

## Effects of Bleaching and Heat Treatments on *Indosasa angustata* Bamboo in Vietnam

Quang Trung Nguyen,\* Thiiphuong Nguyen, and Ngoc Nguyen

Effects of bleaching and heat treatment were investigated for *Indosasa angustata* bamboo grown in Vietnam. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) at a concentration of 30% was used for the bleaching process. For the heat treatment, the bamboo strips were heat-treated at two different temperatures (120 °C and 140 °C) for 3 h. There was a slight increase in the lignin and hemicellulose contents of the samples treated at 100 °C for 1 h when H<sub>2</sub>O<sub>2</sub> was used. The water absorption rate did not change much, and the treated bamboo strips became brighter after undergoing bleaching. For the heat-treated samples, the lignin content slightly increased after the samples were heated at both 120 °C and 140 °C for 3 h. Moreover, the higher heating temperature led to a lower hemicellulose content, a darker colored bamboo sample, and a lower water absorption rate for the treated bamboo strips.

*Keywords:* Heat treatment; Bleaching; *Indosasa angustata*; Color difference; Water absorption

*Contact information:* Research Institute of Forest Industry, Vietnamese Academy of Forest Sciences, 46 Duc Thang Street, Bac Tu Liem District, Hanoi, Vietnam;

\* Corresponding author: [nqtrung-icd@vafs.gov.vn](mailto:nqtrung-icd@vafs.gov.vn)

### INTRODUCTION

Vietnam has a tropical monsoon climate with high temperatures and high humidity, which is advantageous for the growth of bamboo and the development of the bamboo industry. Currently, the area of the bamboo forests in Vietnam is approximately 1.4 million hectares. Bamboo is widely distributed throughout Vietnam. A mature Vau Dang (*Indosasa angustata*) bamboo plant can reach nearly 20 m in height with an average internode length of 30 cm to 50 cm. The internode can grow up to 80 cm in length. The *I. angustata* has a large wall thickness (approximately 12 mm) (Nguyen 2009). The “Science and Technology for the Sustainable Development of the Northwest Region” project was established to study the application of bamboo and its economic development in the Northwest region of Vietnam. Indigenous bamboos were the focus of this study, and *I. angustata* was one of the priority bamboo species used for the project. Currently, *I. angustata* is only used for making handcrafted products; thus it is worth investigating its other uses to better utilize this resource.

For bamboo to be as a durable product, its susceptibility to attack by insects and fungi must be addressed. While the structure of bamboo gives its unique quality, it also indicates that bamboo will be difficult to treat with suitable substances. Many studies have been conducted on bamboo preservation methods. There are management strategies that involve cutting bamboo at an appropriate age and time of the year, which can reduce its appeal to insects but not entirely prevent attack. There are traditional treatment methods, such as soaking in water to leach out the nutrients, lime-washing, and baking. However,

there is an ongoing need for bamboo treatment methods that are environmentally friendly and effective.

Prior to the manufacturing process, bamboo is usually treated with bleach and heat to enhance the appearance and natural durability of the products (Sharma *et al.* 2018). The use of bleaching and heat treatment methods under certain conditions do not affect the physical properties of bamboo, but they can increase the bamboo's resistance to mold (Zhang *et al.* 2018). Bleaching methods increase the brightness of bamboo and prevent mold during use (Ma *et al.* 2001; Ashaari *et al.* 2004; Chai and Li 2014). Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is commonly used in the bleaching process because it is an effective bleaching agent (Ma *et al.* 2001; Chai and Li 2014).

Bamboo heat treatment methods have been studied and applied throughout the world. The main components of bamboo cell walls are hemicellulose, cellulose, and lignin, which are also the main components found in wood. These components change after heat treatment (Wang *et al.* 2001). A higher heating temperature and a longer heating time will create a darker, more stable bamboo (Luo *et al.* 2009; Li *et al.* 2010). According to the standard LY/T 1815 (2009) for non-structural glued laminated bamboo, colors can be divided into 3 product categories: natural color glued laminated bamboo, bleached glued laminated bamboo, and carbonized glued laminated bamboo. Different conditions of treatment were conducted by Bui *et al.* 2017, such as heat treatment at 100 °C or 180 °C; with flax oil, sunflower oil, or without oil; treatment durations of 1 h, 2 h, or 3 h. The results showed that the treatment methods could increase both the durability and the compressive strength of heat treated samples, compared to untreated bamboo.

The aim of this study was to investigate the effects of the bleaching and heat treatment processes on the *I. angustata* species used to produce laminated bamboo. The effect of the different methods on bamboo's main components, its water absorption rate, and color were investigated. In addition, scanning electron microscopy (SEM) was used to analyze the transformation of the bamboo structure.

## EXPERIMENTAL

### Materials

#### *Sample preparation*

The *I. angustata* bamboos selected were between 4 and 5 years old. The bamboo was grown in the Phu Tho province of Vietnam. The 1.3-m-long bamboo culms were cut and then split into strips. The bamboo strips had dimensions of 400 mm × 25 mm × 5 mm (length × width × height) and were prepared at a moisture content of 6% (MELB UNI 2074-4; Shepherd Systems P/L, Victoria, Australia). The number of the samples used for each experiment was 12.

### Bleaching Process

#### *Solution preparation*

A 30% concentration solution of H<sub>2</sub>O<sub>2</sub> (Hebei Guanlang Biotechnology Co., Ltd., Hebei, China) was prepared in the laboratory at the ambient temperature of 20 °C ± 3 °C.

#### *Bleaching method*

The soaking (dipping) method was used to conduct the experiments. All the experiments were conducted at a controlled temperature of 23 °C in the laboratory. The

experimental framework of the bleaching process is shown in Fig. 1. The bamboo strips were submerged in the 30% H<sub>2</sub>O<sub>2</sub> solution at 100 °C for 1 h. The samples were then washed using distilled water and dried to a moisture content of 6%.

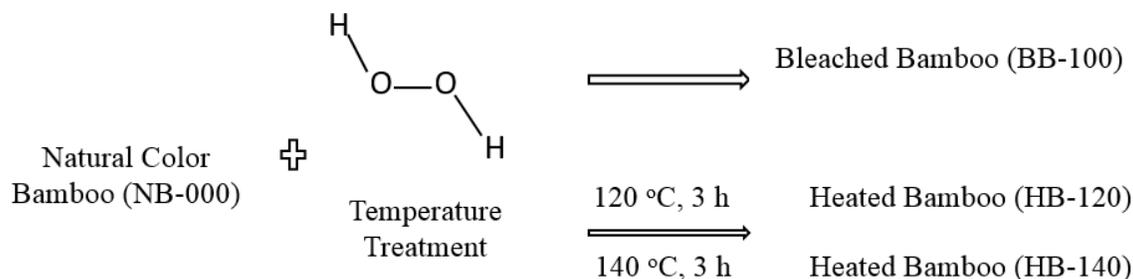


Fig. 1. Experimental framework for the bleaching and heat treatment process

### Heat Treatment Process

The bamboo strips were heat-treated (RYQ09-2; Sumpot, Shandong, China) for 3 h at temperatures of 120 °C and 140 °C. The experimental framework of the heat treatment process is shown in Fig. 1. After the treatment, the samples were washed and dried to a moisture content of 6%.

### Methods

#### *Determination of the basic components of the treated bamboo*

To determine the basic components of the bamboo, the standard TAPPI T221 om-93 (1993) was used to determine the ash content, TAPPI T207 cm-97 (1999) was used for the alcohol extraction determination, TAPPI T207 cm-99 (1999) was used to determine the solubility of the bamboo in cold and hot water, and TAPPI T222 om-98 (1998) was used to determine the lignin, cellulose, and hemicellulose contents, as well as for the pentosane determination.

#### *Measurement of the water absorption*

The bleached and heat-treated bamboo samples were soaked in water. The water uptake of each sample was measured by submerging the sample in water for different times (2 h, 8 h, 24 h, 48 h, 96 h, and 168 h). The samples were removed from the water at their respective times and were placed on a balance (BP 410S; Sartorius, Gottingen, Germany). For example, a sample was submerged in water, and after 1 h, it was removed and weighed. Additionally, after 8 h, 24 h, 48 h, 96 h, and 168 h, the sample was removed and weighed. The water uptake was measured against the submersion time because different periods of time indicated different water absorption rates for the samples.

#### *Measurement of color differences*

The color measurements were recorded using a digital color apparatus (BYK-Gardner, Columbia, MD, USA) at an ambient temperature of 20 °C ± 3 °C. The results of the color measurements were presented in the CIELAB color system.

The  $L^*$ ,  $a^*$ , and  $b^*$  color coordinates were calculated based on the CIE illuminant D65 (representing Northern daylight at noon and the international standard for daylight 160 exposure) and an observation angle of 10° (Hunter and Harold 1987), which represents

average midday light in the northern hemisphere, as defined by the International Commission on Illumination (CIE). An observation angle of 10° was used.

The color measurements were performed on all the samples prior to the bleaching and heat treatment processes. After the treatments, the bamboo strips were dried to 12% moisture content in a drying kiln and measured to compare with the previous measurements of the untreated samples.

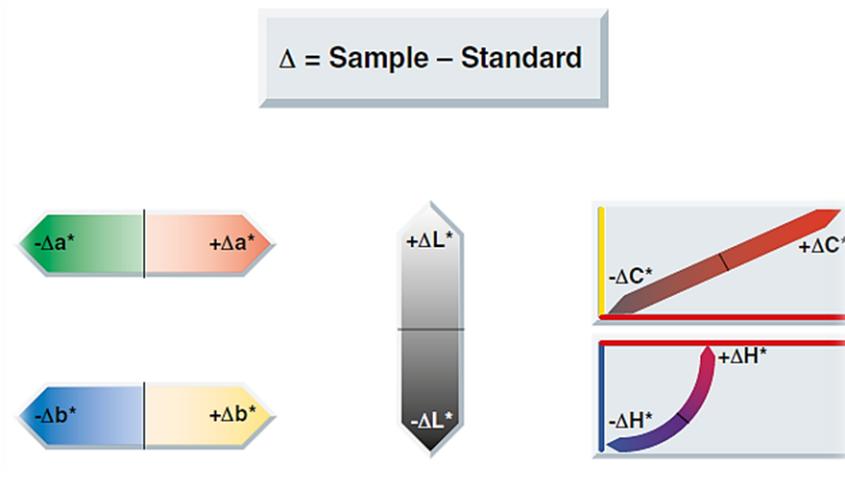
The color of every sample was scanned at four spots, and the result was automatically calculated using the BYK-Gardner digital color apparatus as the average value of the four scans. The same spots were used for measuring the color of the samples before and after the bleaching and heat treatment processes.

The difference in the bamboo color and their locations on the samples were determined using the color coordinates  $L^*$ ,  $a^*$ , and  $b^*$ . The  $L^*$  axis specifies the lightness, and it runs from top to bottom in the range from black (0) to white (100). The  $a^*$  coordinate represents green at the negative ( $-a^*$ ) values and red at the positive ( $+a^*$ ) values. The  $b^*$  axis is blue and yellow, where the negative ( $-b^*$ ) values are blue and the positive ( $+b^*$ ) values are yellow. Both the  $a^*$  and  $b^*$  values are positive/negative co-ordinates that define the hue and intensity of the color. The total change of the color ( $\Delta E^*$ ) is commonly used to represent a color difference, as shown in the following Eq. 1,

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (1)$$

where  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  are the changes in the samples'  $L^*$ ,  $a^*$ , and  $b^*$  values, respectively, before and after the dyeing of the samples.

To determine the actual change in the color, the individual colorimetric components  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  or  $\Delta L^*$ ,  $\Delta C^*$ , and  $\Delta H^*$  were used. The calculation and interpretation of the differences are shown in Fig. 2.



**Fig. 2.** The actual calculation and interpretation of the color differences

A low  $\Delta E_{L^*a^*b^*}$  corresponds to a low color change or a stable color. According to Lovrić *et al.* (2014), the color differences of the treated veneers can be classified by  $\Delta E$  (Table 1). This classification was applied for the assessment of the color changes of the treated bamboo strips.

**Table 1.** Classification of the Color Difference by  $\Delta E$ 

No.	Color Change ( $\Delta E$ )	Description
1	$\Delta E < 0.2$	No noticeable difference
2	$0.2 \leq \Delta E < 2$	Small difference
3	$2 \leq \Delta E < 3$	Color differences noticeable at high quality screen
4	$3 \leq \Delta E < 6$	Color differences noticeable at middle quality screen
5	$6 \leq \Delta E < 12$	Great difference
6	$\Delta E \geq 12$	Different colors

*Delamination test of non-structural glued laminated bamboo*

A pressing machine (LP-S-80; Labtech Engineering Co., Samutprakarn, Thailand) was used to manufacture the laminated bamboo products. The schedule of the process was as follows: a hot-pressing time of 1.1 min/mm, a pressure of 1.0 MPa, a temperature of 110 °C, and a melamine urea formaldehyde (MUF) adhesive loading level of 220 g/m<sup>2</sup> (using double glue lines).

The Chinese standard LY/T 1815 (2009) was used to determine the values of the peel strength test. Samples with dimensions of 75 mm × 75 mm were prepared. The samples were submerged in 60 °C water for 3 h, and then dried at 60 °C for 10 h. The quality of the samples met the standard when the total crack length on the adhesive film was less than 25 mm.

**Statistical Analysis**

An analysis of variance was conducted on the water absorption and the color differences with the factors as explanatory variables. Main effects and two-factor interactions were included in the model. The assumption of constant variance was checked with a plot of residual values vs. fitted values. Main effects were considered to be significant if the P-value was less than 0.05 and two-factor interactions were reported for  $P < 0.01$  or close to it. Least Significant Difference (LSD) values at  $P = 0.05$  were used to estimate the variability between the means of the samples for each combination of factors. The software GENSTAT (16th Edition, VSN International Ltd, Hemel Hempstead, UK) was used for the statistical evaluation of the data.

**RESULTS AND DISCUSSION****Basic Components of the Treated Bamboo**

The effects of the bleaching and heat treatments on the basic components of the *I. angustata* are presented in Table 2.

There were insignificant changes to the lignin, cellulose, and hemicellulose components of the bamboo after the bleaching and heat treatment ( $P = 0.95$ ). After bleaching, the lignin content slightly decreased from 21.9% to 19.6%, while the hemicellulose content increased 2.2%. This was due to the hemicellulose hydrolysis and the presence of the H<sub>2</sub>O<sub>2</sub> oxidizing agent. Moreover, only a small fraction of the lignin dissolved, and the chemical components slightly changed. Some of the hemicellulose dissolved into the bleaching solution, but most of it was retained. Therefore, the hemicellulose content did not change much.

For the heat treatment method, the samples were treated at 120 °C and 140 °C for 3 h. The hemicellulose from the HB-120 and HB-140 samples decomposed greatly.

However, the lignin did not decompose, which proved that the lignin from the bamboo was stable at high temperatures. This decomposition reduced the hemicellulose content of the HB-120 and HB-140 samples.

**Table 2.** Ratio of the Components in *I. angustata* Bamboo at Different Treatment Conditions

Components	NB-000	BB-100	HB-120	HB-140
Ash content	0.66	0.45	0.49	0.45
Alcohol extraction	6.34	6.44	6.91	10.77
Soluble in cold water	5.73	13.07	7.37	13.16
Soluble in hot water	8.78	18.55	17.72	15.64
Cellulose	43.99	40.41	44.35	49.54
Pentosane	16.24	16.43	17.83	14.70
Lignin	21.93	19.55	22.62	22.65
Soluble in sodium hydroxide (NaOH) (1%)	27.46	41.96	42.49	44.19
Holocellulose	71.02	73.17	69.4	66.57

Notes: NB-000: Natural color bamboo; BB-100: Bamboo bleached at 100 °C; HB-120: Bamboo heated at 120 °C; HB-140: Bamboo heated at 140 °C.

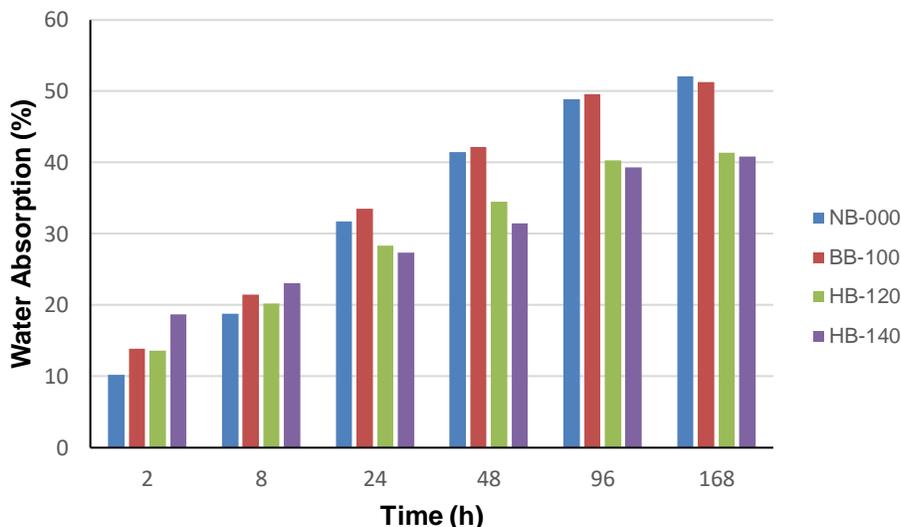
The lignin was very sensitive to the oxidizing agents when H<sub>2</sub>O<sub>2</sub> was used to treat the BB-100 sample. Meanwhile, the acidic environment remarkably decreased the lignin content. This process was involved in the decomposition of the less stable ether bonds in the lignin and the dissolution of the small particles. In contrast, the high-temperature and prolonged heat treatment led to a noticeable reduction in the hemicellulose content (mostly xylan), which resulted in a reduced holocellulose content.

For the BB-100 sample, the H<sub>2</sub>O<sub>2</sub> treatment agent removed the lignin and reduced the cellulose content in the amorphous area of the plant cell wall to 40.4%. The HB-120 and HB-140 samples did not decompose the cellulose, but the treatment for these samples resulted in the separation of the less stable and easily hydrolyzed hemicellulose, which increased the proportional cellulose content of the sample.

The extractive contents of the original bamboo samples in the cold and hot water were 6% and 9%, respectively. After the heat treatment and the bleaching treatment, the extractives content nearly doubled. The biodegradable substances of the bamboo samples were soluble in 1 wt% NaOH solution. The biodegradable substance content of the bamboo samples was initially 27.5% and their contents reached over 41% after different treatment methods. Therefore, the heat treatment and bleaching methods softened and loosened the structure and components of the bamboo, resulting in the easy dissolution of these parts in the 1 wt% NaOH solution.

### Effect of the Bleaching and Heat Treatment on the Water Absorption

The effects of the bleaching and heat treatment on the water absorption of the samples are shown in Fig. 3. In general, the water absorption that achieved significant changes ( $P < 0.01$ ) in all the samples at the different periods of time.



**Fig. 3.** The water absorption after the bleaching and heat treatment

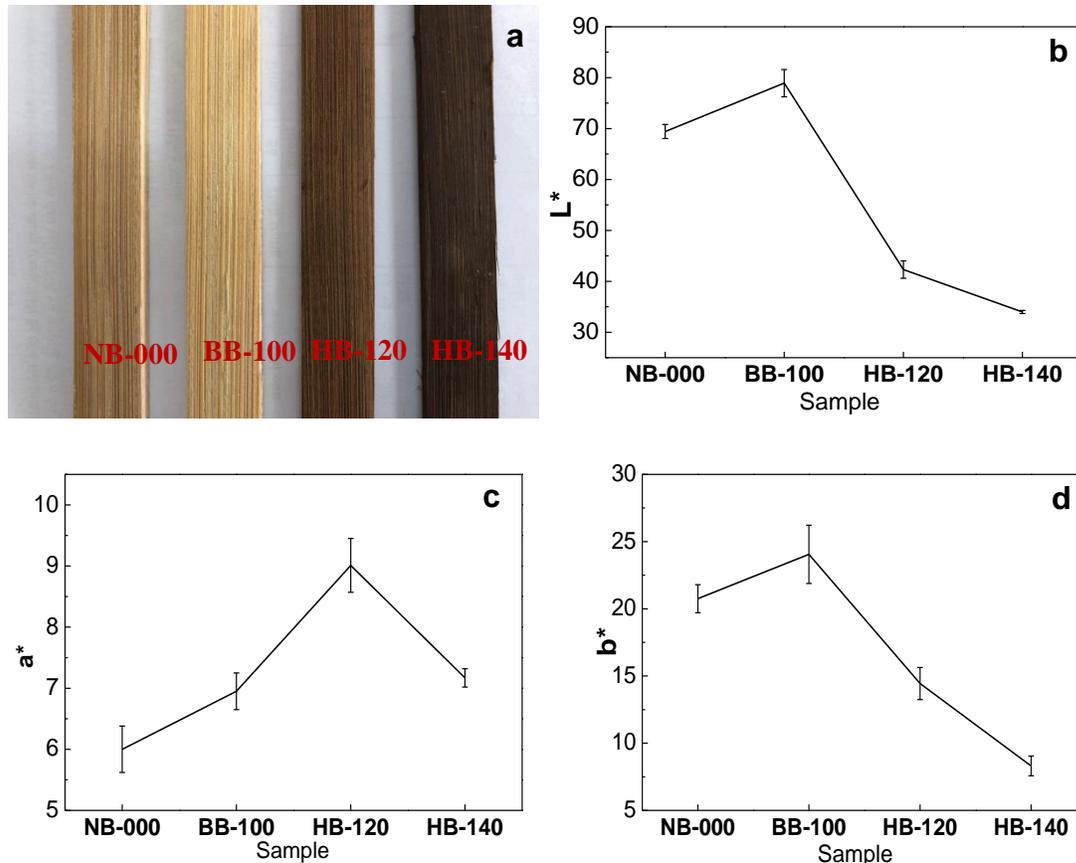
After 8 h of soaking time, the NB-000 sample had a water absorption of 18.8%, while the BB-100 sample had a water absorption of 21.5%. The HB-120 and HB-140 samples had the water absorption of 20.2% and 23.1%, respectively. The wettability of the *I. angustata* bamboo surface increased after the bleaching and heat treatments. This was due to the increased relative amount of carbonyl groups and the alcohol level of the carbon atoms on the surface (Ma *et al.* 2010). This had an impact on the ability of the bamboo to absorb the adhesive when the samples were hot-pressed.

However, when the samples were soaked for 24 h, the water absorption of the NB-100, HB-120, and HB-140 samples were 31.7%, 28.3%, and 27.4%, respectively. After soaking for 168 h, the water absorption of the NB-100, HB-120, and HB-140 samples were 52.1%, 41.4%, and 40.8%, respectively. The strongest water-absorbing component among the three major components is hemicellulose (Tjeerdsma and Militz 2005; Li *et al.* 2010). The heat treatment process additionally reduced the hemicellulose content (Table 1), therefore it was possible to decrease the permeability of the heat-treated samples.

### Effect of the Bleaching and Heat Treatment on the Color Differences

The hemicellulose and cellulose components of wood do not absorb visible light, but the lignin displays lignin color, which exists in the small cellulose fibers. The basic structure is phenyl propane, in which the phenol, quinones, and monoclonal carbonyl carboxyl chains contain carbon-oxygen (C-O) and carbon-carbon (C-C) bonds. These bonds form a conjugated double bond with a color genetic structure, which is an important source of color in wood (Peng *et al.* 2005; Liu *et al.* 2009; Martina *et al.* 2013). After the heat treatment, the color of the *I. angustata* bamboo changed, which caused its brightness to decrease sharply. A higher treatment temperature resulted in a lower brightness. Specifically, the color of the HB-120 sample decreased from 69.4 to 42.3, while the HB-140 sample dropped to 34.0 (Fig. 4b). This was because the lignin content in the *I. angustata* bamboo increased and the heat treatment process made the oxygen-containing functional groups (carboxyl and acetyl groups) disappear. This caused the carbon content to increase and for the samples to become darker (Bekhta and Niemz 2003; Sundqvist *et al.* 2006; Windeisen *et al.* 2007). The bleaching method improved the brightness of the

bamboo. The brightness of the BB-100 sample increased to 78.93 NBS, which was consistent with the results of previous studies conducted by Ashaari *et al.* (2004) and Hou *et al.* (2012).



**Fig. 4.** The *I. angustata* bamboo samples (a) visual appearance, (b)  $L^*$  values, (c)  $a^*$  values, and (d)  $b^*$  values

The changes in the  $a^*$  index values on the green-red axis can be seen in Fig. 4c. Compared to the natural color of the NB-000 sample, both treatment methods had a larger  $a^*$  coefficient. However, regarding the heat treatment method, the  $a^*$  coefficient decreased at a temperature of 140 °C when it was compared to the treated condition of 120 °C.

The changes in the  $b^*$  index values on the blue-yellow axis can be seen in Fig. 4d. After bleaching, the  $b^*$  value of the BB-100 sample increased and the color of the bamboo sample turned golden (Fig. 4a). However, the  $b^*$  index decreased for the heat-treated samples. Meanwhile, the HB-120 and HB-140 samples turned to a darker color.

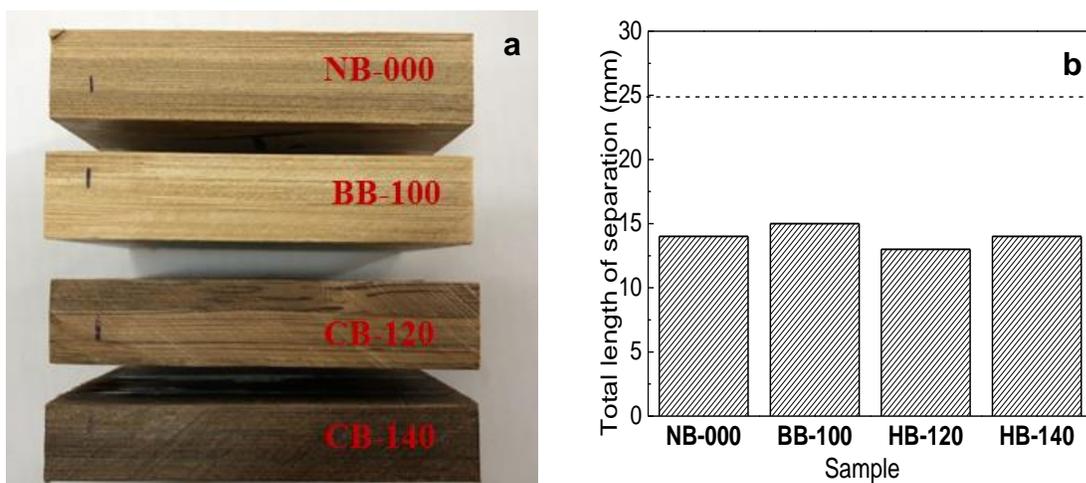
The color difference in the treated bamboo samples was markedly different ( $P < 0.05$ ) for the different treatment methods (Table 3). The bleaching treatment method increased the  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  to values all greater than zero. The *I. angustata* bamboo color tended to turn slightly red and yellow. For the heat treatment method, the  $\Delta L^*$  and  $\Delta b^*$  values were less than zero, while the  $\Delta a^*$  value was greater than zero. For the heat-treated samples, the *I. angustata* bamboo color tended to turn green and blue.

**Table 3.** Color Difference of *I. angustata* Bamboo after the Bleaching and Heat Treatment

Sample	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta E^*$
NB-000	-	-	-	-
BB-100	9.50	0.45	3.30	10.10
HB-120	-27.13	3.01	-6.31	28.01
HB-140	-35.45	1.16	-12.43	37.59

### The Delamination Test

After 10 h of drying at a temperature of 63 °C, the laminated bamboo was removed from the oven to calculate the total peeling crack length on every adhesive film. Considering the pressure and temperature, the crack length of the samples was small ( $P < 0.001$ ), and the total crack length on a glue film was smaller than 25 mm for all the samples (Fig. 5). Therefore, the bleaching and heat treatments did not affect the adhesive quality on the bamboo surfaces (Zhang *et al.* 2018). The two treatment methods were suitable for producing bamboo products for use in furniture products.



**Fig. 5.** (a) Visual examination of the bamboo samples and (b) the total length of separation for the sliver laminated bamboo adhesive film

### CONCLUSIONS

1. Both the bleaching and heat treatment methods affected the chemical composition and the structure of the treated bamboo strips. However, these methods had no effect on the quality of the adhesive paste of the laminated bamboo.
2. The bleaching method reduced the chromophoric nature of the lignin in the samples, which is the main light-absorbing element.
3. The heat treatment method did not reduce the hemicellulose content, which is the main water absorption factor in bamboo cells.

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