Processing bamboo for structural composites: Influence of preservative treatments on surface and interface properties

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\textbf{A B S T R A C T}

Engineered bamboo is being increasingly explored for structural use in the construction sector. To ensure durability, products such as laminated bamboo undergo essential preservation treatment steps during their manufacture. However, the effects of hygro-thermal \textit{caramelisation} and chemical \textit{bleaching}, two commonly used treatment procedures in industry, on the surface and interfacial properties of laminated bamboo are not yet known. The latter governs the structural quality of the final product. Our dynamic wettability studies through contact angle measurements revealed lower water contact angles, higher total surface energy and a slightly greater polarity ratio for bleached bamboo in comparison to caramelised and untreated bamboo. In addition, lap-joint shear tests and Weibull reliability analysis established the significantly better adhesive bonding performance of bleached bamboo with all five surveyed adhesives. Our observations are explained through the changes in chemical composition and structure of the raw bamboo material upon treatment, where changes in caramelised and bleached bamboo are dominated by hemicellulose degradation and lignin degradation, respectively. The bleaching process is conclusively identified as having favourable effects on bond strength of the glue line.

1. Introduction

Bamboo is an outstanding natural composite: it is rapidly growing, low-cost and abundantly available, light, yet stiffer and stronger than both timber and (chopped strand mat) glass fibre composites [1,2]. Structural use of the material in raw form – as a hollow cylinder – is limited by geometrical and mechanical variability. Laminated bamboo composites [3,4] exploit the inherent composite structure of bamboo and maintain the longitudinal fibre direction, and therefore the inherent strength of the raw material. A typical process used in industry to produce such an engineered material is illustrated in Fig. 1.

The high moisture and carbohydrate (both starch and sugar) content of bamboo make the untreated material highly susceptible to bio deterioration [5–7]. Untreated bamboo has a natural durability of less than two years, improving to up to seven years if exposure to the elements (vis. water and UV light) is limited [5–7]. For applications in the built environment, the service life of a construction material needs to be much longer. Therefore, an important step in the production of laminated bamboo, and in fact any raw or engineered bamboo product, is the preservative treatment for improved durability, by up to three to five times the natural lifespan [6]. Currently, two methods are in primary use to modify carbohydrates in laminated bamboo [8]: \textit{a}) \textit{caramelisation} or \textit{steaming} (a hygro-thermal treatment), using pressurised (0.20–0.25 MPa) wet steam at 120–130 °C over 5–6 h yielding a darker brown colour, or \textit{b}) \textit{bleaching} (a chemical treatment) in a hydrogen peroxide bath at 70–80 °C over 4 h resulting in a lighter yellow colour. Both processes are followed by steam-assisted drying at 50–60 °C for a total of 240 h for caramelised bamboo and 72 h for bleached bamboo. Foreseeably, the preservative treatment step is energy intensive, accounting for up to 30% of the embodied energy of the finished board products [8]. The selection of the treatment process in industry has been driven by the resulting colour and hue of the bamboo material and was adopted from wood processing.

Amongst the sparse literature on the properties of these treated bamboo composites, recent investigations have demonstrated that the treatment method affects the mechanical and fracture behaviour of the laminated bamboo material [3,9]. Specifically, bleached bamboo composites are \textit{i}) softer and more ductile, \textit{ii}) stiffer/stronger in tension but weaker in flexure and compression (the latter being more relevant for construction materials), and \textit{iii}) more prone to fibre pull-out, than their caramelised counterparts [3].

Lamination of the bamboo strips to form structural composites relies...
on adequate inter-ply bonding via adhesives. A handful of studies in literature have assessed adhesion and bonding properties of bamboo laminates and the effects of (less common) preservative treatments such as wet (e.g. with steam or oil) or dry heat treatment [10–12], and chemical treatment [13–15]. All studies have found that treatments tend to have adverse effects on both wettability and bonding strength of the glue line, with the latter reducing by 15% to 75%. For engineered bamboo products to be seriously considered for external building and construction applications, the effects of processing and conditions during service life (e.g. humidity cycles) on interface properties need closer inspection.

In the present industrially-relevant study, we perform dynamic contact angle measurements on untreated (raw) and preservative-treated (caramelised or bleached) bamboo to determine surface properties (i.e. surface energy). Notably, there are only limited studies in existing literature that have assessed the time-dependent wetting behaviour [16,17] and surface energy [18] of bamboo. This is valuable information which will give insight into adhesive wettability of bamboo; that is, the ability of adhesives to make intimate contact with bamboo surfaces. In addition, through a systematic study exploring the bonding performance of a wide range of common petroleum-based and novel bio-based adhesives through lap-joint shear tests, we aim to ascertain which adhesives may be better suited for the different processed materials. By studying commercially treated bamboo, we are also able to describe variability in shear strength in terms of Weibull failure probability for improved quality control and reliability analysis.

2. Experiments and methods

2.1. Materials

Raw Moso bamboo (Phyllostachys pubescens) was obtained in whole culm form from China (supplied by UK Bamboo Supplies Limited). The culms were air-dried and sun-bleached for three weeks, and for the purposes of this study are referred to as the ‘untreated’ material. The culm was machined and planed to obtain strips of dimensions approximately 115 mm in length in the grain direction, 20 mm wide, and 5 mm thick. For the ‘treated’ Moso bamboo specimens, commercially produced bleached strips and caramelised laminated sheets were obtained from Moso International B.V. (Netherlands) and Plyboo, Smith & Fong Co. (U.S.A) respectively. The caramelised laminated sheets were cut to obtain monolayer strips. All specimens were conditioned at 23 °C and 55% relative humidity for at least two weeks before testing. Fig. 2a) shows examples of the bleached, untreated and caramelised bamboo materials.

In the contact angle measurements, purified water (Milli-Q, Millipore Corporation) and diiodomethane (Sigma Aldrich) were used as the probing fluids.

For adhesion testing, five commercially available structural adhesives were used: polyurethane (PU: Purbond, Henkel, Switzerland), polyvinyl acetate (PVA: Lumberjack wood adhesive, Everbuild, UK), soy-flour based adhesive (Soy: Soyad, Solenis, USA), resorcinol phenol formaldehyde (RPF: Polyproof, Polyvine, UK), and urea phenol formaldehyde (UPF: Cascamite, Polyvine, UK).

2.2. Contact angle measurement

Dynamic contact angle measurements were made using the sessile drop technique on a FTA1000 instrument (First Ten Angstroms, USA), equipped with an AV-GC750 CCD camera (Allied Vision Technologies, Germany). To eliminate the effects of differences in surface roughness, all specimens were dry-polished with 120 grit sandpaper followed by air-blowing to remove debris prior to testing. Tests were carried out at room temperature (20 °C) at a relative humidity of around 50–60%.

Two different probing fluids were used: purified water (polar) and diiodomethane (non-polar). Drops were formed on a micro-syringe to a
critical volume (~8 μL for water and ~2 μL for diiodomethane) until they dropped onto the surface of the material. The shape of the drops on the surface was photographed at regular time intervals (0.2 seconds) until they reached steady-state at t = t eq (up to 60 seconds for water and up to 10 seconds for diiodomethane). Evaluation of contact angles was carried out using the FTA32 software (First Ten Angstroms, USA) by ellipse matching, and taking an average of left and right contact angles. The contact angle θ t at time elapsed t is defined as the angle between the bamboo surface and the liquid-air interface (Fig. 3a). Dynamic contact angle measurements were performed for each probing fluid and bamboo material combination.

2.2.2. Surface energy analysis

Surface free energy of the bamboo materials γSV was determined through the OWRK (Owens, Wendt, Rabel and Kaelble) geometric mean method (Eq. 1) [19], and a system of equations based on the equilibrium contact angles (θ t at t = t eq) for water and diiodomethane.

\[
\frac{\gamma_{LV}(1 + \cos \theta_t)}{2\sqrt{\gamma_d^2 + \gamma_p^2}} = \sqrt{\gamma_d^2 + \gamma_p^2} \sqrt{\frac{\gamma_{SV}}{\gamma_{LV}}}
\]

where, γSV is the surface free energy of the bamboo material, γLV is the surface tension of the probing liquid, and the superscripts d and p denote dispersive and polar components of surface energy.

Surface free energy is a sum of the independent dispersive and polar components [20]; dispersive interactions are associated with van der Waal interactions and electron dipole fluctuations, whereas polar components are associated with a combination of Lewis acid-base and hydrogen bonding components [20]. For water, a polar liquid, γLV = 72.8 mJ/m², γdLV = 21.8 mJ/m², and γpLV = 51.0 mJ/m² [19,20]. For diiodomethane, a non-polar liquid, γLV = 50.8 mJ/m², γdLV = 50.8 mJ/m², and γpLV = 0.0 mJ/m² [19,20].

2.2.2.2. Adhesive lap-joint shear strength testing

The interfacial properties of the bamboo materials were determined through single lap-joint shear tests. Strips of raw and treated bamboo, cut to sizes recommended by ASTM D3163 [21], were bonded with the five different commercial adhesives listed in the materials section, as schematised in Fig. 3b. All adhesives were applied using a glue rate of 180 g/m² and clamped manually at 0.6 MPa for 8 h. All specimens were left to fully cure and equilibrate for at least 72 h prior to testing. Mechanical testing was conducted on an Instron universal test frame, equipped with a 150kN load cell, in displacement control at a rate of 1.27 mm/min. The ‘apparent’ shear strength was determined from the failure load. The failure surface was visually examined and qualitatively classified as an adhesive, cohesive/mixed, or substrate failure (Fig. 2b). Thereafter, moisture content of the samples was determined by the oven drying method.

2.2.3. Weibull analysis of strength

Often, strength properties (resulting from brittle failure) exhibit significant scatter [22]. The Weibull distribution is commonly used to describe strength distribution, and therefore assess the probability of survival (or failure) for a given applied stress. This is useful for reliability analysis and to obtain characteristic strength (for comparative purposes). Importantly, the distribution can be physically explained through the weak-link theory, which in the context of this study would suggest that the interface (at the lap-joint between the bamboo surface and the adhesive) is composed of multiple ‘links’, and failure will occur at the weakest-link (i.e. where there is a detrimental flaw).

Here, we use the two-parameter Weibull probability distribution (Eq. 2) [22].

\[
P_F(n) = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^m\right)
\]

where, P_F is the failure probability ( = 1 – survival probability), σ is the apparent shear strength, σ_0 is the Weibull scale factor or the characteristic strength (refers to the applied stress at which failure probability is 63%), and m is the Weibull shape parameter or modulus (describes scatter in strength data, and therefore defects).

For the analysis, first, strength data was ranked n in order of ascending strength, so that n = 1 corresponds to lowest and n = N corresponds to highest. A failure probability P_F(n) (Eq. 3) was then assigned to each data-point. Weibull plots were produced with P_F against σ on a log-log scale. Linear regression analysis enabled determination of the Weibull scale and shape parameters.

\[
P_F(n) = \frac{n}{N + 1}
\]

3. Results and discussion

3.1. Dynamic wettability and surface energy

Dynamic contact angle measurements on the untreated and treated bamboo surfaces were performed with water and diiodomethane as the probing fluids (Fig. 4). In Fig. 4a1-a3, images of typical droplet shapes of water (at t = 0 and t = t eq ~ 60 s) and diiodomethane (at t = t eq ~ 10 s) are shown on the different bamboo surfaces. Fig. 4b1–b2 plot the evolution of contact angle over time.

While the contact angle for water exhibited an exponential decay, becoming fairly stable for t > 20 s, the contact angle for diiodomethane was stable throughout. The reducing contact angle in the case of water, a polar liquid, was due to a combination of spreading on the bamboo surface and absorption into the bamboo cellular structure. This is commonly observed on wood surfaces and various exponential decay models have been proposed to describe such a dynamic wetting process [23–27]. Such models have yet to be applied to bamboo, and in fact, the dynamic wettability of bamboo surfaces (not extracted fibres) is rarely...
reported in literature [16,17]. We found that simple models, such as the natural decay model (Eq. 4) commonly applied to wood [26], had a good fit to our experimental data for bamboo (Fig. 4b1), $R^2 > 0.85$.

Fitted parameters to the experimental data are shown in Table 1.

$\theta_t = a + be^{-kt}$  

(4)
where \( a \) denotes the equilibrium contact angle, \( a + b \) gives the initial contact angle (at \( t = 0 \)), and \( K \) is a constant which dictates the rate of decay (and is influenced by the rate of spreading and penetration).

The initial and equilibrium water contact angles with the different bamboo surfaces followed the trend of untreated > caramelised > bleached (Fig. 4, Table 1). Bleached bamboo therefore was most wettable and formed a more intimate contact with polar liquids. Bleached bamboo also exhibited better droplet spreading and liquid penetration. In contrast, with diiodomethane, the equilibrium contact angle at the liquid-bamboo interface was comparable for both bleached and caramelised surfaces, implying comparable wettability with non-polar liquids, however substantially poorer than that for polar liquids.

Dynamic contact angle measurements on bamboo in literature, although sparse, are comparable to results in the present study. In their wettability study, Chen et al. [16] observed that the initial and equilibrium contact angles at the water-untreated bamboo interface was at 60–70° and < 5°, respectively. Li [17] found that urea formaldehyde resin (a relatively polar liquid) and untreated bamboo (in the middle section) had an initial and equilibrium contact angle of 60° and 35°, respectively, although this can vary substantially with different sections (e.g. epidermis and inner-most surface). For reference, equilibrium contact angle measurements at the water-untreated wood interface tend to be much higher than bamboo, ranging between 40–85° [26,28–30]; the large variability is due to both inter- and intra-species differences, but also due to factors such as surface roughness, moisture content and extractives content. Nevertheless, for polar liquids the higher water contact angles for wood indicate poorer wettability in comparison to bamboo.

Based on the equilibrium contact angles of water and diiodomethane with the different bamboo surfaces, total surface energy and its polar and dispersive components were determined. These are presented in Table 2. Untreated bamboo had a total surface energy of 68.0 ± 2.1 mJ/m², lower than that of caramelised bamboo (70.7 ± 1.3 mJ/m²) and bleached bamboo (73.9 ± 0.1 mJ/m²). The polarity (ratio of polar component to total surface energy) ranged between 0.37–0.56 for the different bamboo surfaces. Specifically, the processing treatments had a minor influence on the balance of polarity of the substrate, with both caramelisation and bleaching processes increasing the polar component of surface energy and reducing the dispersive component of surface energy (relative to untreated bamboo), with effects being the greatest in the bleaching process. The surface energy of raw bamboo (not its fibres) is not well-reported in literature: Zhang et al. [18] calculate it to be of the order of 40–50 mJ/m², much lower than our measurements. For comparison, untreated woods have total surface energies ranging between 35–85 mJ/m², with the ratio of the polar component to the total surface energy ranging between 0.2 to 1.0 [29,30].

### 3.2. Bonding strength

Fig. 5 and ESI Table 1 present measurements and variation of apparent shear strengths for the various adhesive/bamboo surface interfaces. The datasets are illustrated in Weibull failure probability plots in Fig. 6, which show that the datasets do follow a Weibull distribution, and therefore brittle fracture theory is applicable.

For all adhesives, bleached bamboo interfaces displayed significantly \( (p < 0.005, \text{2-tailed } t \text{-test}) \) higher mean shear strengths than caramelised bamboo interfaces (Fig. 5, ESI Table 1). This was further validated through the differences in the characteristic strength \( \phi_0 \) in the Weibull distribution (Fig. 6), with bleached bamboo interfaces exhibiting higher shear strengths at a failure probability of 63%, and in fact for all failure probabilities. For PVA and phenol formaldehyde-based adhesives (RPF and UPF), untreated bamboo surfaces exhibited intermediate bonding properties to their processed counterparts – lower than bleached bamboo, but higher than caramelised bamboo. Notably, the untreated bamboo material showed comparable bonding strength to bleached bamboo with PU as the adhesive. Nonetheless, improved adhesive bonding strength of bleached bamboo (up to 130%, ESI Table 1) is exciting, in light of observations in literature [10–15] that all other preservative treatment processes reduce bonding strength of the glue line by 15–75%.

The different surfaces exhibited lowest and highest shear strengths with different adhesives: the mean shear strength for i) caramelised bamboo ranged from 2.8 MPa (for PVA) to 5.1 MPa (for PU), ii) untreated bamboo ranged from 2.9 MPa (for Soy) to 8.0 MPa (for PU), and iii) bleached bamboo ranged from 6.7 MPa (for Soy) to 8.5 MPa (for RPF). The soy-flour bio-based adhesive performed poorly in comparison to the other commercial adhesives for all (untreated and treated) bamboo surfaces. Nonetheless, the bonding strength of Soy/bleached bamboo was of an acceptable level at 6.6 ± 1.3 MPa for industrial applications. PVA and Soy produced mostly adhesive failures with all three bamboo surfaces, while the phenol formaldehyde-based adhesives (RPF and UPF) often produce mixed/cohesive or substrate failures with all three adhesives. PU was the only adhesive that showed substantial differences in failure surfaces, with both treated materials having mainly adhesive failures, but the untreated surface having cohesive and even substrate failures. Our observations reveal that the differences in surface chemistry between untreated, bleached and caramelised bamboo not only (generally) lead to lower adhesive bonding strengths for caramelised and untreated bamboo, but also affects their affinity and bonding performance with different adhesives.

The Weibull distribution further revealed that the shape parameter \( m \) was consistently highest for untreated bamboo interfaces (with the exception of Soy adhesive), followed by bleached bamboo interfaces, indicating less variability in strength due to fewer defects and critical flaws, in comparison to caramelised bamboo surfaces. However, we do note that the low \( m \)-values (ranging from 1.5 to 12.2), although not atypical of natural materials (including bamboo fibres and wood) and composites [22,31–34], clearly indicate that there is no ‘true’ strength. Therefore, understanding the spread in shear strength data is important for reliability analysis and quality control in industry. This is the first time Weibull failure probability plots have been constructed for the bonding strength of bamboo materials with different adhesives.

#### 3.3. Linking surface and interface properties to chemical composition

The dry chemical composition of untreated bamboo primarily includes holocellulose (60–70 wt%); 61.2% for our material [35]) – of which 15–25% (19.0% for our material [35]) is accounted for by hemicelluloses – and lignin (20–30 wt%); 22.7% for our material [35] [6,7,17,35]. A small content, typically around 2–4 wt% for Moso bamboo, of low molecular weight extractives, such as waxes and resins, are also present [6,36]. Parenchyma cells in living bamboo are filled with starch, of the order of 2–6 wt% (but even up to 10 wt%), which is an important stored energy resource [7,17]. Preservative treatments aim to chemically decompose the starch or physically limit access to it; an example of the latter is the substantial collapse and shrivelling of parenchyma cells from traditional smoking treatments [7]. However, invariably, treatments also alter other chemical constituents and the physical structure of bamboo.
We have recently studied the effects of caramelisation and bleaching on the chemical composition of bamboo in Sharma et al. [35]. Unlike caramelisation, which is a hygro-thermal treatment, bleaching is a chemical treatment at lower temperatures [8]. We find that the bleaching process results in a more pronounced degradation of lignin in comparison to caramelised bamboo. Due to the mild conditions used (vis. pH, temperature, peroxide concentration), our bleached bamboos have undergone ‘lignin-preserving bleaching’ rather than ‘lignin-degrading bleaching’ [35]. Hence, although the bleaching process leads to cell wall disruption and partial lignin oxidation (of chromophoric groups evidenced by the visual colour change), it is not harsh enough to solubilise or remove the lignin polymers. Caramelisation, on the other hand, has only subtle effects on holocellulose content, and a relatively higher proportion of lignin [35]. We also find that the density of untreated bamboo is lowest (at 562 kg/m³), despite having the highest moisture content [35]. This is also true with bleached bamboo whose density (644 kg/m³) is lower than that of caramelised bamboo (686 kg/m³) [3], despite the former’s higher moisture content. This suggests that untreated bamboo would have the highest porosity content at 60%, 9% and 6% (absolute) higher than that of caramelised and bleached bamboo, respectively.

Based on these changes in chemical composition and structure, differences in surface properties for differently treated bamboo materials can be explained. The high holocellulose and carbohydrate content in untreated bamboo results in a hydroxyl-rich surface with high surface energy. However, the presence of relatively ‘hydrophobic’
extractives, particularly waxes, on the untreated surface, lead to high contact angles with polar liquids (e.g., water), and a relatively larger dispersive component of total surface energy. The contact angle then reduces with time due to spreading of the liquid droplet, particularly along the fibres and porosity vessel channels, and absorption and penetration through the cellular structure, facilitated by pits in the pachyenchyma and fibre cells. Caramelised bamboo has a slightly reduced hemicellulose content, and increased proportion of hydrophobic lignin. Furthermore, the polar units in hemicellulose and lignin may be decomposed. However, hydrophobic extractives, such as surface waxes, are also likely to be reduced. These effects lead to still high (but relatively lower than that of untreated bamboo) contact angles with polar liquids (i.e. poorer wetting), lower polarity ratio, lower surface energy and lower spreading rates. The curious case of comparable (or even better) adhesive bonding strength of untreated bamboo in comparison to caramelised bamboo despite the former’s unfavourable surface properties (higher water contact angle, surface energy (total and dispersive)) can be explained by the inclusion of an intermediate processing step, viz. wiping the bamboo surface with wet paper towel prior to glue application, which we hypothesise diminishes hydrophobic surface waxes, and increases the local moisture content (and polarity ratio). Bleached bamboo bonds better with all the adhesives. This may be because, apart from more thorough removal of extractives possible through chemical treatment, degradation of hydrophobic lignin increases the polarity ratio, reduces contact angles, improves spreading rate and results in a higher surface energy (than caramelised bamboo) and more favourable chemical interactions. In addition, the chemical treatment may be increasing surface roughness, permeability and porosity (by breaking down material, including pit membranes [37]) and therefore facilitating penetration of the adhesive and mechanical interlocking. Our hypothesis on mechanical interlocking is supported through evidence from qualitative inspection of fractured lap-joints: bleached bamboo had a significantly higher percentage of mixed/cohesive and substrate failures than both untreated and caramelised bamboo.

4. Conclusions

To optimise industrial manufacturing and material performance of products such as laminated bamboo, it is imperative to understand processing-property relations specific to bamboo. Our study finds that commercial preservative treatments have significant effects on the wetting behaviour and adhesive bonding performance of the laminated bamboo material. Specifically, bleached bamboo exhibits better wettability, higher surface energy and better liquid penetration than both untreated and caramelised bamboo. This translates to higher adhesive bonding strengths for bleached materials for all adhesives tested here. Improved adhesive bonding strength of bleached bamboo (up to 130%) is exciting, in light of observations in literature that all other preservative treatment (bleaching) produce these changes in surface and interface properties.

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Data accessibility

The datasets supporting this article have been uploaded as part of the Supplementary Material. Additional data is available at Cambridge University’s Data Repository: https://doi.org/10.17863/CAM.22798.

Appendix A. Supporting information

Supplementary data associated with this article can be found on the online version at https://doi.org/10.1016/j.jjadhal.2018.05.009.

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