



Maderas. Ciencia y Tecnología

ISSN: 0717-3644

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Chile

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Maderas. Ciencia y Tecnología, vol. 18, núm. 4, 2016, pp. 715-722

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Concepción, Chile

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# SURFACE WETTABILITY OF BRAZILIAN TROPICAL WOOD FLOORING TREATED WITH He PLASMA

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## ABSTRACT

This study investigated the effect of plasma treatment on changes of surface wettability of wood flooring from two Brazilian tropical species, *Hymenaea spp.* (rode locus) and *Tabebuia spp.* (lapacho). Wood flooring samples were plasma treated at low pressure in a helium atmosphere. Energy level was set at 100 W and four glow discharge times (5, 15, 30 and 45 s) were tested. Changes in wettability were investigated by measuring apparent contact angle, droplet volume and spreading contact area. The results showed less susceptibility of lapacho wood to the plasma treatments, while reduction of apparent contact angle in rode locus wood reached up to 76% for longer discharge times. In general, discharge time of 15 s produced the same effect as discharge of 45 s on wettability, which is important for industrial applications. Visual analyses revealed increase of water droplet spreading on lapacho wood surface, even though the variation of spreading contact area was not significant. Plasma treatment is feasible to improve wettability of tropical woods. Nevertheless, these findings should be investigated further due to the intrinsic characteristics of woods from tropical species.

**Keywords:** Contact angle, glow discharge, plasma treatment, spreading, surface modification, wetting.

## INTRODUCTION

Wood flooring from tropical species is an important market niche for sale of wood and wood-based products. Brazil is the third leading producer of certified tropical logs - around 30,8 million m<sup>3</sup> in 2013-2014 - only behind Indonesia and India, respectively (International Tropical Timber Organization 2015). Among these tropical species, rode locus (*Hymenaea spp.*) and lapacho (*Tabebuia spp.*) are widely used as raw materials to make flooring due to their attractiveness, especially related to their high natural durability and hardness. For example, *Tabebuia serratifolia* is one of the most widely used species for wood flooring in Italy (Romagnoli *et al.* 2013).

In the manufacturing process, the surface of wood flooring is commonly coated with finishing chemical products to improve various surface properties, such as resistance against weathering and scratching, and hardness. Nevertheless, surface adhesion of coatings can be limited due to intrinsic characteristics and/or surface inactivation of the wood. Wettability of wood surface is one of these characteristics that significantly affects the gluing and/or coating process (Thenepalli *et al.* 2015), since it is a measure of how fast a liquid spreads, wets or is repelled by a material's surface (Akgül *et al.* 2012). Furthermore, surface inactivation influences the wettability of wood. This phenomenon of inactivation can occur due to migration of extractives to the wood surface, surface oxidation and/or modification of surface hydroxyl bonding sites (Christiansen 1991). The severity of wood inactivation can be influenced by moisture content, temperature and exposure of the material in the environment (Aydin and Demirkir 2010).

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Received: 29.03.2016 Accepted: 25.08.2016

Because of these aspects, plasma technology has been used to increase wettability and surface adhesion of wood and wood-based products, reducing or avoiding these problems in the wood/coating interface (Asandulesa *et al.* 2010; Acda *et al.* 2012; Avramidis, G. *et al.* 2012; Tang *et al.* 2012; Riedl *et al.* 2014; Cademartori *et al.* 2015; Novák *et al.* 2015; Hünnekens *et al.* 2016). The effect of plasma on wettability of these materials is generated mainly by changes in their chemical structure and surface cleaning process (Dineff *et al.* 2011), which occur only in the outermost layer of the material's surface (Denes *et al.* 1999). These chemical changes, and consequently the increase of hydrophilicity, occur by the introduction of new functional groups on the substrate surface and/or by breaking the polymer chains on the outermost layer followed by crosslinking (Liston 1989; Carlotti and Mas 1998; D'agostino *et al.* 2008).

Experiments are initially necessary to understand the effect of plasma treatment on the surface of solid wood flooring products. This is necessary to extend practical applications, since the effects of plasma on the wood flooring materials have not been thoroughly explored, especially of tropical species with high market potential. In this study, radiofrequency glow discharge in helium (He) was used to improve surface wettability of solid wood flooring from two Brazilian tropical species, rode locus (*Hymenaea spp.*) and lapacho (*Tabebuia spp.*). The effect of treatment time on surface wettability was investigated in a low pressure plasma glow discharge. Changes in apparent contact angle (CA), volume of water droplet and spreading contact area (SCA) of droplet were investigated, expanding the existing results to other high-quality woods.

## MATERIAL AND METHODS

### Raw material

Rode locus (*Hymenaea spp.*) and lapacho (*Tabebuia spp.*) wood floor tiles measuring 80 x 27 x 20 mm (length, width and thickness) were purchased in a local market. These tiles were cut into small pieces of 25 x 20 x 20 mm (radial x tangential x longitudinal). Then, the samples were sanded with 120-grit sandpaper and were kept in a climatic chamber (20°C and 65% relative humidity) to reach the equilibrium moisture content (around 12%).

### Plasma treatments

Plasma treatments were performed in a homemade cylindrical stainless steel reactor (capacitively coupled plasma) with 50 cm diameter. This reactor works at low pressure with a radiofrequency power supply. It was previously described in detail by Cademartori *et al.* (2015).

Before the plasma treatments, He (>99% purity) was inserted inside the chamber at 0,032 Pa for 180 s to remove contaminants. Then, glow discharges were performed at ~10,64 Pa pressure with a He gas flow of 20 sccm. Energy level (power) was set at 100 W and four discharge times (5, 15, 30 and 45 s) were tested. All untreated and plasma treated samples were kept in a desiccator for 24 h to avoid the contact with humid air and contaminants until the wettability characterization. Plasma treatments were performed on the tangential wood surface.

### Wettability parameters

Wettability parameters of both untreated and plasma treated wood flooring samples were investigated by the sessile drop contact angle technique in a DataPhysics OCA 15+ goniometer. Three droplets of distilled water with 5 µl volume were deposited on the tangential surface of each sample. The measurements of CA were performed in the parallel direction to the fibers. Ten samples for each treatment time and each species were used, totaling 100 wood samples. All the CA measurements were performed at the same time in an air conditioned room at 20±5 °C and 65±5% relative humidity, to avoid external influences.

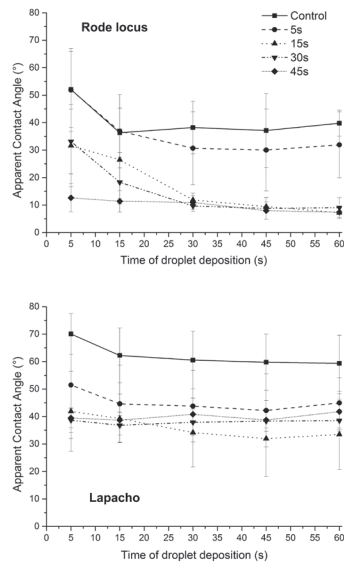
The apparent CA and water droplet volume were determined 5, 15, 30, 45 and 60 s after droplet deposition on the tangential wood flooring surface. High-resolution images were captured 15 s after droplet deposition using a Zeiss Discovery 1.0 stereomicroscope to determine SCA in mm<sup>2</sup> (Cademartori *et al.* 2015).

Statistical analysis

The wettability parameters were analyzed by descriptive statics and analysis of variance (ANOVA) at 5% probability of error. The Durbin-Watson test was used to confirm the normality of data variance. When the null hypothesis was rejected ( $p < 0,05$ ), the average values were compared by the Tukey HSD (Honestly Significant Difference) test at 5% probability of error.

RESULTS AND DISCUSSION

Kinetics of apparent CA were investigated as a function of time after droplet deposition to show that distinct behavior of wettability of the Brazilian wood floorings after plasma treatments is not specific to a single measurement point (Figure 1). Apparent CA of plasma treated lapacho wood flooring slightly decreased for all discharge times, especially up to 15 s after droplet deposition. On the other hand, the reduction of apparent CA of rode locus wood flooring as a function of time after droplet deposition was more intense than the decrease observed for lapacho wood. Fast penetration and spreading of water droplets were observed, especially in samples submitted to plasma treatments for 15 s or longer discharge times.



**Figure 1.** Kinetics of apparent CA of lapacho and rode locus wood flooring as a function of time after droplet deposition.

ANOVA results revealed a significant influence of plasma treatment on both apparent CA and droplet volume of lapacho wood flooring. Regarding rode locus wood flooring, only the apparent CA parameter changed significantly after the plasma treatments (Table 1). Previous studies (Avramidis, G. *et al.* 2012; Potočnáková *et al.* 2013; Novák *et al.* 2015) with wood and wood-based products have also reported this variation of wettability as a function of discharge time.

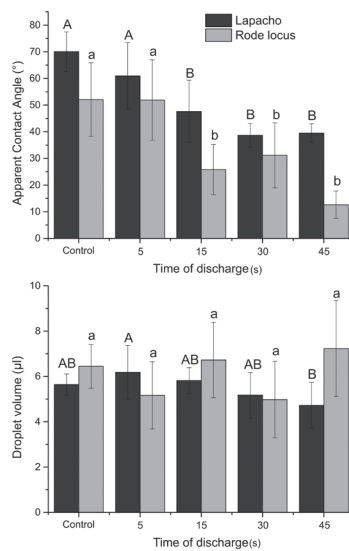
**Table 1.** Summary of analysis of variance (ANOVA) for apparent CA at 5 s, droplet volume at 5 s and SCA of wood floorings as a function of glow discharge time.

Species	Parameter	Glow discharge time	
		F-ratio	p-value
Lapacho	Apparent CA	21,14*	0,0000
	Droplet Volume	4,00*	0,0073
	SCA	0,83 <sup>ns</sup>	0,5148
Rode locus	Apparent CA	13,28*	0,0000
	Droplet Volume	2,23 <sup>ns</sup>	0,0950
	SCA	-	-

\* Significant at 5% probability of error. SCA was measured only in lapacho wood flooring because droplets fully spread across the surface of rode locus wood 15 s after deposition.

In general, apparent CA decreased with increasing discharge time, followed by stabilization (Figure 2). Apparent CA of lapacho wood flooring decreased up to ~44%, while rode locus wood flooring presented the highest reduction (~76%). This confirms significant changes in surface free energy of wood floor tiles due to the action of plasma treatments. The active species - such as He metastable, ions, electrons or UV radiation - in a He glow discharge strike the wood surface, causing crosslinking of the molecules. In addition, they decrease the contact angle and increase the surface free energy (Placinta *et al.* 1997), which was proved in this study for both lapacho and rode locus wood floor tiles. Removal of hydrophobic extractives from the plasma-treated wood surface contributes to increase the surface energy, and consequently to reduce the water contact angle (Avramidis *et al.* 2012). Moreover, changes in anatomical structure of wood - such as porous modification - by plasma etching result in the opening of liquid penetration paths (Jamali and Evans 2011), which also contributes to increase wood wettability.

Selection of most favorable processing parameters, such as energy levels and discharge times, is interesting due to the possibility to reducing energy cost and processing times (Cademartori *et al.* 2015). Thus, from an industrial point of view, 15 s discharge time produces the same effect as 45 s discharge time in the wettability of both lapacho and rode locus wood floorings. This suggests an increment of surface adhesion of wood flooring, which may improve finishing and bonding steps and decrease processing times in a wood flooring production line.

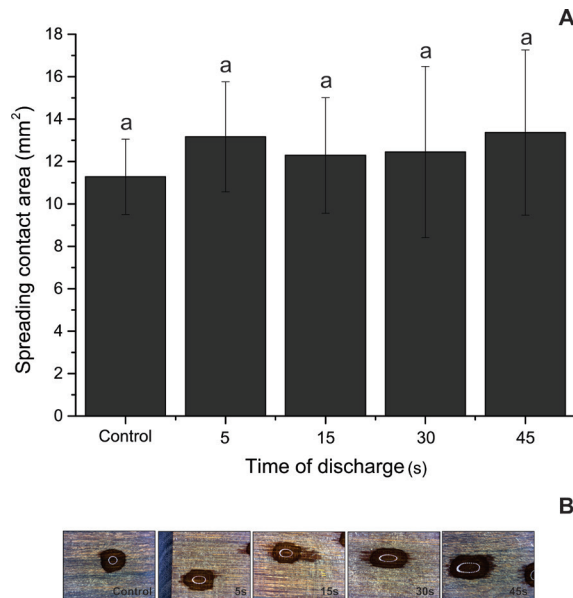


**Figure 2.** Apparent CA and droplet volume of rode locus and lapacho wood floorings as a function of discharge time. Average values followed by different upper case letters for lapacho wood and lower case letters for rode locus wood are statistically different at 5% probability of error.

Water droplet volume significantly changed only in plasma treated lapacho wood flooring (Figure 2). No significant loss in volume means only the occurrence of spreading phenomenon on the surface of rode locus wood flooring. On the other hand, the significant loss of droplet volume in plasma treated lapacho wood flooring suggests (in addition to the spreading phenomenon) a strong influence of both sorption and saturation phenomena in water-surface interaction. Nevertheless, the changes in droplet volume on lapacho wood surface were not systematic as a function of discharge time.

Lapacho wood flooring was less susceptible to plasma treatment in comparison to rode locus, since apparent CA tended to stabilize from 15 s to 45 s glow discharge time. This lesser susceptibility of lapacho wood may be related to the plentiful presence of extractives on the surface, especially lapachol, which is one of the most abundant extractives in *Tabebuia* spp. wood (Romagnoli *et al.* 2013). This phenolic compound is a naphthoquinone with hydrophobic character and low water solubility, which may have affected the interaction between water droplet and wood surface. Furthermore, the ample presence of extractives facilitates migration of these compounds from bulk to surface of wood, which may reduce the effect of plasma treatment due to the surface inactivation.

SCA of rode locus wood was not measured due to the fast spread of water throughout the surface of the samples. Water droplets spread over the entire surface, which hampered the SCA measurement. Visual and qualitative analyses allowed inferring higher spreading of water droplets on the plasma treated lapacho wood surface as a function of discharge time. Changes in SCA of lapacho wood remained between ~8 and ~15% (Figure 3), while SCA did not vary statistically after plasma treatments (Table 1). This was expected, since different characteristics influence SCA, such as contact angle, droplet diameter, speed of impact and surface roughness (Chandra *et al.* 1996; Fujimoto *et al.* 2001; Kannangara and Shen 2008; Wang *et al.* 2009).



**Figure 3.** Variation of SCA (A) and plan high-resolution images (B) of lapacho wood flooring as a function of glow discharge time. Average values followed by different lower case letters are statistically different at 5% probability of error.

From an industrial processing standpoint, the experimental observations in the present paper confirm the feasibility of applying plasma treatment to improve wettability of tropical wood flooring surfaces, and consequently can help to improve the quality of finishing steps. However, increase of wettability and surface adhesion by plasma in tropical woods should be better understood due to the natural variability and intrinsic characteristics of each species, such as roughness and wood anatomy.

## CONCLUSIONS

Increase of wettability of the wood flooring samples depended on glow discharge times. Plasma treatment for 15 s was sufficient to decrease significantly the wettability, producing the same effect of 45 s discharge time on wettability. Lapacho wood flooring was less sensitive to plasma treatment, presenting a more stable behavior as a function of time after water droplet deposition. This may be related to the characteristics of the extractives present on lapacho wood surface. Plasma treatment on rode locus wood flooring resulted in full spreading of water droplets.

## ACKNOWLEDGMENTS

We thank the National Counsel for Technological and Scientific Development (CNPq, grant number 141260/2013-0), National Hardwood Flooring Association (ANPM) and the International Tropical Timber Organization (ITTO) for supporting this work.

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