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Propose of models to estimate toughness as a function of physical and chemical properties of commercial thermally modified hardwoods

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ABSTRACT

The present research intended to propose and evaluate regression models which estimate toughness property as a function of physical, chemical properties of thermally modified hardwood and thermal treatment temperature, using linear, quadratic, cubic, exponential, logarithmic, geometric and multiple linear models. Commercial thermally modified woods were used on the study, being characterized for all referred properties, totalizing 450 experimental determinations. The analyzed models presented a low and moderate coefficient of determination, indicating the impossibility to use such models in the estimation of toughness as a function of physical and chemical factors

Keywords: Thermal modified wood. Hardwoods. Chemical properties. Physical properties. Regression models

1. INTRODUCTION

An alternative to chemical preserved wood, using creosote, CCA or CCB, is to adopt thermally modified wood on civil construction, industry and furniture, being a cleaner option, increasing wood capacity to face biological attacks and severe weathering conditions [1-4].

The process of thermal modification consists on heat wood on vacuum, steam or oil on temperatures that vary from 150°C to 280°C, which lead to a change on wood constituents, such cellulose, hemicellulose, lignin and extractives, and wood anatomy for a controlled time, increasing wood dimensional stability, diminishing hygroscopicity, shrinkage and permeability [1, 5-7]. Otherwise, proportionally with thermal treatment temperature increase, mechanical properties decrease due degradation of cellulose and hemicellulose [8-10].

On commercial purposes, several methods are available for wood thermal treatment, like Le Bois Perdure®, Plato Wood®, Reti Wood® and ThermoWood® [6, 11-14]. The most utilized method on market, ThermoWood®, is divided in three phases: dry and heat the wood until 130°C, then the thermal modification, elevating the temperature from 180°C until 200°C and cool and stabilize wood, controlling humidity content [14-17].

On the literature, several researches had already studied about Eucalyptus grandis, studying the influence of thermal treatment on physical, chemical, mechanical and anatomical properties [18-25]. For Indian Cedar (Acrocarpus fraxinifolius) and Australian Cedar (Toona ciliata var. australis), few researches are available, evaluating their use on particleboards, indicating the possibility on commercial purpose on civil construction, industry an furniture [26-30].

One form to possibility and encourage to use thermally treated hardwoods is to use models to estimate physical, chemical and mechanical properties as a function of thermal treatment temperature. It is consolidated on the literature that thermal treatment temperature interferes on wood properties, being possible to correlate the increase of temperature with raise or reduction of wood properties [23, 31-35]. Such generalization of

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models for hardwoods is possible due the similarity of wood anatomy and constituents on this wood class [36–38]. Also, for different temperatures which tests were not performed, is possible to estimate such properties with precision, respecting model limiting, such density and temperature range.

The only research using regression models to estimate physical, chemical and mechanical properties as a function of thermal treatment temperature was performed by KACIKOVÁ *et al*. [31]. The authors submitted Norway spruce wood specimens to temperatures up to 270°C for 30 minutes. Several chemical, physical and mechanical properties were evaluated and exponential models were used to estimate such properties as a function of thermal treatment temperature, ranging from 20°C and 237°C. The models presented elevated precision, with coefficient of determination varying from 75% to 99%.

In order to propose and analyze models to estimate toughness property as a function of thermal treatment temperature, apparent density, extractives content, lignin content, holocellulose content on thermally treated hardwoods, the present research evaluated three wood species thermally treated (Eucalyptus grandis, Acrocarpus fraxinifolius and Toona ciliata var. australis) on industry considering four different temperatures (155°C, 165°C, 175°C and 185°C) and reference temperature (20°C).

2. MATERIALS AND METHODS

The logs of Eucalyptus grandis, Indian Cedar (Acrocarpus fraxinifolius) and Australian cedar (Toona ciliata M. Roem var. australis), used in the present research were provided by planted industry, located in Ribeirão Branco, São Paulo, Brazil, and the average age of logs was 9 years old. The logs were sawn in lumber with transversal dimension of 6 cm x 16 cm and 3 m length. The lumber were dried on open air until reach moisture content of 12 % ± 2 %.

The thermal modification of wood was performed on industrial company, heating wood using autoclave with pressure and temperature control and saturated steam. The heating rate used by the company was 1.66 °C.min-1. The process used on thermal treatment can be described in five stages: Initial heating, autoclave loading, heating, thermal treatment and cooling. Initially, the autoclave on room temperature (20 °C) was heated with saturated steam and without wood until 100 °C, lasting about one hour. Then, the autoclave door is open to load wood in its interior. This process reduced the temperature from 100 °C to nearly 40 °C. On the third stage, the wood on the autoclave is heated from 40 °C until the desired thermal treatment. On the present research, four temperatures were considered on thermal treatment: 155 °C, 165 °C, 175 °C and 185 °C. On fourth stage, the thermal treatment is performed, with wood being modified for two hours, with maximum pressure of 735 kPa. On cooling stage, the pressure is relieved until the inner temperature on the autoclave reach room temperature (20 °C).

After thermal modification, lumber was sawn to produce test specimens to characterize Eucalyptus grandis, Indian Cedar (Acrocarpus fraxinifolius) and Australian cedar (Toona ciliata M. Roem var. australis) considering physical, mechanical and chemical properties. For physical and mechanical properties, the specimens were following the disposed on the Brazilian Standard ABNT NBR 7190 [38].

The following physical and mechanical properties were determined: apparent density (ρ) and toughness (fbw). For each temperature of thermal modification (4) and for the reference temperature, 12 specimens of each property were extracted for each wood specie.

For chemical analysis, the samples of wood were obtained according TAPPI Standard [39, 40]. The wood was crushed to reach small particles passing a 42 mesh (0,355 mm). The total extractive were evaluated by standard TAPPI 204 cm-97 [39], checking the volume of extractives on the samples. These samples were extracted in phases in a soxhlet with a mix toluene/ethanol for 6 hours (1:1 v/v); ethanol 95% pure for 5 hours and boiling distilled water for 30 minutes. After extractives remove, the samples were washed with distilled water and dried in oven at 103 °C ± 2 °C for 24 hours. The extractive content was calculated by mass difference. The resulting extractive-free wood was used to determine Klason lignin content by modified Klason method [41], by the sum of insoluble and soluble lignin. The holocellulose content was determined by difference between lignin content and extractive-free wood mass [42].

Regression models (Eqs. 1-6) were used to estimate toughness properties as a function of the thermal treatment temperature, apparent density, extractive content, lignin content, holocellulose content, individually and considering all factors, with Y being the estimated property (variable dependent), X the independent variable and b and the parameters adjusted by the least squares method:

$$Y = a + b \cdot X$$ [Lin - linear] (1)

$$Y = a \cdot e^{b \cdot X}$$ [Exp – exponential (2)

$$Y = a + b \cdot Ln(X)$$ [Log - logarithmic] (3)

$$Y = a \cdot X^{b}$$ [Geo - geometric] (4)

$$Y = a + b_1 \cdot X + b_2 \cdot \left(X\right)^2$$ [Quad – Quadratic] (5)

$$Y = a + b_1 \cdot X + b_2 \cdot \left(X\right)^2$$ [Cub – Cubic] (6)

The determination coefficient (R^2) was used to assess the quality of the adjustments obtained, making it possible to choose the best precision for each evaluated relationship. It is important highlight that 12 specimens were used to determine physical and mechanical properties for each temperature levels, including the reference temperature (in natura) for each wood specie and 6 samples for thermal treatment temperature for chemical properties, resulting in 630 determination at all. Determination coefficient R^2 with values between 0,10 and 0.30 are classified as low, between 0,4 to 0,6 as moderate and between 0,7 to 1,0 as high [43].

3. RESULTS AND DISCUSSION

Table 1 lists the mean values and extreme values of coefficient of variation (CV) for all physical, chemical and mechanical property evaluated for all three wood species.

Table 1: Results of physical, mechanical and chemical properties of Eucalyptus grandis, Acrocarpus fraxinifolius and Toona ciliata M. Roem var. australis wood for different thermal treatment temperatures.

Wood Specie	T (°C)	ρ (g/cm³) (CV - %)	Ex (%) (CV - %)	L (%) (CV - %)	H (%) (CV - %)	f_{bw} (MPa) (CV - %)
Eucalyptus grandis	20	0,537 (10,93%)	6,06 (3,47%)	28,93 (0,71%)	65,01 (0,51%)	105,81 (17,26%)
	155	0,520 (8,39%)	21,49 (2,68%)	32,34 (2,40%)	46,17 (1,48%)	41,00 (17,29%)
	165	0,502 (10,64%)	23,49 (1,77%)	37,96 (2,88%)	38,55 (2,66%)	26,71 (25,08%)
	175	0,457 (15,49%)	25,61 (25,61%)	35,77 (2,31%)	38,62 (1,88%)	16,00 (16,69%)
	185	0,434 (22,66%)	28,75 (3,25%)	33,14 (6,25%)	38,12 (4,49%)	13,12 (11,79%)
Acrocarpus fraxinifolius	20	0,425 (9,00%)	2,13 (11,00%)	31,66 (2,00%)	66,20 (1,00%)	124,50 (43,00%)
	155	0,643 (2,00%)	20,23 (2,00%)	29,41 (9,00%)	50,35 (8,00%)	45,56 (6,00%)
	165	0,554 (6,00%)	23,45 (0,00%)	28,27 (7,00%)	48,28 (6,00%)	307,70 (47,00%)
	175	0,545 (11,00%)	27,18 (0,00%)	26,12 (9,00%)	46,70 (6,00%)	32,53 (29,00%)
	185	0,534 (7,00%)	30,86 (2,00%)	24,68 (9,00%)	44,46 (6,00%)	16,91 (19,00%)
Toona ciliata M. Roem var. *australis*	20	0,368 (6,92%)	6,69 (1,08%)	27,89 (9,72%)	65,42 (4,07%)	40,09 (19,63%)
	155	0,361 (2,32%)	9,76 (6,37%)	39,90 (3,77%)	50,34 (3,56%)	38,80 (10,77%)

	165	0,314 (4,41%)	15,19 (2,77%)	41,28 (3,85%)	43,53 (2,97%)	23,28 (10,00%)
	175	0,304 (3,67%)	20,07 (5,98%)	37,72 (10,84%)	42,21 (8,13%)	16,70 (23,71%)
	185	0,283 (10,49%)	20,11 (3,61%)	37,73 (14,83%)	42,16 (14,44%)	16,00 (13,46%)

T: thermal treatment temperature; ρ: apparent density; Ex: extractive content; L: lignin content; H: holocellulose content; f_{bw}: toughness property.

Observing the behavior of physical, chemical and mechanical properties, the coefficient of variation (CV) increases with elevation of thermal treatment temperature. Such performance is corroborated by literature [6, 35, 44–47], which can be explained by a major degradation of wood constituents and wood hysteresis [31, 46, 48], increasing the inherent material variability after thermal modification.

Comparing the results of apparent density, the values obtained are close to reached by BAL and BEKTAŞ [24] for Eucalyptus grandis thermally treated at 150°C and 180°C (from 0,545 g/cm³ to 0,554 g/cm³ and CV varying between 11% and 14%), CALONEGO *et al*. [46] that studied Eucalyptus grandis treated at 20°C and 180°C (from 0,445 g/cm³ to 0,477 g/cm³ and CV ranging between 5,17% and 7,89%) and close to the reached by SÁ *et al*. [29] for in natura Australian cedar (0,320 g/cm³).

Considering chemical properties, extractive content rise with progressive thermal temperature increase, which can be explained by wood degradation and the production of new products along thermal treatment. Similar results were found by POCKRANDT *et al.* [6] evaluating Sterculia appendiculata K. Schum and Azadirachta indica A. Juss wood species, by KACÍKOVÁ *el al*. [31] analyzing Norway spruce wood, by ČABALOVÁ *et al.* [49] evaluating thermal modified Querus robur L. wood specie, by ZANUNCIO *et al.* [50], with extractive content varying from 6,05% (20°C) to 6,84% (200°C) for Eucalyptus grandis thermally treated. BATISTA *et al*. [51] found an increase of 613% on thermally modified Eucalyptus grandis, varying from 2,22% (untreated) to 15,85% (180°C).

For lignin content, the values disposed demonstrate an increase on content until 165°C and then, a stabilization on lignin content. Different behavior is found on the literature for On Corymbia citriodora Hook [52], Norway spruce wood [31], Quercus robur L. [49] and Pinus sylvestris L. [44]. For Eucalyptus grandis themally modified, Zanuncio et al. [50] reached a progressive increase considereing temperature rise, from 28,76% (untreated) to 30,36% (200°C). MOURA *et al.* [22] encountered an increase of 10%, from 31,92% (untreated) to 35,18% (180°C). Such behavior can be explained by the thermal degradation of carbohydrates, hemicellulose decomposition and condensation reaction [2, 31, 49].

For holocellulose, all species displayed the same behavior after thermal modification, with an average decrease of 40%, higher than obtained by ZANUNCIO *et al.* [50] (reduction of 2%), MOURA *et al.* [22] (reduction of 9%), all considering thermally modified Eucalyptus grandis. Such behavior of holocellulose content may be major explained by hemicellulose degradation, due to low amount of cellulose that can be degraded at temperatures below 200°C [42].

Table 2 lists the regression models with best adjustment of physical and chemical properties estimating the toughness properties.

Table 2: Results of regression models.

Model	R^2 (%)
$f_{bw} = 105,07 - e^{-0,0076 \cdot T}$	28,98
$f_{bw} = 121,62 \cdot \rho^{1,48}$	17,30
$f_{bw} = 190,29 \cdot (\mathrm{Ex})^{-0,61}$	25,69
$f_{bw} = -9872,66 + 922,39 \cdot L - 28 \cdot L^2 + 0,28 \cdot L^3$	30,37
$f_{bw} = 0,0002 \cdot (H)^{3,16}$	44,24

The regression models obtained to estimate toughness (fbw) property as a function of thermal modification temperature (T), apparent density (ρ), extractive content (Ex), lignin content (L) and holocellulose content (H) presented coefficient of adjustment below 70% [53], indicating low to moderate precision for the models, i. e., the factors were not able to estimate uniquely the toughness property on thermally treated hardwoods. It is important highlight that on the literature there is only one research using regression models is the research of KACIKOVÁ *et al.* [31], which used exponential models to estimate physical, mechanical and chemical properties as a function of thermal treatment temperature for one wood specie thermally treated Norway spruce oak. The models presented elevated precision, above 75%, being possible to be used as wood properties estimators.

To evaluate all factors in one regression model, taking into account the contribution of each factor for estimate toughness property, on Table 3 a multiple linear regression model is presented and its coefficient of determination (R^2).

Table 3: Result of multiple regression model.

Model	R^2 (%)
$f_{bw} = -302390 + 0,92 \cdot T + 298 \cdot \rho + 3016 \cdot \mathrm{Ex} + 3019 \cdot \mathrm{L} + 3026 \cdot \mathrm{H}$	27,01

Moreover, including all factors, the model precision is low, below 70%, indicating the impossibility to use thermal modification temperature, physical and chemical factors to estimate toughness property. Such behavior on the literature is unique and impossible to be compared with other wood species. This impossibility can be explained due fragile nature of toughness property and along thermal modification process, the elevated degradation of hemicellulose and the production and storage of extractives on wood makes imprecise the correlation of any of these factors to the behavior of toughness property on hardwoods thermally treated, demanding a major number of species in order to obtain a more precise model in further researches [23, 31, 49, 53, 54].

4. CONCLUSION

Considering the results of the present research, it is possible to conclude:

- The physical and chemical results of thermally treated wood species presented in this research (Eucalyptus grandis, Acrocarpus fraxinifolius and Toona ciliata M. Roem var. australis) are compatible to other thermally modified hardwood on similar treatment temperatures;

- Observing the coefficient of determination R^2 reached on the regression models considering physical and chemical factors to estimate toughness property, the models were considered imprecise, being not possible to perform such estimate.

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