EFFECTS OF DRICON TREATMENT ON SURFACE PROPERTIES AND FIRE RESISTANCE OF TAIWAN AND CHINA MOSO BAMBOO (*PHYLLOSTACHYS PUBESCENS*)

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LEE CJ, CHUNG MJ, CHENG SS, CHANG TC, YANG TH & CHANG ST. 2016. Effects of Dricon treatment on surface properties and fire resistance of Taiwan and China moso bamboo (*Phyllostachys pubescens*). This study examined the effects of Dricon fire-retardant treatment on surface properties and fire resistance of Taiwan and China moso bamboo (*Phyllostachys pubescens*). Experimental results obtained by fourier transform infrared spectroscopy (FTIR) analysis revealed that the surface chemical properties for both inside and outside of untreated Taiwan and China moso bamboo were similar. After Dricon fire-retardant treatment, the hydroxyl groups on cellulose could react with cyanoguanidine, producing carbonylamide group, especially in China moso bamboo. However, different effects of Dricon fire retardant on China and Taiwan moso bamboo were observed due to their different material properties, and accordingly their colours after treatment also varied. Moreover, the gloss in all bamboo specimens decreased after treatment with Dricon. Owing to chemical modification by fire-retardant treatment, the contact angle with water decreased markedly, indicating that Dricon-treated moso bamboo had better wettability and could be processed with subsequent water-soluble reagents. Results obtained in this study also demonstrated that Dricon-treated moso bamboo had better combustion properties, which in turn enhanced its fire resistance.

Keywords: Fire-retardant, fourier transform infrared spectroscope (FTIR) analysis, UV spectroscopy analysis

INTRODUCTION

Global bamboo forest resources are estimated to be of 2,000 million hectares. Asia accounts for about 1,400 million hectares of bamboo forests, which is 70% of the total area of bamboo worldwide (Sharma et al. 1980). Bamboo, a green material, is abundantly available in the world mainly due to its rapid growth rate, renewable nature, high productivity and short maturity cycle (Hunter 2002). Moreover, owing to its multiple uses, bamboo has been widely employed for construction, manufacturing handicrafts and many other purposes (Wu 1982, Li 2000). In recent years, increasing concern for environment also enhanced bamboo as an environmental-friendly material. Previous research found that among different species of bamboo, moso bamboo (*Phyllostachys pubescens*) not only has good mechanical strength, hardness, wear resistance and dimensional stability, but also a rapid growth rate which attains mature and strong tissue structure within a short period of 3 to 5 years. It is the most widely used and common bamboo species of high economic value in Taiwan and China (Li 2000, Lu 2001, Jiang et al. 2002, Cheng et al. 2006, Lin et al. 2006).

There has been much research in the past two decades on the mechanical properties and utilisation of moso bamboo, aiming to expand its market potential and, in particular, to explore the feasibility of its application as a possible alternative to wood. Moso bamboo has similar properties to wood and has been used for making furniture and construction (Lee et al. 1996, Zhonghua 2002, Talabgaew & Laemlaksakul 2007). In addition, laminated moso bamboo has also been widely used in non-structural components, such as wall partition, flooring, ceiling and interior decoration (Nugroho & Ando 2000, 2001, Shan & Li 2008).

On the other hand, when using wood and wood-based composites in construction, fire and safety are the main concerns because the woody composite materials are inflammable and do not conform to the specifications for fire protection (Talabgaew & Laemlaksakul 2007). To address such concerns, these materials were treated with fire retardant, such as borax, boric acid, mono- and di-ammonium phosphates (MAP and DAP) and ammonium sulphamates, by vacuum pressure impregnation (VPI) or intumescent flame retardant coating (LeVan & Winandy 1990). According to Zaihan et al. (2009), laminated *Shorea parvifolia* plywood treated with Dricon fire retardant containing MAP, DAP and mixture of borax and boric acid (BBA) at 20% could remain inflammable for a longer period of time. Previous research also reported that fire-retardant treatment on laminated wood significantly affected the gluing of adhesives and the processing of coating (LeVan & Winandy 1990, Lebow & Winandy 1999). While structural performance of wood-based composites treated with fire retardant has been studied (Wang & Rao 1999), there is little research on the impact of fire-retardant treatment on surface characteristics of bamboo culms or laminated bamboo materials, which would in turn affect both subsequent processing and final quality of the treated materials. Such knowledge would be significant for future development and application of fire-resistant composite materials.

In view of the above, this study aimed to examine the effects of fire-retardant treatment on surface characteristics and fire resistance of moso bamboo. Moso bamboo was treated with Dricon fire retardant and its fire resistance was tested according to ASTM E1354 standard measurement protocol. Furthermore, the chemical structure, colour, gloss, contact angle with water and pH of Dricon-treated moso bamboo were investigated to show the changes in surface chemical characteristics after treatment. Comparison was also made between Taiwan and China moso bamboo. The results thus obtained provided better understanding of the properties and quality of fire retardant-treated moso bamboo, which would not only help reduce production cost but also increased the diversity of its application, thus enhancing the potential development of the bamboo industry.

MATERIALS AND METHODS

Materials

Three-year-old stripe moso bamboo (*Phyllostachys pubescens*) culms were obtained from the experimental forest of National Taiwan University, Taiwan and Wuyi Mountain in Fujian Province, China. All specimens were cut into strips with dimensions of 180 (length) × 2.0 (width) × 0.5 (thickness) cm. Prior to treatment, all specimens were first bleached and disinfected using 2% H_2O_2 solution at 100 °C for 30 min, then dried in a kiln and finally stored in the dark. The Dricon fire retardant was composed of cyanoguanidine ($C_2H_4N_4$), ammonium dihydrogen phosphate ($NH_4H_2PO_4$), phosphoric acid (H_3PO_4) and boric acid (H_3BO_3). According to the material safety and data sheet (MSDS) of Dricon, the proportion of Boric acid is < 5% and guanylurea phosphate is < 10%. The Dricon solution was diluted to 12% concentration at pH 2.8. The percentage of weight increase in Taiwan and China moso bamboo were 8 and 14% respectively.

Methods

The fire-retardant treatment was conducted inside a commercial vacuum impregnation chamber using Bethell process. All specimens were first impregnated with an initial vacuum of 60 cm Hg^{-1} for 30 min, followed by treatment with Dricon at 12% concentration under a pressure of 14 kgf cm^{-2} for 4 hour and a vacuum of 50 cm Hg^{-1} for 30 min. All specimens were weighed before and after treatments to determine the chemical loading according to the difference in weight. Treated specimens were washed with water and dried in a kiln. After drying to a final moisture content of 12 ± 3%, treated bamboo specimens were heat pressed and spliced together using a high-frequency dielectric bonding machine with adhesive and cut into square specimens with dimensions of 10.0 (length) × 10.0 (width) × 2.0 (thickness) cm. The treated specimens were stored at ambient temperature without exposure to light prior to subsequent analyses.

Colour measurement

Following the procedures employed by Chung et al. (2008), the surface colour of the Dricon-treated moso bamboo specimens was measured using a colour difference meter under D_{65} light source and a test-window of 10 mm diameter. The changes in brightness (L*) and tone (a*and b*), parameters recommended by the Commission Internationale d'Eclairage, were calculated using the following formulas (CIE 1976).

$\Delta L^* = Lt^* - Ls^*$
$\Delta a^* = at^* - as^*$
$\Delta b^* = bt^* - bs^*$

where L* = value on the white-black axis, a* = value on the red-green axis, b* = value on the blue-yellow axis, Lt*, at* and bt* = treated specimen and Ls*, as* and bs* = control.

Fourier transform infrared spectroscope (FTIR)

The FTIR analysis of the bamboo specimens was conducted using a spectrometer incorporated with a diffuse reflectance accessory unit. All the spectra were measured at a resolution of 4 cm^{-1} and 64 scans were recorded per specimen.

Gloss and wettability measurement

The 60° gloss value was probed by a reflectometer from the surface of both untreated and treated bamboo specimens. Wettability of specimen epidermis was evaluated using a contact-angle meter.

Surface pH measurement

A pH meter with a flat surface electrode was employed to measure the surface pH of bamboo specimens. The meter was calibrated prior to measurements using standards at pH 4.0 and 7.0. The electrode was dipped in distilled water before being applied to the area measured.

Fire resistance

A cone calorimeter was employed to evaluate the efficiency of the fire retardant, as specified by the ASTM Standard E 1354 method. For evaluating the fire resistance of laminated moso bamboo, the adhesive used for splicing the dried Dricon-treated moso bamboo was composed of styrene butadiene latex (SBR) and 15% isocynate. Specimens were placed in a horizontal orientation with an external igniter and the flux of the cone heater was set at 50 kW m^{-2}. The test duration was 300 s and all tests were conducted in triplicates. The following heat properties were determined; average heat release rate [HRR_{av} (kW m^{-2})] within 300 s, peak heat release rate [PHRR (kW m^{-2})], total heat release [THR (MJ m^{-2})], time to ignition [Tig (s)] and time to PHRR (s). The PHRR and HRR_{av} values correspond to the acceleration of thermal degradation of the materials, which is related to the spread of fire. Total heat release (THR) indicates the total heat capacity during the combustion process.

RESULTS AND DISCUSSION

Surface chemical structure of Dricon-treated bamboo

Figures 1 and 2 shows the variations in fourier transform infrared spectroscopy (FTIR) spectra of treated China and Taiwan moso bamboo respectively. As seen in Figure 1, the FTIR results indicated no difference in chemical structure between the outside and inside of untreated China specimens. The infrared (IR) absorption peaks at 2193 (C≡N), 2155 (C=N–C) and 1643 (C=N) appeared at both outside and inside of Dricon-treated China moso bamboo. The new absorption peak at 1558 belonged to the N-H deformation frequencies of secondary amide (Rowell & Ellis 1981). Obvious decrease in intensity of C-O absorptions of lignin and cellulose at 1134, 1092 and 1065 cm^{-1} and the C-N absorption band of cyanoguanidine at 2155 suggested that the N-H absorptions at 1558 could be from the reaction of cellulose/lignin and $C_2H_4N_4$. Saunders et al. (1967) studied the curing mechanism of $C_2H_4N_4$-epoxy resin and reported that the cyanide group (–C≡N)

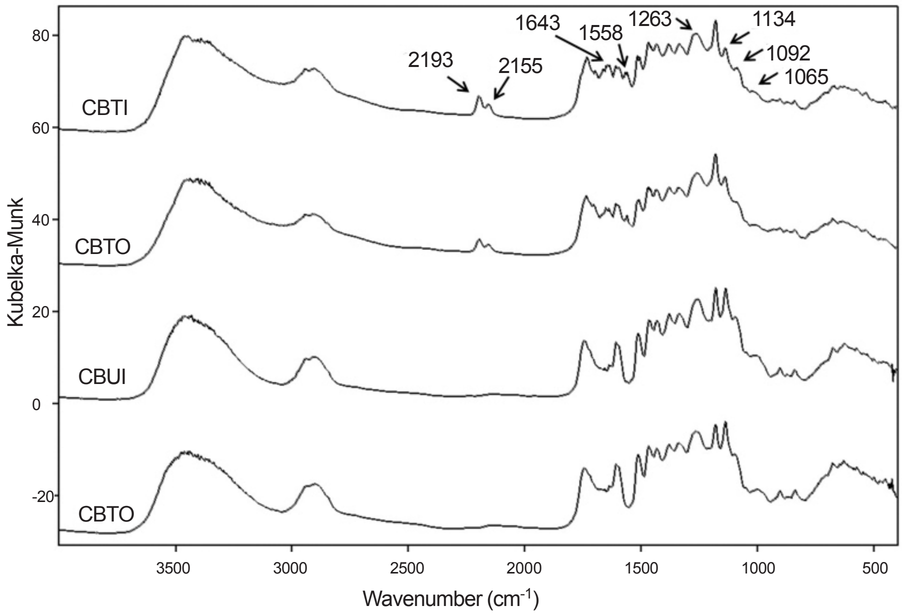

Figure 1 FTIR spectra of Dricon-treated China moso bamboo (CBUO = outside of untreated specimen, CBUI = inside of untreated specimen, CBTO = outside of treated specimen and CBTI = inside of treated specimen

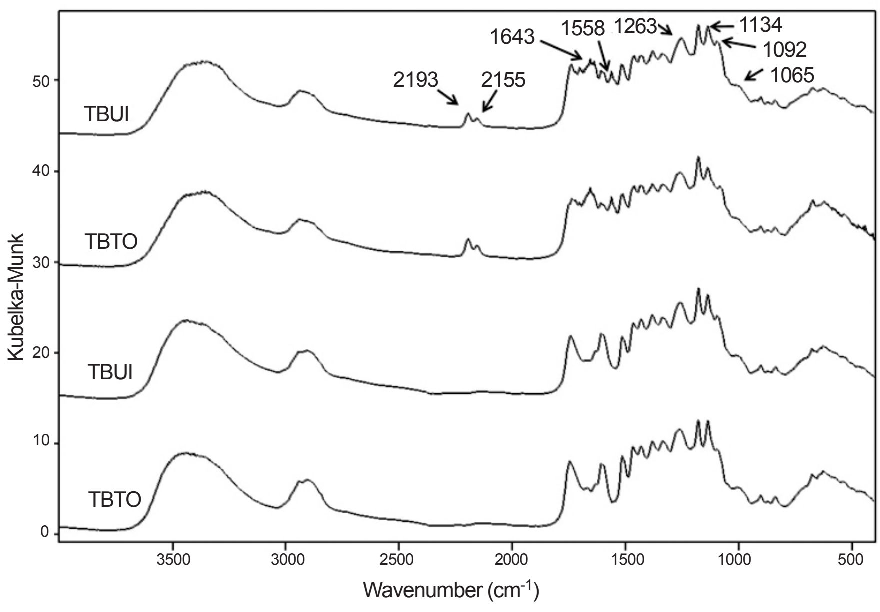

Figure 2 FTIR spectra of Dricon-treated Taiwan moso bamboo, TBUO = outside of untreated specimen, TBUI = inside of untreated specimen, TBTO = outside of treated specimen and TBTI = inside of treated specimen

of $C_2H_4N_4$ could react with hydroxyl group (–OH) to form imino ether group (–O–C=NH). Then the carbonylamide group (–N(H)–C(O)-) would be formed from rearrangement of imino ether group. According to this mechanism, it may be presumed that similar reaction would occur in the Dricon-treated bamboo. However, no variation was observed in the intensity of C–O absorption band of lignin at 1263 cm^{-1}, thus it could be inferred that the reaction occurred between the cellulose and cyanoguanidine in Dricon. The conceivable reaction mechanism is shown in Figure 3. In other words, the chemical structure of moso bamboo would change after being impregnated with Driconfire retardant.

Comparison between Figures 1 and 2 revealed that Dricon-treated China and Taiwan moso bamboo yielded similar FTIR results. However, the extent of decrease in intensity of cellulose absorption was smaller in Taiwan specimens than in China ones, implying that the surface chemical structure of Taiwan moso bamboo was less affected by treatment with Dricon fire retardant. Such difference might be attributed to the reactivity of cellulose with various crystallinities. The rate of chemical reactivity would be reduced with cellulose of higher crystallinity (Tsoumis 1991). The crystallinity, hardness and elastic modulus of latewood cell walls were higher than those of earlywood cell walls. Higher crystallinity can endow lignocelluosic material with larger modulus of elasticity (MOE) (Wimmer et al. 1997, Chung et al. 2008). In this study, Taiwan moso bamboo was found to have larger MOE than China moso bamboo, implying that Taiwan moso bamboo had higher cellulose crystallinity, and thus lower reactivity with cyanoguanidine, compared with China moso bamboo.

Surface colour of Dricon-treated bamboo

Table 1 shows the changes in colour of China and Taiwan moso bamboos after treatment with Dricon fire retardant. The inside of treated China

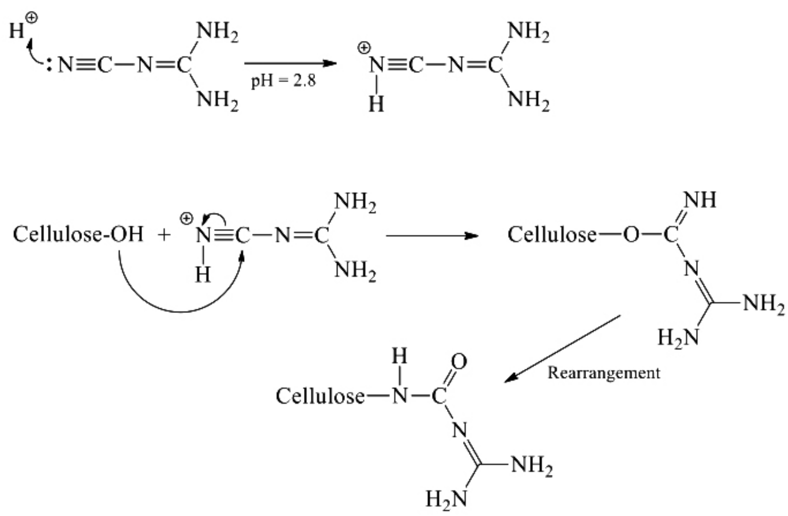

Figure 3 Conceivable mechanism of the reaction of cyanoguanidine and cellulose

Table 1 Changes in colour of China moso bamboo and Taiwan moso bamboos after treatment with Dricon fire retardant

Specimens			L*	a*	b*
China bamboo	Inside	Untreated	84.38 ± 1.31 a	3.47 ± 0.63 a	27.88 ± 0.45 ab
		Treated	79.60 ± 0.84 b	4.36 ± 0.53 a	29.04 ± 1.85 ab
	Outside	Untreated	80.46 ± 2.50 ab	5.67 ± 1.43 a	31.45 ± 1.09 a
		Treated	78.69 ± 1.25 b	4.73 ± 0.65 a	25.72 ± 1.54 b
Taiwan bamboo	Inside	Untreated	83.81 ± 0.37 a	2.61 ± 1.02 b	26.90 ± 1.22 a
		Treated	65.64 ± 0.68 d	5.84 ± 0.46 a	25.23 ± 2.22 a
	Outside	Untreated	81.36 ± 0.56 b	3.69 ± 0.92 b	27.44 ± 1.22 a
		Treated	67.83 ± 0.79 c	7.58 ± 0.37 a	28.66 ± 1.89 a

Data are expressed as mean (SD) (n = 3), i.e. numbers followed by different letters (a–d) are significantly different at the level of $p < 0.05$ according to Scheffe's test

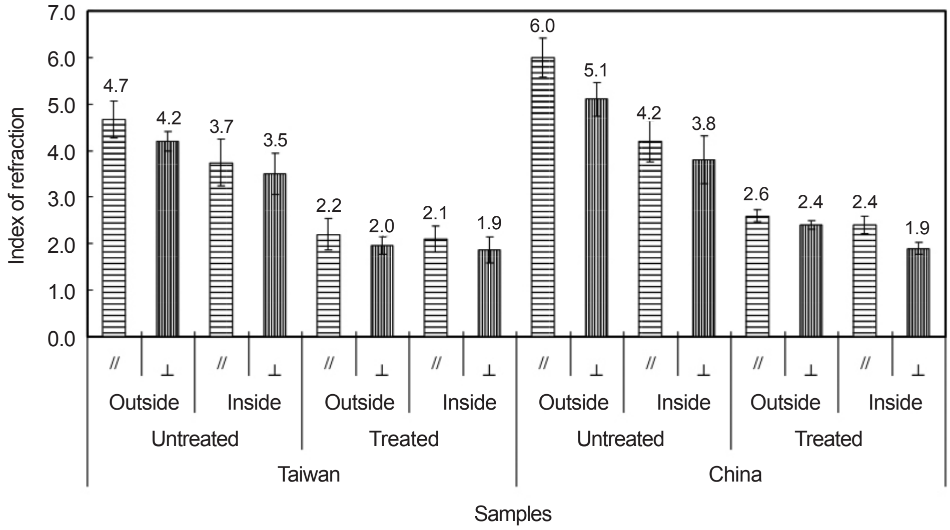

Figure 4 Changes in 60° gloss values on surface of Dricon-treated moso bamboo, // = incident angle parallel to the grain, ⊥ = incident angle vertical to the grain

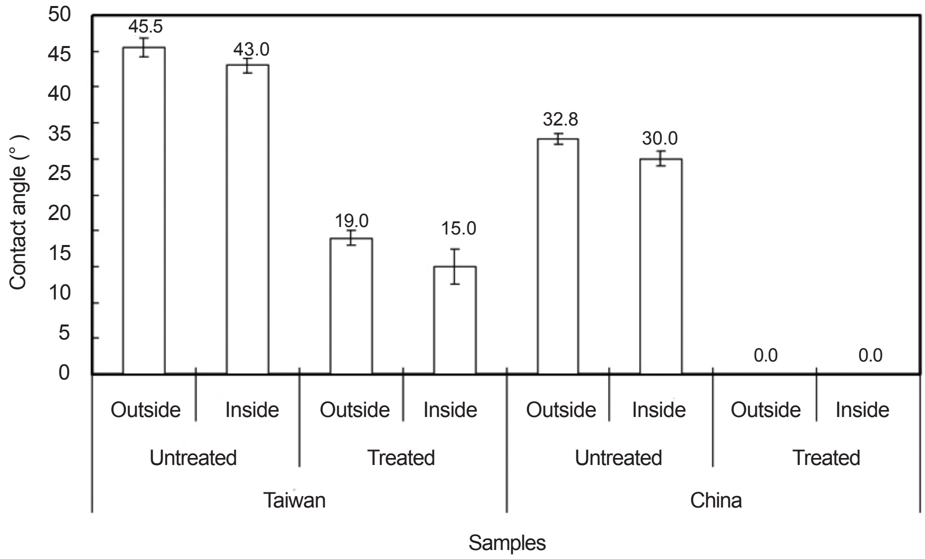

Figure 5 Changes in contact angle on surface of Dricon-treated moso bamboo

specimens showed significant changes in L* value (falling from 84.38 to 79.60, $\Delta L^* = 4.78$), indicating that only brightness decreased. The outside of treated China specimens showed marked changes in b* value (falling from 31.45 to 25.72, $\Delta b^* = 5.73$) revealing fading of the yellow color after treatment with Dricon fire retardant. Both inside and outside of Taiwan moso bamboo specimens showed significant changes in L* value (falling from 83.31 to 65.64 , $\Delta L^* = 17.67$; and from 81.36 to 67.83 , $\Delta L^* = 13.53$, respectively) and a* value (rising from 2.61 to 5.84, $\Delta a^* = 3.23$; and from 3.69 to 7.58, $\Delta a^* = 3.89$, respectively). In other words, fire-retardant treatment led to decrease in brightness and increase in redness both inside and outside of Taiwan moso bamboo.

In comparison, China and Taiwan specimens showed different trends of color changes, which might be attributable to the different effects of Dricon fire retardant. After Dricon treatment, the density of China specimens decreased from 0.81 ± 0.01 to 0.62 ± 0.04, whereas the density of Taiwan specimens increased from 0.53 ± 0.08 to 0.70 ± 0.06 (t-test, $\alpha = 0.05$). Furthermore, higher cellulose crystallinity of Taiwan specimens,

Table 2 Changes in pH values on the surface of Dricon-treated moso bamboo

Samples	Taiwan				China			
	Untreated		Treated		Untreated		Treated	
	Outside	Inside	Outside	Inside	Outside	Inside	Outside	Inside
Average	4.54	4.60	4.70	4.70	4.40	4.50	4.71	4.79
SD	0.08	0.02	0.06	0.04	0.07	0.06	0.09	0.06

Data are expressed as mean of 9 samples and SD values

Table 3 Results from cone calorimeter evaluation of different bamboo specimens

Specimens	HRR_{av} (kW m^{-2})	PHRR (kW m^{-2})		Time to PHRR (s)		THR (MJ m^{-2})
		Stage I	Stage II	Stage I	Stage II	
Untreated Taiwan bamboo	115 ± 3.4	184 ± 3.8	119 ± 11.4	52 ± 3.0	226 ± 4.7	34 ± 1.2
Treated Taiwan bamboo	64 ± 4.8	126 ± 7.7	66 ± 6.7	43 ± 3.0	166 ± 16.0	19 ± 1.5
Untreated China bamboo	110 ± 4.7	198 ± 20.2	-	44 ± 6.0	-	33 ± 1.4
Treated China bamboo	46 ± 0.8	73 ± 2.8	-	49 ± 2.1	-	14 ± 0.3

HRR_{av} = average heat release rate, PHRR = peak heat release rate and THR = total heat release

compared with that of China specimens, retarded the reaction with cyanoguanidine. Hence, Dricon fire retardant tended to accumulate in vascular bundles of Taiwan specimens. In this way, Dricon fire retardant contributed more to the natural colour of treated Taiwan moso bamboo.

Gloss of Dricon-treated bamboo

Figure 4 shows the 60° gloss in two different fibre orientations on the outside and inside of both treated and untreated bamboo specimens. The gloss values of untreated Taiwan specimens in both parallel and vertical fibre directions were higher on the outside than on the inside (4.7 and 4.2 vs 3.7 and 3.5 respectively). The same phenomenon was also observed in untreated China specimens (6.0 and 5.1 vs 4.2 and 3.8 respectively), whose gloss values were higher than those of their Taiwan counterparts. However, after Dricon fire-retardant treatment, the gloss values in both fibre directions on the outside and inside of Taiwan specimens showed no significant difference (2.2 and 2.0 vs 2.1 and 1.9 respectively). The same was also observed for the treated China specimens (2.6 and 2.4 vs. 2.4 and 1.9 respectively). In addition, the gloss values between treated Taiwan and China specimens became similar. Thus, it could be concluded that the gloss of both Taiwan and China moso bamboo decreased significantly after Dricon fire-retardant treatment and there was no significant difference between both treated specimens.

Contact angle of Dricon-treated bamboo

Figure 5 shows changes in contact angle of Dricon-treated bamboo surface with water. Both outside and inside of untreated Taiwan specimens had higher contact angles than their China counterparts (45.5° and 43.0° vs 32.8° and 30.0° respectively), indicating that untreated China moso bamboo had better wettability. After Dricon fire-retardant treatment, there was an apparent decrease in contact angle for all specimens, i.e. the outside of treated Taiwan moso bamboo dropping from 45.5° to 19.0° while both outside and inside of treated China moso bamboo, even reaching 0°. The significant decrease in contact angle after fire-retardant treatment could be attributed to the presence of the C–NH functional group from cyanoguanidine. This hydrophilic functional group produced after Dricon treatment enhanced the wettability of moso bamboo.

The pH value of Dricon-treated bamboo

A droplet of water was dropped on the surface of bamboo specimen. Then the pH probe was

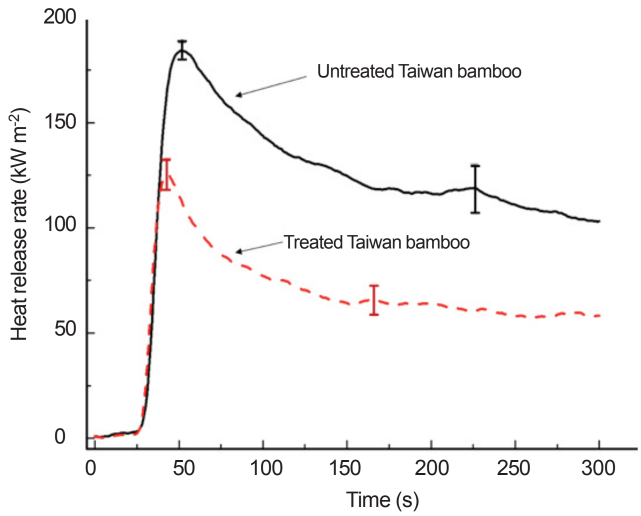

Figure 6 Heat release profiles of untreated and treated Taiwan moso bamboo

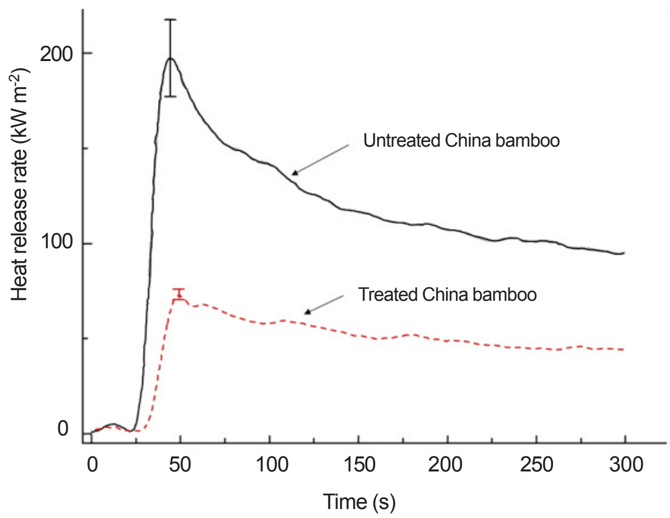

Figure 7 Heat release profiles of untreated and treated China moso bamboo

put on the wetted surface to measure the pH value. Table 2 shows changes in pH value of Dricon-treated bamboo. The outside and inside of all specimens, both untreated and treated, had similar pH values. Although treatment with Dricon led to a slight increase in pH values, the difference was not marked, indicating insignificant influence of fire-retardant treatment on pH value of moso bamboo.

Combustion properties of Dricon-treated bamboo

Table 3 lists the heated properties of different bamboo specimens. Figures 6 and 7 display the heat release profiles of Taiwan and China moso bamboo, respectively. In comparison, bamboo specimens after fire-retardant treatment had lower HRR_{av} and THR. The PHRR of treated specimens also showed a declining trend. The stage I PHRR of both Taiwan and China specimens decreased significantly from 184 to 126 kW m^{-2} and from 198 kW m^{-2} to 73 kW m^{-2} respectively. The findings shown in Table 4 as well as Figures 6 and 7 revealed that after treatment with fire retardant, both Taiwan and China moso bamboo had better combustion properties.

Fire retardants, such as boric acid and ammonium phosphate, can lower the decomposition temperature and accelerate the formation of a carbonised layer on the materials (Chuang et al. 2008). As a result, THR can be significantly decreased by treatment with Dricon. Dricon comprises boric and ammonium phosphate, therefore, it can form a carbonised layer on the bamboo surface and decrease the heat release rate.

CONCLUSIONS

The FTIR analysis showed that the surface chemical properties were similar for both inside and outside of untreated Taiwan and China moso bamboo. After Dricon fire-retardant treatment, the obvious decrease in intensity of C–O absorptions of lignin and cellulose at 1134, 1092 and 1065 cm^{-1} and the C–N absorption band of cyanoguanidine at 2155 cm^{-1} suggested that the N-H absorptions at 1558 cm^{-1} was form the reaction of cellulose/lignin and $C_2H_4N_4$. However, different effects of Dricon fire retardant on China and Taiwan bamboo were observed due to their different material properties, leading to variations in colour changes. The gloss also decreased in all specimens after treatment with Dricon fire retardant. Finally, the combustion properties of both Taiwan and China moso bamboo became better after fire-retardant treatment, thus enhancing its fire resistance. In short, treatment with Dricon fire retardant can improve both surface properties and fire resistance of moso bamboo.

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