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ORIGINAL

Quality assessment and mechanical characterization of preservative-treated Moso bamboo (*P. edulis*)

Christian Gauss[1,2] · Kent A. Harries[2] · Marzieh Kadivar[1] · Yusuf Akinbade[2] · Holmer Savastano Jr.[1]

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Abstract

Bamboo has been in the focus of attention as a re-discovery of an old and available material to solve environmental problems in the construction industry. The use of full-culm bamboo in the built environment, however, depends on proper quality control/assurance of its mechanical and physical properties. In this work, a quality assessment in terms of treatment control and mechanical properties of a small production of *Phyllostachys edulis* bamboo poles treated with disodium octaborate tetrahydrate (DOT) was performed. A comparison between two commercial preservatives used for exterior and interior applications, chromated copper borate (CCB) and DOT respectively, in terms of the effect on the mechanical properties and treatability behaviour was also investigated. Penetration and retention analyses showed satisfactory results for the samples treated with CCB, with retention of 7.2 kg/m^3, while lower values of retention for the samples treated with DOT by the immersion method (2.2 kg/m^3) was observed. Microstructural and EDS analyses revealed a much higher concentration of chromium and copper from the CCB solution in the bamboo large vessels. The mechanical characterization performed by compression, shear, tension, coupon three-point bending, and flat ring flexure tests showed that the difference between the two treatment conditions was small and, in most cases, not statistically relevant. Low coefficients of variation were observed in all the investigated mechanical tests, suggesting a uniform distribution of mechanical properties within the batch of *P. edulis* bamboo used in this study. The full characterization schedule combined with digital image correlation analyses enabled the calculation of the characteristic values of the mechanical properties, useful for structural design, complementing the treatability and quality assessment.

1 Introduction

Bamboo as an engineered natural material is increasingly being explored for structural uses in construction (e.g., Anuar and Krause 2016; Chow et al. 2019). Traditionally used for centuries for (so-called) informal or vernacular building construction, furniture, and daily necessaries (Zhang et al. 2018), bamboo today has expanded into modern construction techniques. Extensive research on the generally good mechanical properties of bamboo is presented in the literature (e.g., Dixon and Gibson 2014; Jakovljević et al. 2017; Akinbade et al. 2019). Fast- growing and maturing bamboo species such as *Phyllostachys edulis* (Moso) produce material with promising structural properties.

Without suitable treatment, however, bamboo is prone to biological degradation in a short period of time, reducing its utility as a structural material (Janssen 2000). The "bamboo borer" or "powderpost" beetle (*Dinoderus minutus*) is a primary destructive agent of bamboo (Watanabe et al. 2015) and is present across the world's tropical zones (CABI 2019). Other xylophagous organisms, such as decay fungi and termites, can also affect the structural integrity of bamboo and consequently compromise the service life of structures (Jayanetti and Follett 2008; Tiburtino et al. 2015b). Today, conventional wood treatment solutions used in Brazil have good performance but are typically based on heavy metals and other toxic elements, such as CCA (chromated copper arsenate), pentachlorophenol and others (Mohajerani et al. 2018). Whenever technically and economically feasible, replacement of hazardous chemicals with less hazardous substances is an essential objective in the wood and bamboo industry. As a result, novel preservative

✉ Christian Gauss
gausschr@usp.br

[1] Department of Biosystems Engineering, University of São Paulo, Pirassununga, SP, Brazil

[2] Swanson School of Engineering, University of Pittsburgh, Pittsburgh, PA, USA

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formulations are being developed and used for interior and exterior applications.

Less hazardous substances that have been investigated and commercialized for the treatment of bamboo and wood for interior application include low-cost soluble salts, such as boron-based salts, specifically disodium octaborate tetrahydrate (DOT), boric acid and borax (Caldeira 2010; Kim et al. 2011; Liese and Tang 2015; Tiburtino et al. 2015b). Boron compounds are some of the most effective and versatile preservative solutions used today since they combine broad-spectrum efficacy, low mammalian toxicity, and are odourless and colourless (Tondi et al. 2012; Donmez Cavdar et al. 2015; Jit Kaur 2018; Zhou et al. 2018). Preservation with boron compounds can even improve the quality of bamboo, improving some mechanical properties in comparison with bamboo without preservatives in samples with high retention levels (Prinindya and Ardiansyah 2014; Sulaeman et al. 2018; Gauss et al. 2019a). Nevertheless, the use of boron compounds presents restrictions for the treated material because of the leaching of boron in the presence of water, making it unsuitable for the use in exterior applications (Hidalgo-López 2003; BIS IS1902 2006; Freeman et al. 2009; Caldeira 2010). For the exterior use, CCB (chromated copper borate) was developed as an alternative to CCA, substituting arsenic with a boron source, reducing the toxicity to humans and the environment (Vidal et al. 2015; Beraldo 2016). Nonetheless, heavy metals are still used in its composition and the disposal of CCB-treated wood/bamboo continues to be a problem (Caldeira 2010).

From a structural engineering perspective, the challenge is to prescribe a treatment having sufficient retention and penetration in the culm to increase the bamboo service life without sacrificing physical or mechanical properties. Although chemical treatment is commonly used in the construction industry, its impact on material properties is often unclear. Recent investigations have demonstrated that the treatment method selected affects the mechanical properties of laminated bamboo material (Shah et al. 2018). The effects of preservative treatments such as steam, oil or dry heat treatment have been found to have adverse effects on both wettability and strength of the bamboo product (Wahab et al. 2005, 2015; Sulaiman et al. 2006; Li et al. 2015; Bui et al. 2017). Therefore, not only is a quality control assessment of the final structural material necessary but also the effects of chemical treatments on the mechanical properties of treated materials require closer inspection.

Most structural projects utilising bamboo in Latin America and Asia use boron compounds (boric acid, borax or DOT) for bamboo exposed to a protected environment (typically referred to as interior exposure) and CCB or CCA for exterior exposure. Despite this dichotomy, there is no known design practice that differentiates the structural design of differently treated bamboo. Indeed, there are no known studies of the treatability, mechanical performance and durability of differently treated bamboos. The present study aims to investigate the effects of DOT and CCB treatments—as the two most well-known commercially used preservatives—on treatability and mechanical properties of *P. edulis* bamboo (the most widely commercialized bamboo species). The methods of treatment applied to each material follow the same practical methods applied in the industry. Bamboo for exterior exposure is treated using CCB in a vacuum pressure process while bamboo for interior exposure is treated using DOT in an immersion method.

2 Materials and methods

2.1 Material

Approximately 140 *Phyllostachys edulis* (Moso) bamboo culms were obtained from a supplier near Sao Paulo, Brazil. Culms between 3 and 5 years of age were harvested, from which 4–4.5 m long poles (visually free of defects) were extracted. The diameters ranged from 70 to 90 mm, wall thickness from 6.5 to 10 mm and the oven-dry density prior to treatment was 760 kg/m^3.

The culms were divided into two batches (Fig. 1). Batch A comprised 130 poles treated with DOT by immersion (see below). From this large batch, twelve 1 m long samples were extracted from randomly selected poles for the evaluation of mechanical properties and boron penetration analysis. An additional two samples were extracted to assess boron retention following 7- and 10-days' immersion. This series of samples was intended as a means of quality assessment of the entire batch of bamboo and is indicated in this paper as "A-DOT".

A second smaller batch B was used for direct comparison of CCB and DOT treatment. Adjacent 0.8 m long samples were extracted from untreated poles. The adjacent samples were then treated using CCB or DOT. Using adjacent samples in this way was intended to minimize the variation in the mechanical properties from different poles and along the length of the same pole, permitting a direct comparison of the effects of the treatment method. The samples intended for comparison are indicated B-DOT and B-CCB in this paper.

2.2 DOT treatment

Agricultural grade disodium octaborate tetrahydrate (DOT; $Na_2B_8O_{13}$•4.H_2O, molar weight of 412.5 g/mol) supplied by Sulboro (Brazil) was used. A-DOT and B-DOT samples were treated by immersion in an 8% (weight/volume) DOT aqueous solution. The 4.7 m long immersion tank is

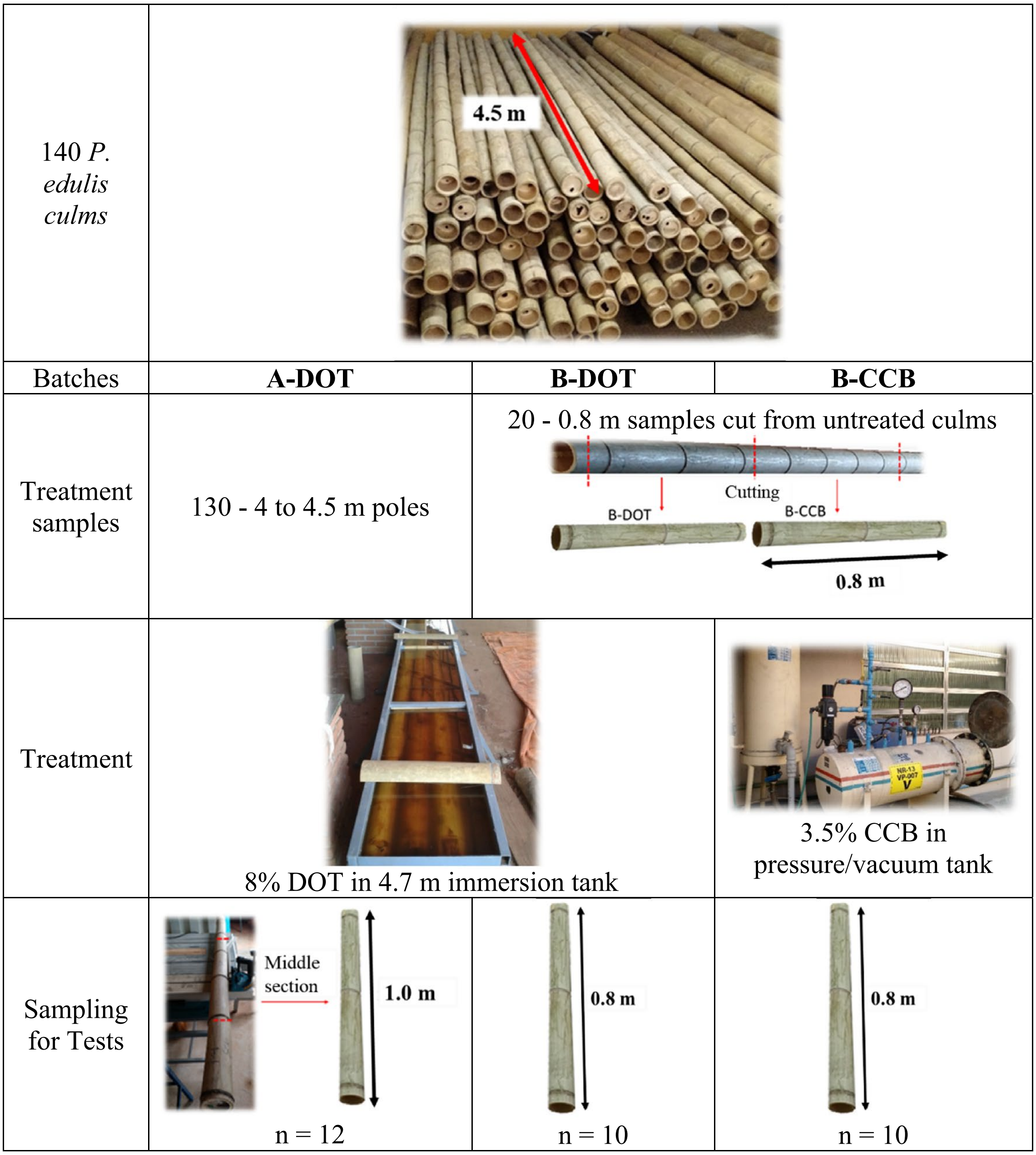


Fig. 1 Sampling and treatment methods

shown in Fig. 1. A small amount of tannin extract supplied by Tanac (Brazil), was also added to the solution (0.5 kg per 1000 L) in order to facilitate the cleaning (and thereby reuse) of the solution and as an additional fungicide agent. The solution was conditioned, and the concentration adjusted after each treatment batch using a conductivity meter (according to a standard concentration curve). The A-DOT samples were kept in immersion between 7 and 10 days (depending on the batch) and the B-DOT samples were immersed for 7 days.

2.3 CCB treatment

B-CCB samples were treated using commercially available chromated copper borate (CCB), MOQ OX 50, supplied by Montana Química Ltda (Brazil). The product is an oxide-based CCB having approximately the following constituency of active ingredients: 32% CrO_3; 13% CuO; and 5% B (as trivalent boron); and 50% inert ingredients. The molar weight of the three active constituents is 100, 79 and 10.8 g/mol, respectively. A 3.5% (active ingredient weight/volume) aqueous solution was used in a pressure vessel using a full-cell process: − 600 mmHg (0.8 bar) vacuum for 30 min, followed by 10 kgf/cm^2 (10 bar) pressure for 60 min, followed by − 600 mmHg vacuum for 15 min. Treatment was conducted in a pressure/vacuum tank shown in Fig. 1.

2.4 Treatment characterization

2.4.1 Retention and penetration analysis

Following treatment, samples were subjected to boron (in case of DOT) and chromium, copper and boron (in case of CCB) retention analyses conducted according to Brazilian standard ABNT NBR 6232:2013 (2013) (Penetration and retention of preservatives in pressure treated wood) and Indian standard BIS 1902 (2006) (Preservation of bamboo and cane for non-structural purposes). For the chemical analyses, samples extracted from the middle part of each pole were subjected to (sulphuric) acid digestion, diluted and analysed by atomic absorption spectroscopy. The samples treated with DOT were analysed at IPT (Technological Research Institute, São Paulo) and the samples treated with CCB at Montana Química (São Paulo).

Penetration analysis was also performed in accordance with ABNT NBR 6232:2013 (2013) to observe the presence of boron (for DOT treated samples) and copper (for CCB treated samples). The cross-sectional area of samples extracted from the central region of the treated poles was reacted with the etching solutions:

For boron penetration analysis, a solution composed of curcumin (earth turmeric) and ethyl alcohol (10% wt/vol alcohol) was applied to the treated bamboo section and permitted to dry. Then, a saturated salicylic acid alcoholic solution (13 g per 100 mL solution) and 20 mL of concentrated hydrochloric acid were applied. The observation of red colour indicates the presence of boron. For copper penetration analysis, a solution with 0.5 g of chrome azurol S and 5.0 g of sodium acetate in 300 mL of water is applied; a dark blue colour indicates the presence of copper.

2.4.2 Optical and scanning electron microscopy

The transverse section of the treated bamboo samples was analysed in a ZEISS Smartzoom 5 optical microscope in order to evaluate observable effects of the different treatment conditions. For the analysis, small samples were cut with a diamond disc and subjected to fine grinding and polishing with (sequentially) 15, 3 and 1 µm diamond polishing paste. After polishing the samples were cleaned with isopropyl alcohol and dried at room temperature.

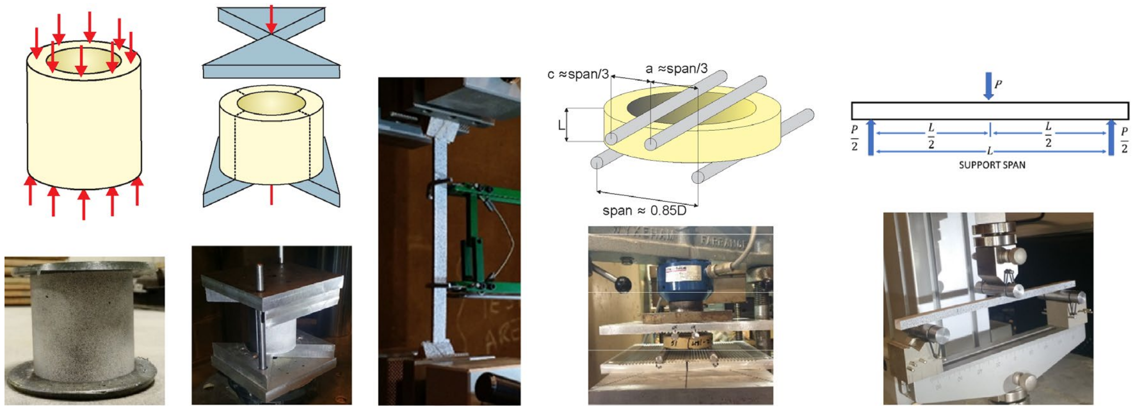
Clean cuts of the longitudinal section of the bamboo samples (parallel to the fibres), obtained using a sharp chisel and subjected to no further surface preparation, were used for microstructural and chemical characterization in a FEI Apreo scanning electron microscope (SEM) equipped with a field-emission gun. This procedure was performed in order to preserve the chemicals within the bamboo structure for chemical analysis. Prior to the analysis, the samples were coated with palladium in a Cressington Sputter Coater. Elemental mapping was performed using energy-dispersive X-ray spectroscopy (EDS) operated at 20 kV.

2.4.3 Mechanical characterization

Mechanical characterization of the samples treated with DOT and CCB was performed according to procedures described below. Full culm specimens were used for compression, shear and flat ring flexure tests, while machined coupon specimens were used for tension and bending tests. All required specimen dimensions were obtained using a digital calliper having a precision of 0.01 mm. Digital image correlation (DIC) techniques were used in all tests to determine strain fields and thereby modulus. DIC is a well-established contact-free means of obtaining full-field surface deformations (and therefore strains). Specimens are painted with a speckle pattern prior to testing (photocopier toner broadcast onto wet white spray paint, the result is seen in Fig. 2). During the test, consecutive high-resolution images (2448×2049 pixels) are taken every 0.5 s. and deformation patterns (based on sampling of the speckle pattern) are recorded. Post-processing allows relative displacements and specified strain fields to be obtained in three dimensions. The system used in this study is a VIC-3D dual camera system resulting in a resolution better than 1 microstrain on the surface of the specimens. All the obtained data was processed and analysed using the VIC-3D 2012 Digital Image Correlation software (Correlated Solutions). Sample was weighed prior to testing and afterward dried at 100 °C ± 2 for at least 48 h to establish moisture content (MC) in accordance with ISO 22157:2019 2019. Additional description and commentary on the mechanical test protocols are reported in Gauss et al. (2019b).

2.4.4 Compression parallel to fibres

Full-culm compression tests (Fig. 2a) were conducted according to ISO 22157:2019 2019. Specimens had a height equal to their nominal diameter (i.e., L = D). A sulphur capping compound was used in order to ensure a flat loading surface and reduce the friction between the sample and the compression platen. Tests were performed in a 600 kN capacity universal testing machine; load was applied at a crosshead displacement rate of 1.0 mm min^{-1}. Compression modulus, E_c, is determined from DIC analysis.



(a) ISO 22157 compression test

(b) ISO 22157 shear test

(c) ISO 22157 tension test

(d) flat ring flexure test (Virgo et al. 2017)

(e) small coupon three-point bending test (ASTM D7264)

Fig. 2 Mechanical test methods

2.4.5 Shear parallel to fibres

Full-culm shear tests (so-called "bowtie" tests) were conducted according to ISO 22157:2019 2019; also with specimens having a height equal to their nominal diameter (i.e., L = D). In this test (Fig. 2b), the full culm specimen is supported at its lower end over two opposing quadrants and loaded at its upper end over the other two opposing quadrants. In this manner, loading the specimen results in four shear areas. The test is controlled by the first shear plane to fail and therefore the shear strength, f_v, is interpreted as the lower bound shear strength. Tests were performed in a 600 kN capacity universal testing machine; load was applied at a crosshead displacement rate of 1.0 mm min^{-1}. Shear modulus, G, is determined from DIC analysis.

2.4.6 Tension parallel to fibres

Tension tests were performed in accordance with ISO 22157:2019 2019, with some modifications of the specimen design. Radially oriented bamboo strips, 200 mm in length, were extracted from sample poles. The samples were sanded to obtain uniform dimensions with a breadth (b) less than half of the culm wall thickness (t). Softwood tabs were glued on the specimen ends in order to facilitate gripping by the testing machine. Flat samples often exhibited failures associated with grip inconsistencies and/or stress raising effects; for an accurate comparison of the treatment methods, these failures must be mitigated (in practice, grip failures are neglected—this is not possible in this study due to the limited number of samples available). A modified "dog bone" specimen was produced with a region of reduced breadth in the middle of the specimen. The tensile modulus of elasticity of the dog bone samples was validated comparing modulus values obtained from flat samples using both a mechanical extensometer and DIC (Fig. 2c). Only specimens without nodes are considered in this paper. Tests were conducted in a 600 kN capacity universal test machine at a displacement rate of 1.0 mm/min.

2.4.7 Three-point small coupon bending test

The bending test perpendicular to the fibres for bamboo described by ISO 22157:2019 2019 requires specimens having a length L > 30D. This test is intended as a component capacity test, not a materials evaluation test. Since the main objective of this work is to evaluate the influence of the preservative treatments on the mechanical properties of bamboo, reduced size specimens in prismatic form with 200 mm long × 10 mm wide x culm wall thickness depth were used (Fig. 2e). Only specimens without nodes are considered in this paper. A span of 160 mm was used for all the tests, which resulted in an average shear span to depth ratio exceeding 10 in every test. The three point bending tests were conducted following Procedure A of ASTM D7264 (2015) using a 10 kN capacity electromechanical testing machine. A displacement rate of 2.5 mm/min was used for all the tests. Tests reported in this study were conducted with the sample orientated such that the outer culm wall was in compression (OC) (see Gauss et al. 2019b for additional discussion of specimen orientation). Modulus of rupture (MOR) and modulus of elasticity (MOE) were calculated according to ASTM D7264.

2.4.8 Flat ring flexure test

In order to evaluate the mode I fracture behaviour of treated bamboo samples and better understand the splitting behaviour, a flat ring flexure test was performed (Virgo et al. 2017; Akinbade et al. 2019).

Samples with $0.18D \leq L \leq 0.22D$ were cut from the 0.8 m bamboo poles and the dimensions were measured using a digital calliper at four quadrants of the specimen. The flat-ring flexure test was conducted in a four-point bending setup (Fig. 2d) using a displacement rate of 0.76 mm/min (0.03 in/min) in a 45 kN mechanically driven testing machine equipped with a load cell having a precision of ± 0.4 N. In the symmetric specimen, only circumferential stresses are present and the modulus of rupture is calculated only for samples that fail in the constant moment region (dimension “c” in Fig. 2d) (Virgo et al. 2017).

2.4.9 Statistical analyses

The averages of each test are presented with the corresponding coefficient of variation (COV) and number of samples. The differences between the treatment conditions in the mechanical properties were checked by a Tukey’s test and analysis of variance (ANOVA) for significant ($p < 0.05$) differences. All analyses were performed using MINITAB® Release 18 Statistical Software.

3 Results and discussion

3.1 Treatment characterization

Active ingredient penetration and retention analysis results are summarized in Table 1 and discussed in the following sections. Samples for treatment characterization were extracted from the middle part of the treated bamboo poles (away from the cut ends), which represents the region most susceptible to lower retentions.

3.1.1 Active ingredient penetration

Penetration analysis provides a qualitative measure of the efficacy of the treatment process and enables visualization of where the chemicals used in the treatment are located within the culm wall thickness. Depending on the degree of active ingredient penetration across the wall thickness (i.e., area reacting with the etching solutions), a grade between 0 and 4 can be assigned to each sample: 0 = no penetration; 1 = 0–25% penetration; 2 = 25–50%; 3 = 50–75%; and, 4 = greater than 75% penetration (Kim et al. 2011). Examples of penetration grades for boron are shown in Fig. 3.

Table 1 presents a summary of treatment parameters and penetration grade of each condition. For the A-DOT samples used for quality assessment, samples from 10 different poles

Table 1 Summary of treatment methods and assessment

	A-DOT	B-DOT	B-CCB
Sample size, n	12	10	10
Treatment method	7- or 10-day immersion	7-day immersion	vacuum/pressure
Active ingredient and nominal concentration	8% DOT solution	8% DOT solution	3.5% CCB solution
Moisture content before treatment (%)	30.0	14.1	17.0
Weight gain following treatment (%)—(COV in parentheses)	–	17.0 (0.31)	30.2 (0.10)
Retention of active ingredient (kg/m^3)	2.2	2.2	7.2
Penetration grade	Range from 2 to 4 (10 samples)	3 (single sample)	4 (single sample)

Fig. 3 Boron penetration analysis of samples treated with DOT (untreated reference sample at left). Grade 4 (> 75% penetration); Grade 3 (50–75% penetration); Grade 2 (25–50% penetration)

were analysed resulting in penetration grades ranging from 2 to 4 (average = 3). For the B-DOT and B-CCB treatments, a single sample per condition was used resulting in penetration grades of 3 and 4, respectively.

3.1.2 Active ingredient retention

Active ingredient retention is assessed as mass retention of the chemical components of the treatment as given by Eqs. 1 and 2.

$$\text{For DOT} \quad \text{Total retention}\ \left(\text{kg/m}^3\right) = \text{Na}_2\text{B}_8\text{O}_{13} \cdot 4 \cdot \text{H}_2\text{O}\ \left(\text{kg/m}^3\right) \tag{1}$$

$$\text{For CCB} \quad \text{Total retention}\ \left(\text{kg/m}^3\right) = \text{CrO}_3\left(\text{kg/m}^3\right) + \text{CuO}\ \left(\text{kg/m}^3\right) + \text{B}\ \left(\text{kg/m}^3\right) \tag{2}$$

The samples treated with DOT exhibited lower retention levels (2.2 kg/m^3) mainly because this treatment is by "passive" immersion, rather than by "active" vacuum or pressure. Using pressure treatment for boron-based (BB: Boric acid + Borax) solutions, Kim et al. (2011) also observed lower retention levels in comparison with CCB for *B. stenostachya*, *T. siamensis* and *D. asper* bamboos. Values between 2.5 and 15.5 kg/m^3 were observed for BB proportional to the applied pressure (2.5–8.5 bar) and greater when the bamboo epidermal layer had been removed (an advantage for solution absorption). As reference values, the Indian standard BIS IS9096 (2006) recommends 6 kg/m^3 of active ingredient (borax + boric acid treatment, with the same proportion used for the formation of DOT) for indoor applications. A retention of 2.7 kg/m^3 of B_2O_3, equivalent to 4.0 kg/m^3 of DOT, is recommended by the American Wood Preservers's Association (AWPA) for boron-based treatments (Caldeira 2010). Although boron-based treatments are widely used for structural use of bamboo, information regarding retentions values is scarce and affected by treatment methods and bamboo species (Tiburtino et al. 2015a; Kim et al. 2011).

The A-DOT and B-DOT samples presented similar DOT retention values. Although the A-DOT samples were extracted from 4.5 m poles (about 2 m to a cut end), and B-DOT samples were extracted from 0.8 m poles (no more than 0.4 m to a cut end), the length of the bamboo poles did not affect the retention of DOT. Furthermore, no difference was noticed for A-DOT samples from poles treated for 7 and 10 days in terms of retention or penetration.

The CCB-treated samples exhibited retention of 7.2 kg/m^3, higher than that observed by Tiburtino et al. (2015a) in *D. asper* and *B. vulgaris* bamboo samples treated by immersion and by modified Boucherie methods (Tiburtino et al. 2015a). For CCB treatments, the Indian standard BIS IS401 (BIS 2001) recommends retentions values of 10–16 kg/m^3 for applications exposed to weather and in contact with the ground, 6–10 kg/m^3 for applications exposed to weather but without ground contact, and 6 kg/m^3 for applications undercover. Kim et al. (2011) reported values between 11.3 and 16.3 kg/m^3 retention of CCB in samples treated with similar pressure (8.5 bar) to that used in this work. However, in their work they used a 6% CCB solution and no information regarding the density of the material is reported (which can also greatly influence the treatability of bamboo). Baysal et al. (2016) reported that bamboo (*P. bambusoides*) presented lower retention values of several preservatives (CCB, boron, and other copper-based products) than those observed in wood (Scots pine), which was attributed to the anatomical characteristics of bamboo. The same behaviour was observed in the work by Lee et al. (2001), using the same bamboo species as in the present work, but treated with CCA instead of CCB.

3.2 Microstructural characterization and chemical analysis

Optical and scanning electron microscopy (SEM) were used to analyse the microstructure of the treated bamboo samples and investigate any differences between CCB and DOT treatments.

A typical microstructure obtained by optical microscopy of the bamboo used in this study is shown in Fig. 4, in which its structure composed of parenchyma (P), fibre bundles (F), phloem (Ph) and vessels (V) is shown in detail. Using ImageJ analysis software (Rasband 2018), the fibre volume ratio of the *P. edulis* samples was determined. Sixteen images extracted from four randomly chosen culms resulted in determining a fibre volume content, $V_f = 28.8\%$ (COV = 0.07). This value is similar to that reported in a number of other studies (Akinbade et al. 2019).

Optical microscope images of CCB- and DOT-treated samples (Fig. 5) show that there is no visual difference in terms of microstructure between treatments, especially around the vessels, where an effect might be expected.

Using SEM for the evaluation of a section parallel to the fibres it is also possible to identify the main bamboo microstructural elements, i.e. parenchyma, vessels and fibre bundles, as shown in Fig. 6. In this image, the structure of the vessel, including the pit openings on the inner surface of the vessel, can be clearly observed. The SEM images of CCB- and DOT-treated samples also do not show any visual difference in the parenchyma cells close to the vessel (Fig. 6).

Elemental analysis using energy dispersive spectroscopy (EDS) was performed to evaluate the distribution of the active ingredients through bamboo's microstructure. This technique has some limitations and is unable to detect light elements such as boron and sodium. Therefore, only the samples treated with CCB could be characterized. Figure 7 shows an elemental map and the corresponding tables of the

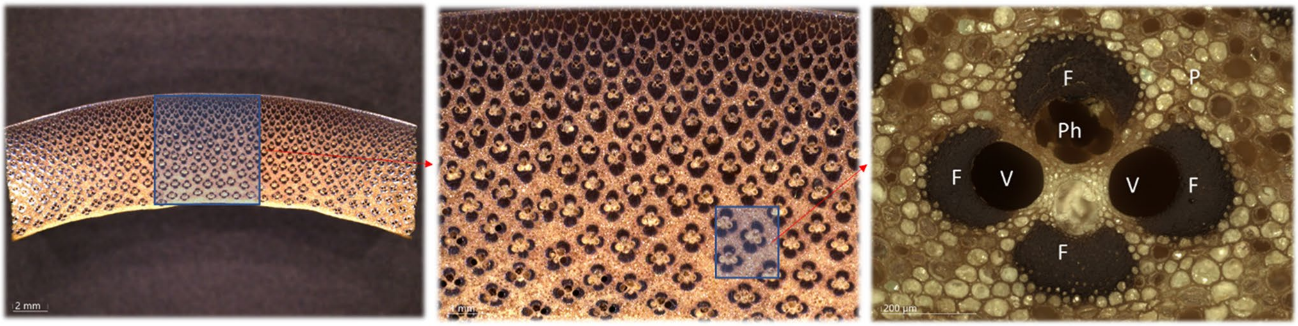


Fig. 4 Microstructure of *P. edulis* bamboo used in this study. *V* vessels, *F* fibre bundles, *Ph* phloem, *P* parenchyma

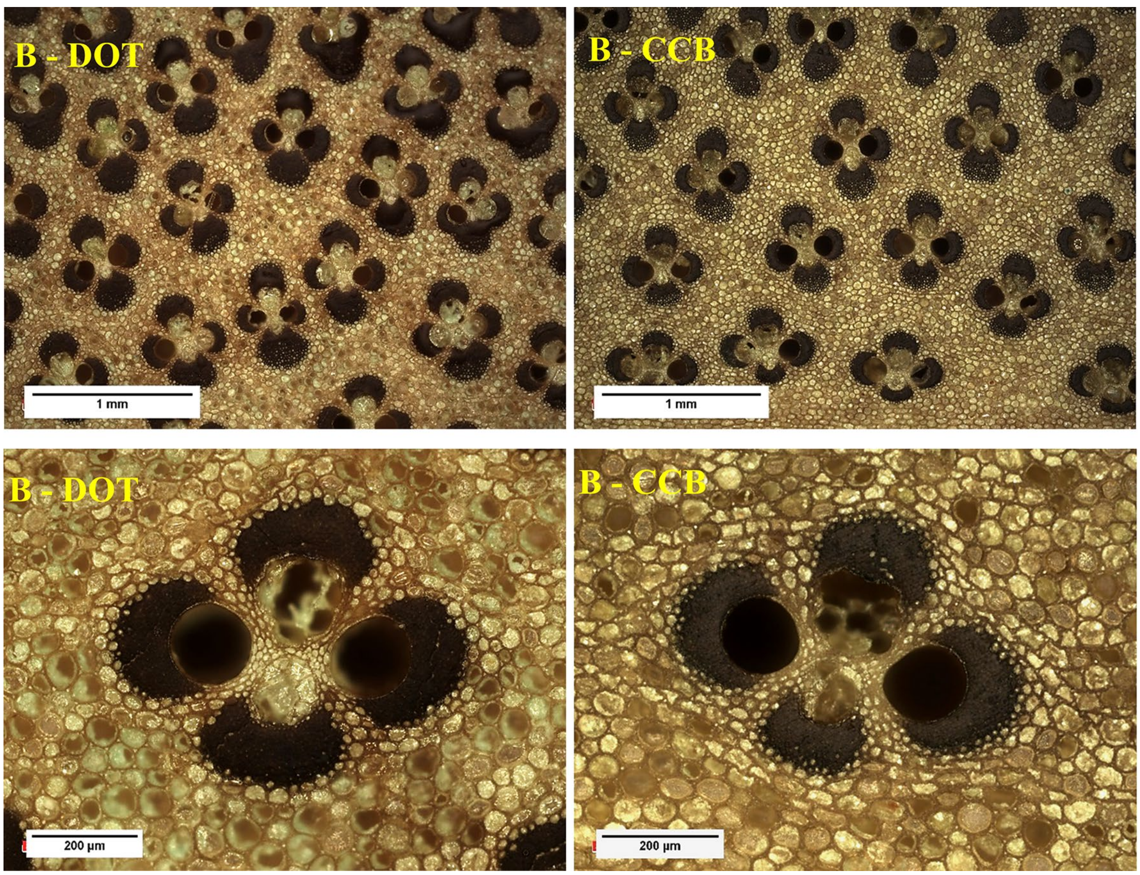


Fig. 5 Optical microscopy images of the B-DOT and B-CCB samples

semi-quantitative analysis of regions composed of parenchyma, vessels and fibres. It can be seen that most of the chromium and copper, in the form of CrO_3 and CuO, is concentrated in the large vessels (point 1 in the upper image). The entire analysed area of this image showed atomic weights of chromium and copper of 1.56% and 1.89%, respectively, whereas for the large vessel much higher values (Cr = 12.47% and Cu = 17.86%) were observed. Only traces of the elements were found in the fibres and parenchyma (points 2 and 3). The same effect can be observed in the lower image of Fig. 7. Although the atomic weights of chromium and copper in the entire area are 3.49% and 4.05%, respectively, the two large vessels (points 1 and 2) presented significantly higher values of these elements. In this image, the phloem (point 3) presented similar values of chromium and copper in comparison with the entire area, but significantly lower than the large vessels. The phloem consists of large thin-walled sieve tubes with small cells and it is used

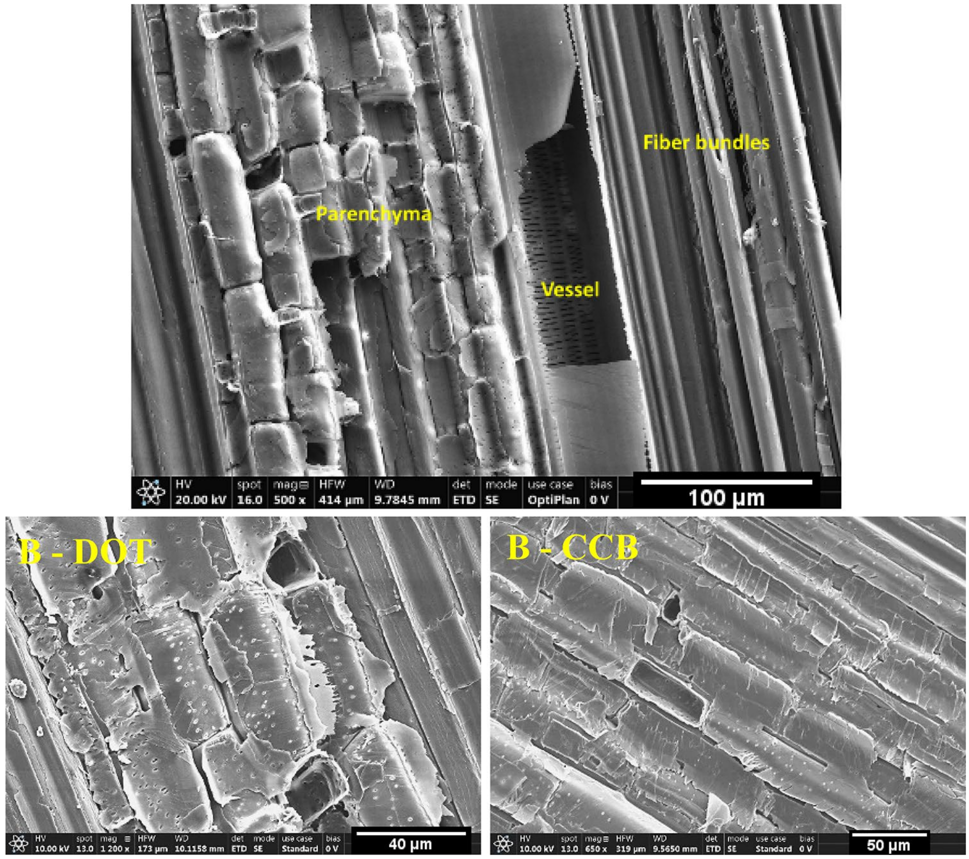


Fig. 6 SEM image of the longitudinal section parallel to the fibres of *P. edulis* bamboo showing its main constituents (upper image) and the parenchyma region of DOT- and CCB-treated samples (lower images)

for the conduction of carbohydrates (instead of water that is conducted in the large vessels) (Liese 1987). It is assumed that the presence of carbohydrates can affect the penetration of the active ingredients in the phloem.

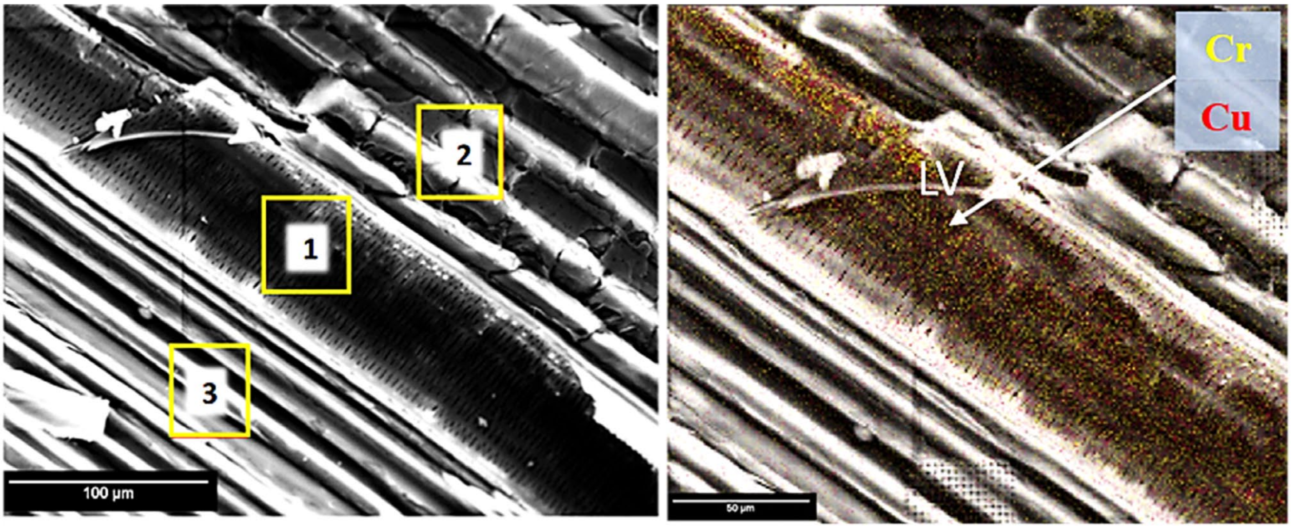
Although satisfactory retention levels were obtained in the CCB-treated samples, there is a heterogeneous distribution of the active ingredients that cannot be detected by the penetration or retention tests. This poor distribution is explained by the low mobility of large and heavy elements such as chromium and copper in the bamboo microstructure. Additionally, there are no pathways for radial penetration in bamboo, like the rays in wood. The metaxylem vessels of the vascular bundles are the main path for penetration and access to the parenchyma is difficult (Liese 2004; Liese and Tang 2015).

The bamboo borer beetle (*Dinoderus minutus*), one of the main insects responsible for bamboo deterioration, lays its eggs on metaxylem vessels (Garcia and Morrell 2009; Watanabe et al. 2015). Because the active ingredients are concentrated in the vessels, larval growth of the beetle is expected to be affected by the copper and chromium elements found in these regions and hence, prevent further insect infestation or new attacks. However, this assumption still needs to be validated in a controlled experiment to determine whether the concentration of active ingredients in the vessels prevents larval growth.

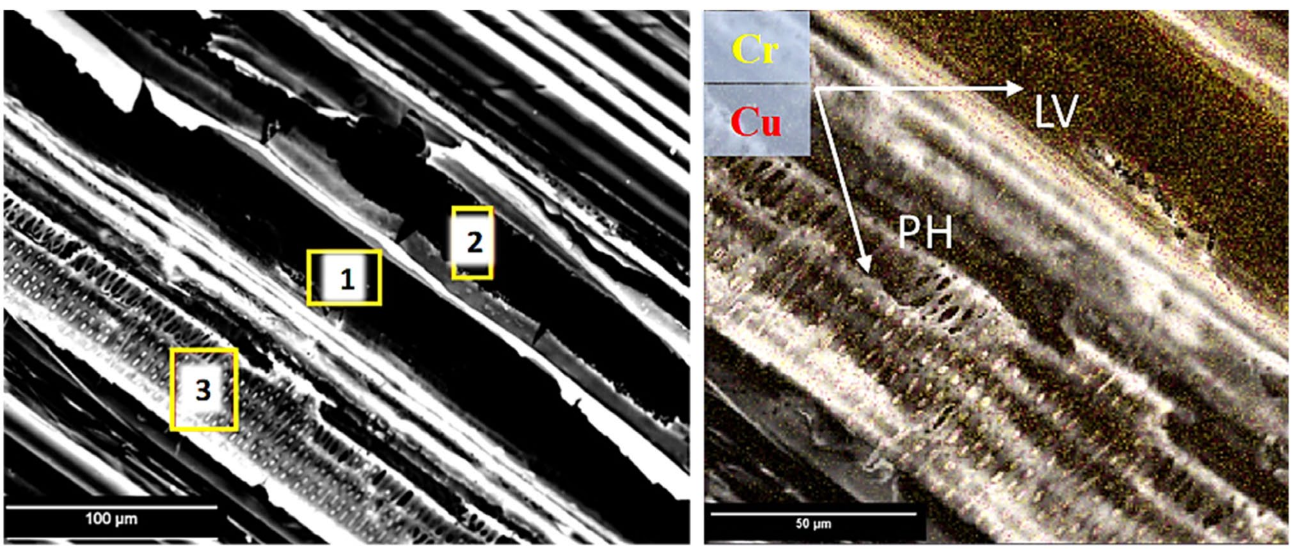
3.3 Mechanical characterization

Results of the mechanical characterization tests are presented in Table 2 and summarized in Fig. 8. In addition to strength (f) and modulus (E and G), the limit of proportionality (LOP) is reported. This value describes the stress at which the material ceases to behave in a linear manner having the modulus shown.

As seen in Table 2, there is little difference between A-DOT and B-DOT beyond the normal variation expected in bamboo material properties. An ANOVA analysis of the results of B-DOT and B-CCB indicates no significant (defined at 95% confidence) difference between the DOT- and CCB-treated samples in most mechanical properties. Only in compression (f_c and LOP) and shear (f_v) a p value < 0.05 was observed. These differences, however, are more a reflection of the relatively small coefficients of variation seen in this study than a real difference in material strength; this is seen in Fig. 8. In fact, analysing the LOP in compression, shear and bending, the values obtained for B-DOT and B-CCB are practically the same.



Element	Entire area		1 large vessel (LV)		2 parenchyma		3 fibre bundle	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C	55.25	63.53	45.26	64.8	60.79	67.85	60.97	67.65
O	41.29	35.64	24.41	26.24	38.00	31.84	38.76	32.28
Cr	1.56	0.41	12.47	4.13	1.22	0.31	0.15	0.04
Cu	1.89	0.41	17.86	4.83	-	-	0.12	0.03



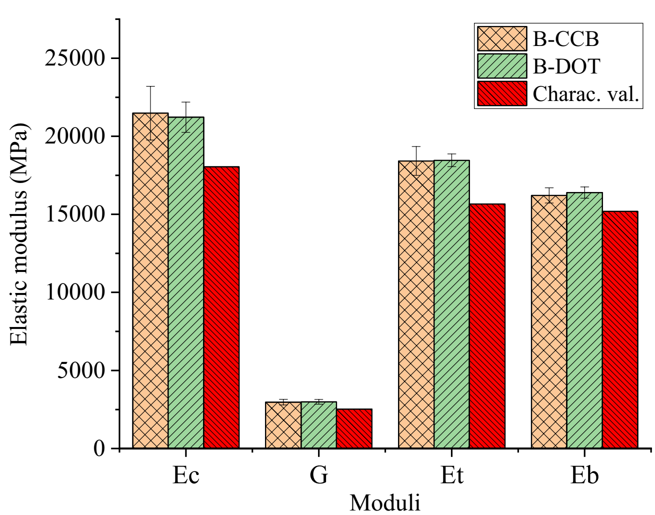
Element	Entire area		1 large vessel (LV)		2 large vessel		3 phloem (PH)	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C	52.85	62.80	34.93	57.03	21.77	49.59	57.24	66.66
O	39.61	35.34	23.52	28.82	10.90	18.63	36.35	31.78
Cr	3.49	0.96	19.33	7.29	29.21	15.37	2.99	0.80
Cu	4.05	0.91	22.22	6.86	38.13	16.42	3.42	0.75

Fig. 7 EDS mapping of a sample treated with CCB showing the higher concentration of chromium-Cr (dots in right image) and copper-Cu (dots) in the large vessels (LV) and phloem (PH) of the bamboo structure

Table 2 Summary of experimentally determined material properties (COV in parentheses)

Standard/Properties		A-DOT	B-DOT	B-CCB	B-DOT B-CCB *p*-value	A-DOT + B-DOT + B-CCB	Characteristic value
Density (kg/m^3)		0.80	0.80	–	0.79	0.80	–
Compression//fiber (ISO 22157:2019 2019)	n	24	15	16	–	55	–
	MC (%)	9.9 (0.04)	10.2 (0.02)	10.4 (0.03)	–	–	–
	f_c (MPa)	59.8 (0.10)	54.9 (0.06)	58.1 (0.07)	0.030	57.9 (0.08)	49.5
	E_c (MPa)	19,070 (0.11)	21,220 (0.09)	21,480 (0.08)	0.690	20,380 (0.10)	18,040
	LOP (MPa)	50.9 (0.12)	49.1 (0.06)	52.9 (0.08)	0.039	50.7 (0.10)	41.5
Shear//fiber (ISO 22157:2019 2019)	n	25	9	15	–	49	–
	MC (%)	10.6 (0.03)	9.7 (0.01)	9.9 (0.02)	–	–	–
	f_v (MPa)	17.5 (0.08)	19.6 (0.05)	18.1 (0.06)	0.003	18.0 (0.08)	15.4
	G (MPa)	2710 (0.10)	2990 (0.10)	2970 (0.06)	0.550	2850 (0.10)	2520
	LOP (MPa)	12.2 (0.09)	12.1 (0.08)	12.6 (0.11)	0.703	12.2 (0.09)	10.0
Tensile//fiber (ISO 22517:2019 2019)	n	20	19	18	–	57	–
	MC (%)	6.8	6.9	6.8	–	–	–
	f_T (MPa)	247 (0.08)	283 (0.07)	292 (0.06)	0.466	275 (0.11)	220
	E_T (MPa)	15,830 (0.08)	18,310 (0.04)	18,420 (0.05)	0.894	17,470 (0.09)	15,660
Three-point bending (ASTM D7264)	n	18	18	18	–	54	–
	MC (%)	6.8	6.8 (0.07)	7.6 (0.08)	–	–	–
	f_b (MPa)	202 (0.07)	208 (0.06)	203 (0.03)	0.165	205 (0.06)	183
	E_b (MPa)	16,210 (0.07)	16,550 (0.05)	16,210 (0.03)	0.584	16,320 (0.06)	15,190
	LOP (MPa)	123 (0.07)	125 (0.06)	125 (0.06)	0.750	124 (0.06)	110
Flat-ring flexure (Virgo et al. 2017)	n	13	7	8	–	28	–
	MC %	9.5 (0.05)	9.7 (0.04)	9.7 (0.08)	–	–	–
	f_r (MPa)	10.7 (0.27)	13.3 (0.10)	13.3 (0.21)	0.989	12.1 (0.23)	6.9

MC moisture content



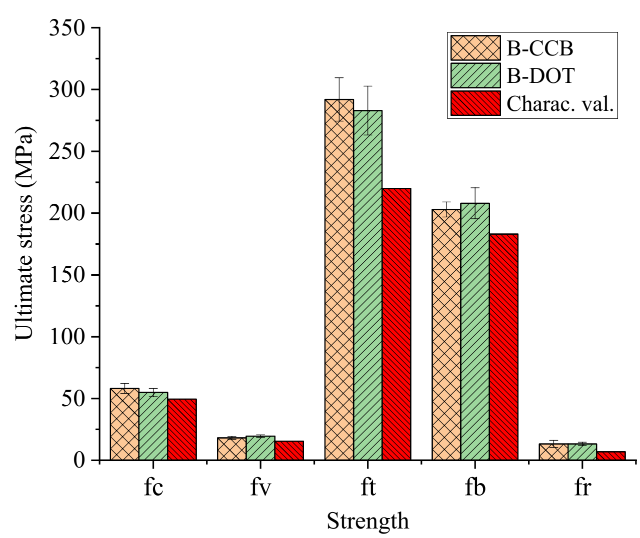


Fig. 8 Comparison between DOT and CCB treated samples, where *Charac-val-* characteristic values, *Ec* modulus of elasticity in compression, *G* shear modulus, *Et* modulus of elasticity in tension, *Eb* modulus of elasticity in bending, *fc* compression strength, *fv* shear strength, *ft* tensile strength, *fb* modulus of rupture in bending, *fr* = transversal tensile strength. (Error bars represent ± 1 standard deviation)

Saikia et al. (2015) investigated the tension and bending strength of three different bamboo species (*B. tulda*, *D. giganteus* and *B. balcoa*) treated with CCB and a new bio-chemical treatment. Although no information regarding retention and penetration of the active ingredients is reported, it was found that CCB-treated and untreated samples had similar values of ultimate tensile and flexural strength in samples exposed to environmental conditions for 6 months (Saikia et al. 2015). The treatment with DOT also does not negatively affect the mechanical properties of bamboo. In fact, a small increase in flexural and compression strengths was observed in *D. asper* bamboo samples with high retentions of DOT (Gauss et al. 2019a).

To the best of the authors` knowledge, no paper was found addressing all the mechanical characterizations used in this work. Nevertheless, some mechanical properties of *P. edulis* bamboo available in the open literature are consistent with the results shown in Table 2 (considering the average of all the samples). For compression and shear strength parallel to the fibres, values of f_c between 46.0 and 48.1 MPa and f_v between 11.2 and 15.9 MPa are reported (in this work, the average f_c and f_v are 57.9 and 18.0 MPa, respectively) (Xu et al. 2014; Huang et al. 2015; Deng et al. 2016; Akinbade et al. 2019). Dixon et al. (2015), using *P. edulis* samples of density similar to this work, reported modulus of rupture and modulus of elasticity in bending of $f_b = 215$ MPa and $E_b = 16{,}680$ MPa, respectively.

3.3.1 Characteristic properties

Since no difference was found in properties of DOT- and CCB-treated bamboo, taking all data together (A-DOT + B-DOT + B-CCB), a sufficiently large sample (taken from 17 randomly selected culms of the original batch of 140 culms) is available to assess characteristic material properties suitable for design.

For strength, the characteristic value is defined as the 5th percentile value determined with 75% confidence (ISO 22156:2004 2004) and for modulus, the mean value established with 75% confidence is used (ISO CD 22156:2019 2019). The calculated characteristic values are shown in Table 2 and graphically compared with the mechanical properties of B-DOT and B-CCB samples in Fig. 8.

4 Conclusion

The treatment and mechanical properties of *P. edulis* bamboo treated with DOT and CCB were assessed. Penetration and retention assessment and microstructural analyses were conducted to investigate the efficacy of the treatment processes. Mechanical testing of treated samples was used to compare the resulting bamboo material properties after treatment. The following conclusions were drawn:

- The bamboo treated with CCB by a full-cell process exhibited higher retention values than the bamboo treated with DOT by immersion: 7.2 kg/m^3 and 2.2 kg/m^3, respectively. Good penetration (between 50 and 100%) was observed in both cases.
- Microstructural analysis using optical and scanning electron microscopy showed no visual differences in the vessels and parenchyma cells between treatment conditions. Elemental analysis using EDS revealed a higher concentration of copper and chromium elements in the conducting vessels of the bamboo treated with CCB. Only traces of these elements were found in the parenchyma cells and fibre bundles.
- Compression, tension (parallel and transverse to fibres), bending, and shear properties were not affected by the treatment procedures. The quality assessment of samples treated with DOT demonstrated low variation in all the investigated mechanical tests, suggesting a uniform mechanical properties distribution within the batch of *P. edulis* bamboo used in this study.
- Combining all the investigated conditions, characteristic values of compression, tension, shear and bending were calculated according to ISO 22157-19: $f_c = 49.5$ MPa; $E_c = 18{,}040$ MPa; $f_v = 15.4$ MPa; $G = 2520$ MPa; $f_t = 220$ MPa; $E_t = 15{,}660$ MPa; $f_b = 183$ MPa; $E_b = 15{,}190$ MPa.

Today, bamboo remains primarily an "informal" structural material. Although standards are available for structural design, in comparison with other conventional materials, there is little or no guidance available regarding quality control of commercially treated bamboo poles. It is recommended that bamboo poles used for structural applications should be subject to a quality control protocol based on treatment evaluation (retention and penetration) and mechanical properties in order to reduce risks and improve the efficient use of bamboo as a load-bearing structural material.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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