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EVALUATION OF COATING PERFORMANCE AND COLOR STABILITY ON THERMALLY RECTIFIED *EUCALYPTUS GRANDIS* AND *PINUS CARIBAEA* VAR. *HONDURENSIS* WOODS

Luiz Fernando De Moura, José Otávio Brito, Adriana Maria Nolasco
Lis Rodrigues Uliana
University of São Paulo (Esalq/Usp), Department of Forest Sciences
São Paulo, Brazil

Graciela Ines Bolzon De Muniz
Federal University of Paraná (Ufpr), Department of Forest Engineering and Technology
Paraná, Brazil

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ABSTRACT

It is recognized that thermal rectification improves some wood properties (e.g. resistance to fungi, dimensional stability, hydrophobicity etc.). However, little information is available on the effect of heat treatments on the finishing properties and color stability of wood. In this work, *Eucalyptus grandis* and *Pinus caribaea* var. *hondurensis* woods underwent a variety of thermal rectification treatments (from 140 to 200ºC). Heat-treated wood was sanded (60-80 grit) and roll-coated with a UV-curable polyurethane. Coating performance was assessed through pull-off adhesion tests and accelerated aging. Samples were also evaluated for color stability before and after aging. The increment in maximum temperature of thermal rectification caused a decrease in coating pull-off adhesion strength. Slight coating cracks and adhesive coating failures occurred after aging of samples thermally rectified at maximum temperatures from 180 to 200ºC. The thermal rectification at 160ºC provided an optimum combination of color stabilization and adhesion upholding after aging.

KEYWORDS: Heating, coating, adhesion, aging.

INTRODUCTION

Impregnation with heavy metals is currently the most usual practice to enhance wood durability. However, these preservative products are highly hazardous to environment and humans, as they may contaminate groundwater and soil. Due to their high toxicity, many of these

heavy metal-based products have already been banned in several countries in North America and Europe. In this context, there is a need for research on alternative clean technologies for wood preservation. In North America and Europe, outdoor wood sidings are increasingly being replaced by other materials requiring less maintenance practices, as aluminum and vinyl. Similarly, outdoor wood furniture has increasingly being replaced by plastics and metals. It is noted, thus, the need for new competitive wood materials and processes for the market of outdoor products.

The thermal rectification is an alternative heat treatment to add value to wood. The thermally rectified wood acquires colors similar to those observed in commercial tropical woods, better resistance to fungi and weathering, higher dimensional stability, and lower hygroscopicity. This material is therefore suitable for outdoor and indoor-humid uses that do not involve high mechanical stresses. The increase in dimensional stability of thermally rectified wood is associated with the decrease in its hygroscopicity. During heating, the most hydrophilic polysaccharides (hemicelluloses) volatilize, reducing the availability of free hydroxyl groups, whereby water molecules would usually adhere. It is also mentioned the increase in lignin proportion which improves the hydrophobic properties of the heat-treated material (Duchez and Guyonnet 1998).

The understanding of adhesion mechanisms on wood surfaces is essential to extend service life of transparent film-forming coatings. Surface wettability plays an important role on surface aptitude for adhesion of coating films. Good wetting is fundamental to good adhesion as it provides better mechanical interlocking between the coating film and the wood surface. For any type of coating, good wetting might contribute to good film performance (Wulf et al. 1997). If a coating cures prior to complete wetting, a weak boundary layer of air bubbles may form in the interface (Lewis and Forrestal 1969). The importance of wood surface wettability on adhesion of a polyurethane varnish has been verified in a previous study (de Moura and Hernández 2005). In this context, it is believed that thermal rectification might hinder surface adhesion aptitude by reducing surface wettability.

Little information is available on the effect of heat treatments on the finishing properties of treated woods. In this work the varnish coating performance and color stability of *Eucalyptus grandis* and *Pinus caribaea* var. *hondurensis* treated with four levels of thermal rectification were evaluated in relation to some weathering agents (i.e. humidity and UV radiation) simulated through accelerated aging.

MATERIAL AND METHODS

Testing materials

Eucalyptus grandis and *Pinus caribaea* var. *hondurensis* woods were selected for this study, as they present relatively fast growing rates and low market values, in comparison to commercial tropical woods. Commercial air-dried flat-sawn 2.500 mm long sapwood boards were stored until they reached 12.2 % (*E. grandis*) and 13.2 % (*P. caribaea* var. *hondurensis*) equilibrium moisture contents (EMC). Each board was crosscut into four 80 by 600 mm oriented-grain matched samples. These matched samples were freshly planed to a thickness of 50 mm, prior to thermal rectifications. Each matched sample underwent a different level of heat treatment. The thermal rectification was applied on groups of seven samples from each species. The average basic densities of the boards were 532 and 394 $kg.m^{-3}$, for *E. grandis* and *P. caribaea* var. *hondurensis*, respectively.

Thermal rectifications

Thermal rectifications were performed in the LQCE (Laboratórios Integrados de Química,

Celulose e Energia – Integrated Laboratories of Chemistry, Pulp and Energy) of the Department of Forest Sciences at ESALQ/USP (Escola Superior de Agricultura "Luiz de Queiroz", University of São Paulo). The boards were heat treated in an electrical resistance oven, equipped with a system for air circulation, with a nominal chamber volume of 0.45 m^3. A rate of heating of 0.033°C.min^{-1} was applied. This heating rate is in compliance with those recommended by Deglise and Magne (1987) and Graham et al. (1984). The maximum temperatures (140, 160, 180, and 200°C) were chosen based on previous studies by Vovelle and Mellottee (1982), and Crow and Pickles (1971). Samples were placed into sealed metallic chambers (Fig. 1) with a unilateral opening, apertures for installing thermocouple wires, pipelines for purging volatilized gases and injection of nitrogen for displacing oxygen (Fig. 1A and B). Samples were oven-dried until 0 % moisture content, prior to starting treatments. The thermal rectification programs are illustrated in Fig. 2. For the purpose of data statistical analysis and graphic presentation, environmental temperature (25°C) was assigned for non-treated samples.

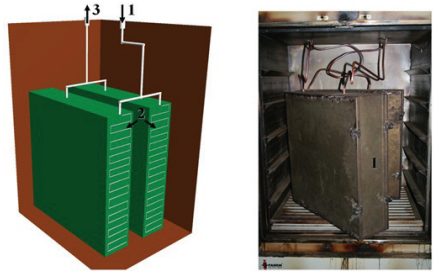

Fig. 1: Thermal rectification chambers designed to nitrogen injection, installed into the oven: A) Right-hand pipeline for gas entry 1), heat exchanger 2), and left-hand pipeline for gas purge 3), B) Overview of the system installed inside the oven, indicating unilateral opening 1).

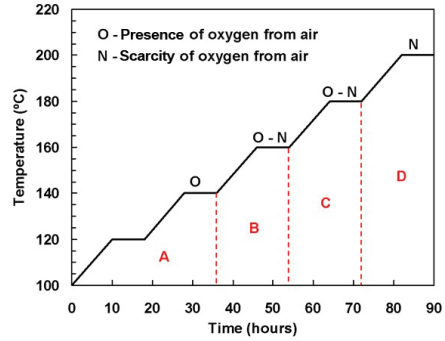

Fig. 2: Heat treatment programs (A = 140°C; A + B = 160°C; A + B + C = 180°C; A + B + C + D = 200°C). Note that treatments over 160°C were performed in presence or scarcity of oxygen from the air. The 200°C treatment was only performed in scarcity of oxygen, to avoid the risk of fire.

Coating procedure

As mentioned in previous works (de Moura et al. 2010), the use of 80-grit sandpaper should be preferred when finally sanding thermally-rectified wood surfaces prior to coating, as this grit size might provide enhanced conditions for wetting and mechanical bonding. Therefore, thermally rectified samples were surfaced with a 60-80-grit sanding program prior to coating. Samples were roll-coated with high-solids UV-curable polyurethane immediately after sanding. The coating film consisted of four layers of sealer followed by two layers of varnish. A light 280-grit sanding was performed before the last layer of varnish. The coating mass applied and UV radiation for curing each layer are presented in Tab. 1. The coating type and process variables are typically used in wood flooring industries. The average thickness of coating film was 52 µm, for *E. grandis*, and 42 µm, for *P. caribaea* var. *hondurensis*.

Tab. 1: Coating mass (g.m^{-2}) and UV radiation for curing (J.cm^{-2}), for six layers of polyurethane-based sealer and varnish, applied on Eucalyptus grandis and Pinus caribaea var. hondurensis samples previously surfaced with a 60-80-grit sanding program.

Layer	Type	Mass (g.m^{-2})	Radiation (J.cm^{-2})
1	Sealer	7.2	238
2	Sealer	13.1	260
3	Sealer	18.3	170
4	Sealer	19.5	289
5	Varnish	10.4	158
6	Varnish	4.1	556
		Total: 72.6	Total: 1.671

Pull-off adhesion tests

The adhesion of coating films was evaluated prior to aging by means of pull-off tests according to ASTM D 4541 (1995). A DeFelsko PosiTest AT-P pull-off tester with maximal capacity of 8 MPa and ±1 % full-scale accuracy was employed. Small 20-mm diameter dollies were glued on the film surface with Araldite 2011 two-part epoxy resin. After 24 hours of curing at room conditions, the perimeters of the glued dollies were carefully incised in order to prevent propagation of failures out of the tested area. A cylindrical actuator connected to a hydraulic pump was placed over the dolly head. Vacuum was applied gradually into the actuator with a rate inferior to 1 MPa.s^{-1} until separation of the dolly. The maximal normal pull-off strength at the rupture was recorded. The pull-off tests were carried out at 20°C and 60 % RH room conditions.

Accelerated aging tests

All coated specimens underwent an accelerated aging treatment in a xenon-arc weatherometer. The treatment consisted of 50 cycles of 102 minutes of UV radiation followed by 18 minutes of UV radiation combined with distilled water spray, totalizing 100 hours of aging. Specimens were exposed without any protection against water penetration.

The degree of deterioration of coating films after aging was evaluated by means of careful naked-eye and tactile inspections of the incidence of cracks and wrinkling, as well as the severity of color changes after aging. All aged samples were ranked by the degree of deterioration of their coatings: grades varied from 1 (lowest coating deterioration) to 30 (highest coating deterioration). Aging resistance was expressed in terms of the global ranking. In order to increase reliability of coating inspections, numerical grades corresponded to the average of two independent evaluators.

Color stability measurements

For color stability measurements, a Konica Minolta CM-2500D spectrophotometer was used. Measurements were undertaken using the D65 standard illuminant (daylight simulator) and a 10° angle of observation. Color data corresponded to the average of three measurements taken from different points on each coated sample prior to and after accelerated aging. The CIELAB lightness (L*) coordinate was chosen as the index of color stability in relation to weathering agents (i.e. humidity and UV radiation) simulated through accelerated aging. The difference between data prior to and after aging (ΔL*) was analyzed to quantify the effect of weathering agents on wood color.

RESULTS AND DISCUSSION

Pull-off adhesion

The results of pull-off adhesion tests, for a polyurethane coating applied on 60-80-grit sanded surfaces of *Eucalyptus grandis* and *Pinus caribaea* var. hondurensis after several thermal rectification treatments, are shown in Tab. 2 and Fig. 3.

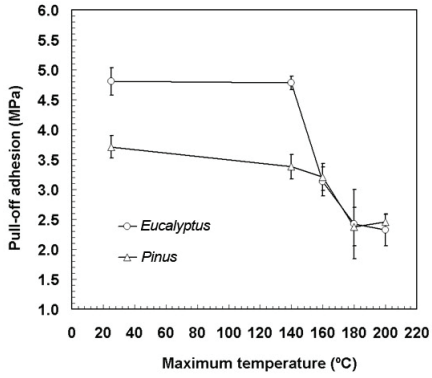

Fig. 3: Pull-off adhesion strength for a polyurethane coating applied on Eucalyptus grandis and Pinus caribaea var. hondurensis previously treated with different levels of maximum temperature in thermal rectification (atmosphere types pooled).

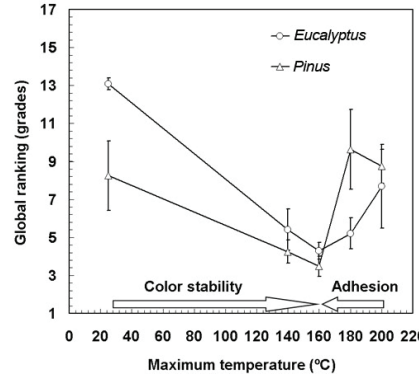

Fig. 4: Global ranking (grades) indicating the degradation level after an accelerated aging program, for Eucalyptus grandis and Pinus caribaea var. hondurensis previously treated with different levels of maximum temperature in thermal rectification (atmosphere types pooled).

There are no references available or direct evidences in this work indicating the presence of chemical linkages between wood and coatings. Hence, we could affirm that mechanical bonding was the major adhesion factor. Given that the 60-80-grit sanding program provided relatively high surface roughness (Ra; 11.41 μm, for *Eucalyptus*; 10.47 μm, for *Pinus*; maximum temperatures pooled), it is assumed that the coating has interacted with surface asperities to establish mechanical bonding.

In general, samples tested according to ASTM D 4541 (1995) were predominantly subjected to cohesive ruptures in wood rather than ruptures in the wood/coating interface. This indicates that for all treatments studied the bonding strength between wood and coating was predominantly higher than the mechanical strength at the wood surface and subsurface. In this context, the pull-off adhesion strength was likely associated with the tensile strength of wood itself.

The satisfactory adhesion on sanded surfaces may be explained by the presence of micro-fibrils torn out from wood cell walls by the abrasive grains. These fibrils diffuse through the liquid coating, so maintaining the film mechanically bound to the wood surface after curing. Thus in sanded surfaces, adhesion takes place in a three-dimensional interface. In this case, a composite of polymer and wood is observed as a transition between wood and coating (Backman and Lindberg 2002). Owing to this interface configuration, adhesive failures are rarely observed on coated sanded surfaces. Failures were predominantly cohesive and propagated through damaged cells at surface and subsurface which behaved as a mechanical weak boundary layer. In fact the test dollies pulled-off from samples showed a considerable amount of fibers pulled out

Tab. 2: Average pull-off adhesions obtained for a polyurethane coating applied on Eucalyptus grandis and Pinus caribaea var. hondurensis after several thermal rectification treatments.

Species	Atmosphere type	Maximum temperature (°C)	Pull-off adhesion (MPa)	Standard error	C1	C2	C3
Eucalyptus grandis	Presence of oxygen	25 (Control)	5.1	0.1	A	A	
		140	4.8	0.1	A	A	
		160	3.9	0.3	AB	A	a
		180	2.3	0.6	B	A	a
	Scarcity of oxygen	25 (Control)	4.5	0.3	A	A	
		160	2.4	0.2	AB	A	a
		180	2.6	0.5	AB	A	a
		200	2.3	0.3	B	A	
Pinus caribaea var. *hondurensis*	Presence of oxygen	25 (Control)	4.0	0.2	A	B	
		140	3.4	0.2	A	B	
		160	3.2	0.4	A	A	a
		180	2.5	0.2	A	A	a
	Scarcity of oxygen	25 (Control)	3.4	0.2	A	A	
		160	3.2	0.1	A	A	a
		180	2.3	0.5	A	A	a
		200	2.5	0.1	A	A	

Means within a column followed by the same letter are not significantly different at the 5 % probability level (Tukey's multiple comparison tests).
C1: Underlined capital letters are for maximum temperature comparisons, for each specie and atmosphere type separately.
C2: Capital letters are for specie comparisons, for each atmosphere type and maximum temperature separately.
C3: Lowercase letters are for atmosphere type comparisons, for each specie and maximum temperature separately. These comparisons were made only for 160 and 180°C maximum temperatures.

from the surface, mainly on specimens treated at the highest temperatures, which were subjected to deeper ruptures. These cohesive ruptures indicate the effectiveness of the mechanical anchorage and the relative weakness of the crushed cell layers. Superficial crushed cells are a result of high normal forces engendered during sanding operation (Stewart and Crist 1982).

Pull-off adhesion averages were similar for both species studied (3.0, for *Pinus*; 3.5 MPa, for *Eucalyptus*; maximum temperatures pooled). *Pinus* manifested lower pull-off adhesion than that observed for *Eucalyptus* only for control samples and thermal rectification at 140°C (Tab. 2, C2). For the other temperatures tested in thermal rectification, the pull-off adhesion strength of both species converged to similar values (Fig. 3).

The increment in maximum temperature of thermal rectification caused a decrease in coating pull-off adhesion strength. This fact was corroborated by a negative Pearson correlation coefficient between maximum temperature and pull-off adhesion strength (r = -0.6449, species pooled).

Pinus was less susceptible to loss in pull-off adhesion strength as a function of the increment in temperature of thermal rectification (32 % loss from 25 to 200°C), in comparison with *Eucalyptus* (52 % loss from 25 to 200°C). For the conifer, changes in pull-off adhesion strength as a function of maximum temperature could not be detected by Tukey tests (Tab. 2, C1). Although *Pinus* has a clear tendency to reduce adhesion strength with increasing temperature which is confirmed by a significant negative correlation between both variables (r = -0.6727).

The pull-off adhesion strength in *Eucalyptus* was significantly reduced with increasing the maximum temperature of thermal rectification, mainly for temperatures above 160°C (Tab. 2, C1, Fig. 3). This reduction is corroborated by a negative correlation between the maximum

temperature of treatment and adhesion strength (r = -0.7052).

Several previous studies have demonstrated that heat treatments are prone to impair mechanical properties of wood (Winandy and Lebow 2001, Kamdem et al. 2002, Bekhta and Niemz 2003, Awoyemi and Westermark 2005). In this work, considering the high incidence of cohesive ruptures observed in pull-off adhesion tests, it is assumed that the reduction in adhesion strength was strongly related to the decrease in mechanical properties of wood, caused by heating.

The thermal rectification considerably increases surface hydrophobicity, as the most hydrophilic polysaccharides (hemicelluloses) volatilize during heating (Duchez and Guyonnet 1998, de Moura et al. 2010). In this context it is assumed that thermal rectification might have hindered surface adhesion aptitude by reducing surface wettability. However, as the ASTM D 4541 (1995) adhesion tests provided predominantly cohesive ruptures in wood, the real impact of wettability on adhesion in the interface wood/coating was not clear. Thus for next studies, it is recommendable to compare these test results with those provided by other adhesion tests (ex. adhesive tape tests).

For both species studied coating adhesion was similar on samples treated in presence (3.0 MPa) or scarcity (2.6 MPa, species and maximum temperatures pooled) of oxygen inside the thermal rectification chamber (Tab. 2, C3). This fact suggests that for the range of maximum temperatures tested the presence of oxygen has no perceptible effect on properties of treated wood.

Accelerated aging

The results of aging resistance for a polyurethane coating applied on samples of *Eucalyptus grandis* and *Pinus caribaea* var. *hondurensis* after several thermal rectification treatments, expressed in terms of the global ranking (grades) of samples exposed to an accelerated aging program, are shown in Tab. 3 and Fig 4.

During accelerated aging, slight coating failures occurred at the end edges of samples whereby water infiltrated through the wood/coating interface, removing gradually the coating film from the surface (Lewis and Forrestal 1969, Yalinkiliç et al. 1999). In general the severity of the aging program was not enough to provoke wood cracks. Even though it was possible to clearly distinguish among coating behaviors in the presence of weathering agents, for the different levels of maximum temperature in thermal rectification.

Both species manifested similar coating behaviors in the presence of weathering agents. The average of grades given to *Eucalyptus* aged samples (6.6) were similar to those given to *Pinus* aged samples (6.8, maximum temperatures pooled). For the aged hardwood color variations were more pronounced in heartwood than in sapwood; for the aged conifer, in turn the most significant color changes were always observed in early-wood.

Although the thermal rectification caused a negative effect on adhesion properties of wood, it provided a positive effect, to some extent, on the condition of samples after accelerated aging. This positive effect was evidenced by an increase in color stability as the maximum temperature of treatment approached 160°C. For *Eucalyptus*, a negative linear correlation was detected between the maximum temperature and grades after aging (r = -0.6393). Thus, for the hardwood, the degradation of coating films by aging was reduced (i.e. lower grades) as the maximum temperature of thermal rectification increased. In *Pinus* however, this linear correlation was not detected as the effect of maximum heating temperature on aging resistance is likely to follow a quadratic pattern with the lowest grades.

For both species the thermal rectification at 160°C provided an optimum combination of color stabilization and adhesion upholding after aging.

Tab. 3: Average global ranking (grades) indicating the degradation level after an accelerated aging program, for a polyurethane coating applied on Eucalyptus grandis and Pinus caribaea var. hondurensis after several thermal rectification treatments.

Species	Atmosphere type	Maximum temperature (°C)	Global ranking (grades)	Standard error	C1	C2	C3
Eucalyptus grandis	Presence of oxygen	25 (Control)	13.2	0.3	A	A	
		140	5.4	1.1	B	A	
		160	4.3	0.4	B	A	a
		180	6.1	1.0	B	B	a
	Scarcity of oxygen	25 (Control)	13.0	0.3	A	A	
		160	4.3	0.5	B	A	a
		180	4.3	0.6	B	A	a
		200	7.7	2.2	B	A	
Pinus caribaea var. *hondurensis*	Presence of oxygen	25 (Control)	8.8	1.8	AB	A	
		140	4.3	0.6	B	A	
		160	2.9	0.1	B	B	a
		180	11.9	2.2	A	A	a
	Scarcity of oxygen	25 (Control)	7.8	1.8	AB	A	
		160	4.1	0.9	B	A	a
		180	7.4	2.0	AB	A	a
		200	8.8	0.9	A	A	

Means within a column followed by the same letter are not significantly different at the 5 % probability level (Tukey's multiple comparison tests).
C1: Underlined capital letters are for maximum temperature comparisons, for each specie and atmosphere type separately.
C2: Capital letters are for specie comparisons, for each atmosphere type and maximum temperature separately.
C3: Lowercase letters are for atmosphere type comparisons, for each specie and maximum temperature separately. These comparisons were made only for 160°C and 180°C maximum temperatures.

Samples heat treated below 160°C strongly darkened due to the UV radiation during aging. On the other hand, samples thermally rectified above 160°C moderately brightened in presence of UV radiation. Thus among samples treated at 160°C, very slight darkening or brightening could be observed after aging.

Slight coating cracks and adhesive coating failures occurred after aging, only in samples thermally treated at maximum temperatures from 180 to 200°C. This behavior indicates that samples thermally rectified at this range of temperature underwent a gradual reduction of their adhesion potential which is probably related to the decrease in wood wettability caused by heating.

Thus for samples treated at the highest temperatures (180 and 200°C), the level of coating deterioration took account of the occurrence of moderate brightening combined with slight adhesive failures and cracks in coating films. For control samples, in turn, deterioration by aging manifested as strong darkening, with virtually no adhesive failures in coating films.

The aging-resistance of coated samples was not significantly affected as a function of the presence (6.1) or scarcity (4.9, species and maximum temperatures pooled) of oxygen during thermal rectification, which confirms the lack of effect of this variable in this experiment.

Color stability

In general, the quantitative measurements carried out using the spectrophotometer confirmed the previous semi-quantitative results obtained by visual inspections. Spectrophotometry confirmed that accelerated aging caused darkening of samples thermally rectified below 160°C,

and indicated that this effect was reduced as the maximum temperature increased until 160ºC (Fig. 5). On the other hand, samples thermally rectified above 160ºC were brightened in the presence of UV radiation. This brightening effect was gradually increased as the maximum temperature of heat treatment augmented, being more pronounced in *Pinus* than in *Eucalyptus* (Fig. 5). *Eucalyptus* control samples were more susceptible to darkening in presence of UV radiation than *Pinus* control samples (Tab. 4, Fig. 5).

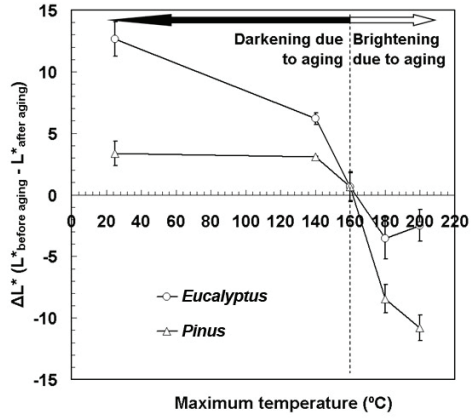

Fig. 5: Variation in lightness (ΔL = L*before aging - L*after aging) induced by an accelerated aging program, for Eucalyptus grandis and Pinus caribaea var. hondurensis previously treated with different levels of maximum temperature in thermal rectification (atmosphere types pooled).*

For both species, samples thermally rectified at 160ºC showed similar alterations in lightness (ΔL*) due to aging. As shown in Fig. 5, ΔL* tended to zero for samples thermally rectified at 160ºC. Thus for both species studied, it is assumed that 160ºC corresponded to the optimum maximum temperature in heat treatment for obtaining color stability in the presence of weathering agents.

Tab. 4: Average variation in lightness (ΔL = L*before aging - L*after aging) induced by an accelerated aging program, for Eucalyptus grandis and Pinus caribaea var. hondurensis previously treated with several thermal rectification programs and coated with a polyurethane film-forming finish.*

Species	Atmosphere type	Maximum temperature (ºC)	ΔL*	Standard error	C1	C2
Eucalyptus grandis	Presence of oxygen	25 (Control)	12.8	1.3	A	A
		140	6.2	0.5	B	A
		160	1.3	0.8	C	A
		180	-3.6	1.5	D	A
	Scarcity of oxygen	25 (Control)	12.6	1.5	A	A
		160	0.1	1.5	B	A
		180	-3.4	1.8	B	A
		200	-2.5	1.3	B	A

Pinus caribaea var. *hondurensis*	Presence of oxygen	25 (Control)	4.0	1.3	A	B
		140	3.1	0.1	AB	B
		160	0.1	0.7	B	A
		180	-8.8	0.7	C	B
	Scarcity of oxygen	25 (Control)	2.7	0.7	A	B
		160	1.2	1.8	A	A
		180	-8.0	1.5	B	B
		200	-10.8	1.0	B	B

Means within a column followed by the same letter are not significantly different at the 5 % probability level (Tukey's multiple comparison tests).
C1: Underlined capital letters are for maximum temperature comparisons, for each specie and atmosphere type separately.
C2: Capital letters are for specie comparisons, for each atmosphere type and maximum temperature separately

CONCLUSIONS

In general, samples tested according to ASTM D 4541 (1995) were predominantly subjected to cohesive ruptures in wood, rather than ruptures in the wood/coating interface. The increment in maximum temperature of thermal rectification caused a decrease in coating pull-off adhesion strength. For the next studies it is recommendable to compare these adhesion test results with those provided by other adhesion test standards.

Although the thermal rectification caused a negative effect on adhesion properties of wood, it provided a positive effect to some extent on the condition of samples after accelerated aging. This positive effect was evidenced by an increase in color stability as the maximum temperature of treatment approached 160°C. However, slight coating cracks and adhesive coating failures occurred after aging in samples thermally rectified at maximum temperatures from 180 to 200°C. This behavior indicates that samples thermally rectified at this range of temperature underwent a reduction of their adhesion potential, which is likely related to the decrease in wood wettability caused by heating.

In addition to enhance wood color and appearance, thermal rectification can be applied, to some extent, for obtaining improved color stability in the presence of weathering agents. For both species studied, the thermal rectification at 160°C provided an optimum combination of color stabilization and adhesion upholding after aging. The color alterations induced by aging were very slender in samples thermally rectified at this maximum temperature.

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Luiz Fernando de Moura, José Otávio Brito, Adriana Maria Nolasco
Lis Rodrigues Uliana
University of São Paulo (Esalq/Usp)
Department of Forest Sciences
Av. Pádua Dias 11- Caixa Postal 9
Cęp 134 18-900 Piracicaba
São Paulo
Brazil
Phone: (19)2105-8622
Correspondig author: demoura.lf@usp.br

Graciela Ines Bolzon de Muniz
Federal University of Paraná (Ufpr)
Department of Forest Engineering and Technology
Curitiba
Paraná
Brazil