21]. The chemical treatment of bamboo fiber increased cellulose (crystalline region) and removed the amorphous region. The absorbance bands at 1730 cm^{-1} (acetate C=O stretching vibration), 1249 cm^{-1} (acetate C-O bending) show that the carboxyl and acetate functional group indicated hemicellulose and lignin, respectively [3], [21], [22], [23]. All two absorbance bands disappearance at FTIR spectra graph of treated bamboo indicates the hemicellulose and lignin removed from the raw bamboo surfaces. The absorbance band at 1635 cm^{-1} show that OH-bending molecular group indicates the absorbed of water [22], [23].

The chemical composition of untreated and treated fibers after chemical treatments shows in Table 3. The chlorite acid modification is based on SNI (Indonesian Standard National) 0492:2008 for calculating the chemical composition such as cellulose, hemicellulose, and lignin. The alkalization decreases the hemicellulose content (22.7% to 19.8%) more significantly than lignin content (28.4% to 27.1%). The alkali-treated, followed by bleaching treatments on the temperature 60ºC, effectively decreases lignin content significantly (28.4% to 14.2%). On the other hand, it causes increases in the cellulose content (42.6% to 68.7%). Therefore, the combined alkali-bleaching is very more effectively reduce the non-cellulosic material than only use alkalization treatment.

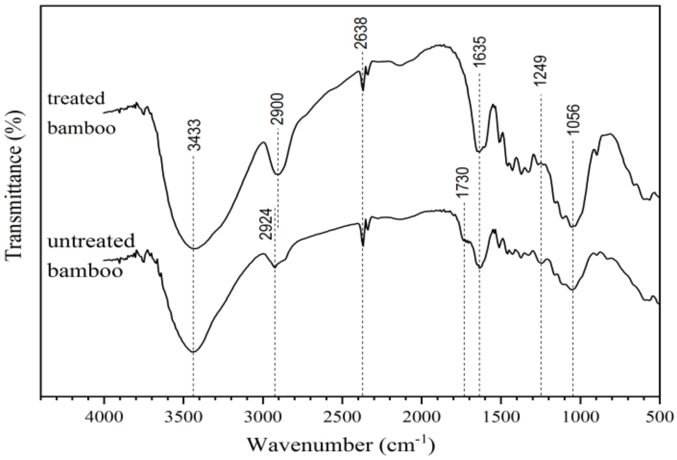


Fig. 7 – FTIR spectra of untreated and treated bamboo fibers

Table 3 - Chemical composition of apus bamboo fiber (Yogyakarta, Indonesia)

Materials	**Cellulose (%)**	**Hemicellullose (%)**	**Lignin (%)**	**Extractive (%)**
Raw (untreated)	42.6	22.7	28.4	1.4
Alkali treated	52.3	19.8	27.1	0.8
Alkali-Bleaching treated	68.7	16.6	14.2	0.5

3.3 Tensile and Flexural Strength

The hybrid laminated composite product was cut and formed according to ASTM D 638 and D 790 standards using laser cutting. It has an average thickness of 1.85-2.00 mm. The minimal number of test specimens prepared following the ASTM standard reference is 5 test specimens (Fig. 8 and 9).

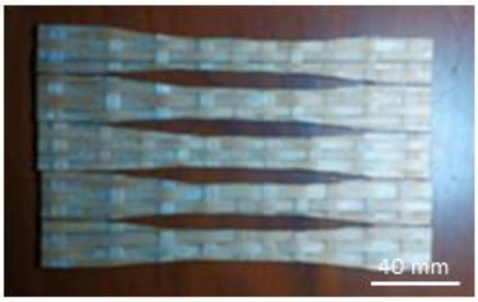


Fig. 8 - Tensile specimens test

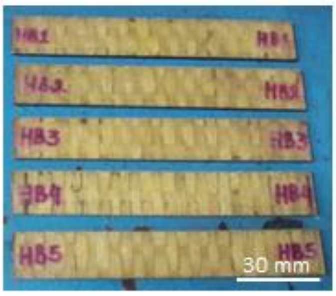


Fig. 9 - Flexural specimens test

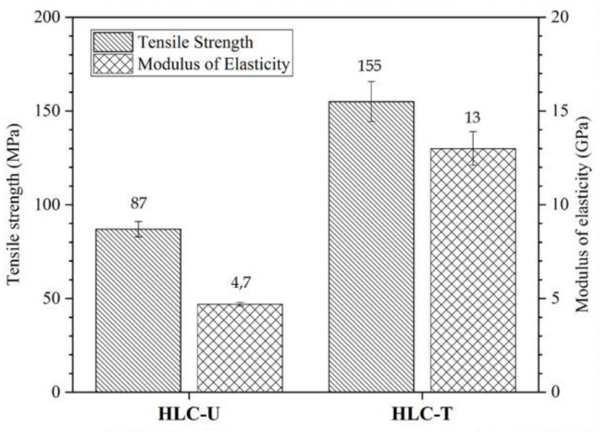


Fig. 10 - Tensile strength and modulus of elasticity

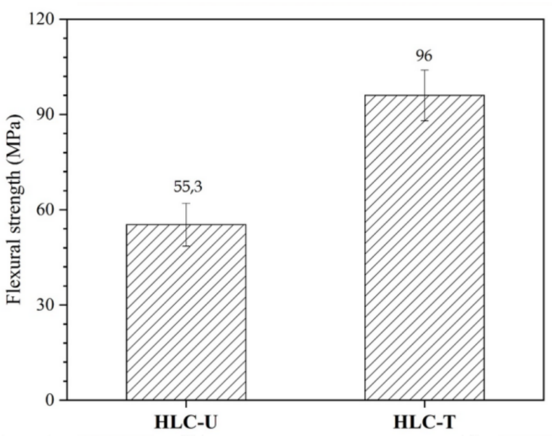


Fig. 11 - Flexural strength

Fig. 10 shows that the tensile strength and modulus of elasticity of HLC-T (hybrid laminated composite bamboo treated) is raise 78% and 177%, respectively, higher than the HLC-U (hybrid laminated composite bamboo untreated). Fig. 11 shows the flexural strength of the HLC-T composite 73.6% higher than HLC-U because alkali-bleaching can reduce the hydrophilic properties and increases the surface roughness. The previous research of laminated bamboo composites carried out by Ali *et al.* has been used the buluh semantan bamboo (*Gigantochloa scortechinii*) with a thickness of 3 mm. It resulted in the best tensile strength of 30 MPa [16]. Another research of HLC using other natural fibers such as *Agave sisalana* and jute combined with glass fiber is a woven mat (600 gsm) conducted by Ramesh *et al.* [17]. The polymer used is epoxy resin by the press mold method. The composite stacking sequence consists of two plies of natural fibers and three plies of synthetic fibers. The tensile strength of sisal/GFRP is 68.5 MPa, and jute/GFRP is 62.9 MPa lower than bamboo/glass hybrid composites having the highest tensile strength of 155 MPa.

3.4 Fracture of Speciment Hybrid Laminate Composite

Fig. 12 shows a bamboo strip fracture of HLC-U, which shows a poor adhesion bonding between the matrix and bamboo fiber. It led to the delamination of interfacial skin and fiber pull out after the tensile test [18]. It is typical of bamboo strip strips failure strain greater than matrix failure strain. Fig. 13 shows bamboo strip fracture of HLC-T, which shows it perfectly binds with the polyester-matrix and only causes a minor multiple crack fracture. It is a typical of matrix and fibers could resist failure strain together. Fig. 14 shows the SEM photo of the flexural fracture of the HLC-U. The fracture in the bending test is almost the same as the tensile test. The fracture that bamboo strips without chemical treatment often found the presence of delamination interfacial skin, bamboo fiber pull out, and multipe crack. Fig. 15 shows the photo SEM of flexural fracture of HLC-T. It shows ductile fracture and does not show any delamination between layers and interfacial skins. It is caused by the lowest moisture content and high cellulose content, as shown in Table 2 and 3.

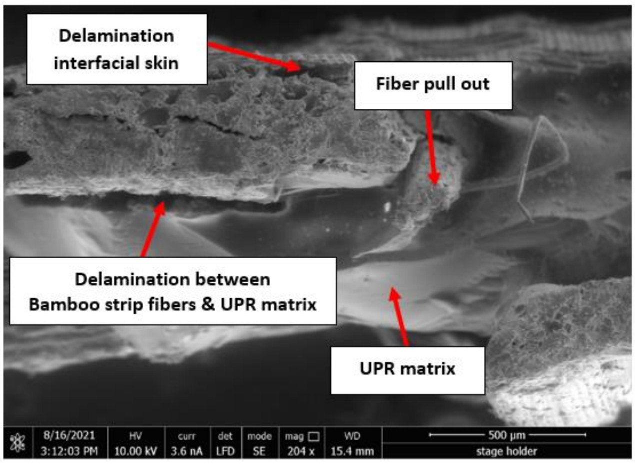


Fig. 12 – SEM Photo of tensile fracture on HLC-U

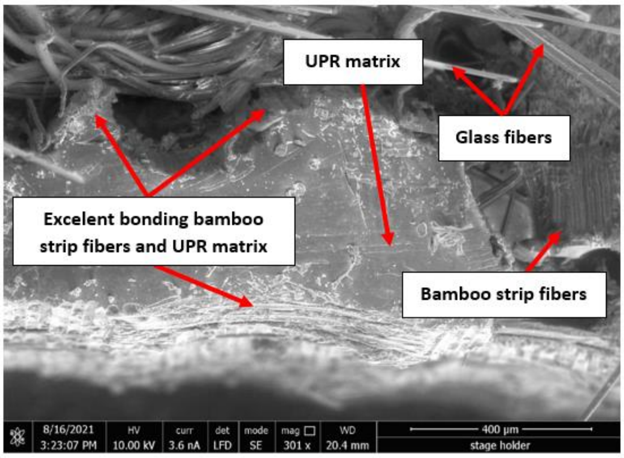


Fig. 13 – SEM Photo of tensile fracture on HLC-T

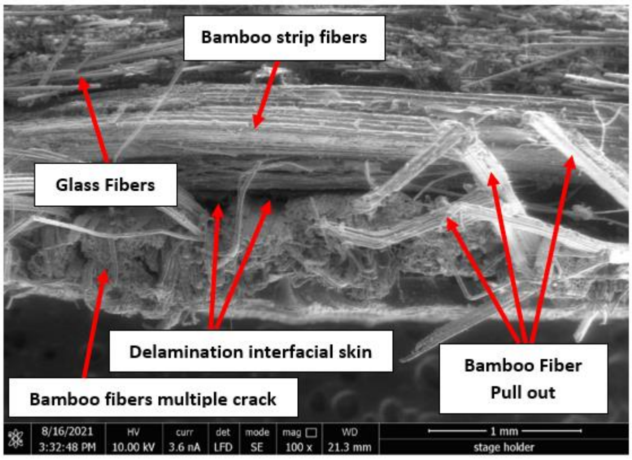


Fig. 14 – SEM Photo of flexural fracture on HLC-U

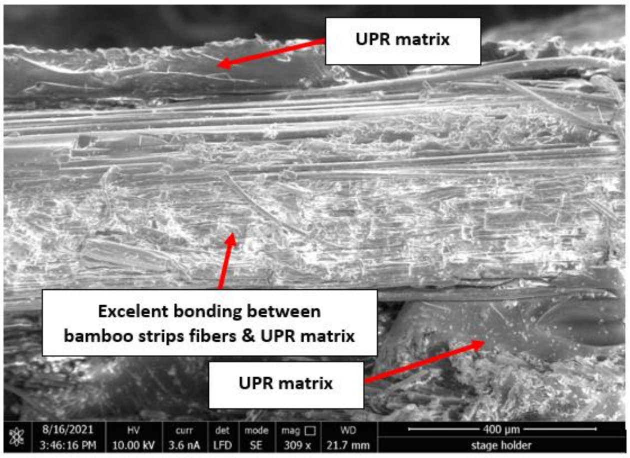


Fig. 15 – SEM Photo of flexural fracture on HLC-T

4. Conclusion

The combination of chemical treatment, i.e., alkali and bleaching process, successfully removes amorphous content (hemicellulose and lignin) on the fiber, which could be shown on the SEM image and FTIR spectra graph. The removal of amorphous impurities makes the surface roughness of the fiber better and lower the moisture content. It could increase the tensile strength, tensile modulus of elasticity, and flexural strength of bamboo strips as reinforcement on hybrid laminate composite by 78%, 177%, and 73.6%, respectively.

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