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Performance of Spray-Dried Nanofibrillated Cellulose as Wood Varnish Reinforcement in Outdoor Environment

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HIGHLIGHTS

- The addition of nanocellulose showed no effects on the varnish properties.
- All changes in color caused by natural weathering were noticeable.
- Exposure to natural weathering caused wood color to become gray.
- Addition of 5% nanocellulose reduced the variation in wood color.

Abstract: The outdoor use of wood can be improved by coating the wood surface with varnish to reduce the effects of natural weathering. However, some varnishes quickly lose their ability to protect wood, and nanoscale additives have been used to mitigate this negative effect, preferably renewable and biodegradable. Therefore, the objective of this study was to evaluate the performance of varnishes reinforced with spray-dried nanofibrillated cellulose (NFC) in a natural weathering test. For this purpose, two varnishes, waterborne and non-waterborne, with 0, 5, and 10% (w/w) NFC addition were applied to *Pinus taeda* tangential wood samples, which were exposed to natural weathering for half a year, during the period from January to June 2019, in Curitiba, PR, Brazil. Adherence and impact resistance tests were performed to evaluate the surface properties of the varnishes, and the effect of natural weathering on the wood surface was evaluated using quantitative colorimetric analyses (CIELab System). The varnishes did not perform well in the adhesion and impact test. While no significant effects were observed in surface tests, due to the addition of spray-dried NFC, varnishes reinforced with 5% NFC exhibited reduced overall color variation, maintaining yellow and red pigmentation. This suggests that incorporating 5% spray-dried NFC into varnishes did not compromise coating properties and aided in mitigating the impact of natural weathering on wood color variation.

Keywords: wood preservation; coatings; weathering; nanotechnology; color; biorefinery.

INTRODUCTION

Wood is a mechanically resistant and relatively light material; therefore, it is often used for structural and building support purposes [1,2]. It is present in almost all stages of civil construction, including formwork, structures, frames, flooring, decks, linings, furniture, and decoration [1]. The presence of wood is indispensable for many architects and engineers because it is a display of beauty and sophistication [3], on top of being a renewable resource and an eco-friendly material [4], offering thermal and acoustic insulation and reducing work costs [6].

As a biodegradable material that is susceptible to weathering, wood is commonly used in indoor applications; however, with appropriate treatment, it can be used outdoors [7]. Heat treatment [8–10], chemical modification [11,12], and wood finishing [13] can prolong the useful life of wood in the outdoor environment.

Wood finishing refers to the process of wood surface protection [13], and coatings such as paints, lacquers, and varnishes are widely used for wood finishing [7,14,15]. After applying varnish on wood, a rigid and transparent film is formed, which protects the substrate and maintains the visual aspect of the wood [16]. Owing to this aesthetic effect, varnish is often the preferred wood coating [16].

The color of wood becomes unstable when exposed to weathering, and the use of varnishes can reduce this instability [17]. However, some wood characteristics may affect the varnish performance. For instance, the texture and vessel obstruction of the wood may influence coating adherence [18], and the chemical characteristics of the wood may affect the capacity for hydrogen bond formation between the wood and varnish [19].

Many researchers have sought to improve the adhesion of wood coatings using additives ranging from organic and inorganic compounds to nanomaterials, such as nanocellulose [7,20–23]. Nanocellulose refers to cellulosic materials with at least one of their dimensions on the nanometer scale (from 0 to 100 nm) [24,25], and when tested as a wood coating additive, it improves rheological properties [26].

Waterborne varnishes use water with a small amount of organic solvent as the solvent [22], and are becoming more popular because they are non-toxic, have no odor, do not cause burning in the skin or in human eyes, and have low organic volatile compound content [22,27]. However, waterborne varnishes do not have a significant advantage over other varnishes in terms of resistance to acid, humidity, water, and heat, and are more difficult to apply [16,28].

The aim of this study was to incorporate cellulose nanomaterials at different contents (0, 5, and 10%) into two types of varnishes (waterborne and non-waterborne) and evaluate their performance in varnish adhesion, impact resistance, and wood color maintenance under natural weathering during a half-year of exposure.

MATERIAL AND METHODS

Wood, nanocellulose and varnishes

In this study, we used *Pinus taeda* timbers. From these timbers, samples with dimensions of 200 × 150 × 20 mm were produced, with the largest plane corresponding to the tangential wood surface. This plane was progressively sanded to 80, 100, and 120 grit granulometries for the subsequent application of varnishes.

The cellulosic pulp used was Brazilian bleached eucalyptus kraft pulp (BEK) produced by an elemental chlorine-free (ECF) bleaching sequence, donated by the Klabin S.A. Pulp and Paper Company (Telêmaco Borba, Paraná, Brazil). An aqueous suspension of cellulosic pulp with 2% consistency was prepared by homogenization. This suspension was then processed in a Masuko Sangyo Super Masscolloider (MKCA6-3, Masuko Sangyo Co., Kawaguchi, Japan) mill for 20 passes at 1,500 rpm rotation frequency.

The product of this process is an aqueous suspension with gelatinous aspect, containing nanofibrillated cellulose (NFC) [29,30]. These nanofibrils present an average diameter of 25±5 nm and a variable length in the micrometer scale, considered as a cellulosic nanomaterial according to ISO standard ISO/TS 20477 [31].

The NFC suspension was dried in a spray dryer (SD 10.0, LabMaq, Ribeirão Preto, Brazil) at a flow rate of 500 ml min^{-1} and temperature of 200 °C. The product of this drying process was a white powder formed by micro-sized particles [32], which returned to the nano-size when rehydrated and was used as varnish reinforcement.

Two varnishes were used, waterborne vanish (WBV) and copal-based non-waterborne vanish (NWBV) that is soluble in turpentine, acquired from Renner Sayerlack S.A. (Cajamar, São Paulo, Brazil). The viscosity of the WBV was 12 ± 2s CF4 at 25 °C, whereas the viscosity of the NWBV was 50 ± 5s CF4 at 25 °C, and both are recommended for indoor and outdoor use. In this study, six varnish formulations were tested and compared with a control treatment (wood without varnish); the formulations are presented in Table 1, and they were mixed using a mixer (711S, Fisatom, São Paulo, Brazil) for 5 minutes at 500 rpm.

Table 1. Description of the treatments with the respective cellulosic nanomaterial contents.

Treatment	Cellulosic nanomaterial content (% in w/v)
NV	0
WBV	0
WBV-5	5
WBV-10	10
NWBV	0
NWBV-5	5
NWBV-10	10

NV - no varnish, WBV - waterborne vanish, NWBV - non-waterborne vanish.

The varnishes were applied by brush strokes in two coats with an interval of 1.5 h between them. For each treatment, 10 repetitions were performed.

Surface testing

To evaluate the influence of the addition of spray-dried NFC on the performance of the varnish surfaces, adherence and impact resistance tests recommended in the ABNT NBR 14535:2008 standard [33] were performed.

The adherence test was conducted using the grid technique, with five cuts of 10 mm in length and 2 mm between them. Subsequently, 32 g mm^{-1} of high-adhesion filament adhesive tape was applied and removed after 2 min. The results were quantified as the percentage of the film detached from the surface, and 22 repetitions were performed per treatment.

The impact resistance test was carried out dynamically with the aid of an apparatus for the launching of a 19 mm diameter steel ball in a free fall from 200 mm in height, and an impact energy of 3.86 J was estimated against the sample. Thereafter, cracks and depressions in the impacted material were observed using a 10x magnifying glass and classified according to ABNT NBR 14535:2008 standard [33] (Table 2). Three replicates were performed per treatment. For this test three repetitions per treatment were performed.

Table 2. Classification of damage caused by impact.

Class of damage	Description
5	Light damage that can present a soft mark, without the presence of cracks.
4	One to two circular or semicircular cracks around the impact area.
3	Moderate or severe crack.
2	Crack extending outside the impact area, and/or light peeling of the film.
1	More than 25% of the varnish removed from the impact area.

Reproduced from ABNT NBR 14535:2008 [33].

Exposure to natural weathering

The samples were exposed to natural weathering in the municipality of Curitiba, state of Paraná, Brazil (Geographical coordinates: 25°26'55.6"S 49°14'16.0"W SIRGAS 2000 and altitude of 925 m above sea level). According to Köppen’s climate classification, the climate in this region is Cfb (subtropical, without a dry season, and with a temperate summer) [34]. The samples were placed on a support with 35° of inclination (optimized angle for solar exposure considering the local latitude and solar geometry) in a north-south orientation, for a period of half a year, beginning in 2019, with monthly color measurements of the samples.

Color measurement

The colorimetric parameters were collected monthly using a Konica Minolta CM-5 spectrophotometer (Konica Minolta, Ramsey, USA) adapted to a D65 light source, and a 10° observation angle was taken directly on the exposed surface of the sample. All collection and calculations were performed according to the ASTM D2244-16 standard [35]. The measured parameters were luminosity (L*), green-red chromatic coordinates (a*), and blue-yellow chromatic coordinates (b*). The total color variation (ΔE) between the unexposed wood and the wood in the last month of exposure was calculated according to Equation 1.

$$\Delta E = \sqrt{\Delta L*^2 + \Delta a*^2 + \Delta b*^2} \quad (1)$$

Where:

ΔE: total color variation; Δa*: difference in green-red coordinate; Δb*: difference in blue-yellow coordinate.

The reflectance at frequencies in the visible range of the electromagnetic spectrum was also determined using data collected at the end of the exposure to natural weathering.

Statistical Procedures

To analyze the surface performance of the varnishes, Kruskal-Wallis non-parametric statistics, based on the H-statistic, were applied for the comparison of treatments.

The results of the color measurements were subjected to descriptive statistical analysis to obtain mean, minimum, and maximum values, and Grubb's test was used to evaluate the occurrence of outliers. Based on Bartlett's test, the homogeneity of variance was obtained, and to verify the difference between the mean values, Tukey's test at 95% probability was used.

Subsequently, the grouping of the colorimetric data at the end of the exposure to natural weathering by similarity was performed using multivariate cluster analysis, Euclidean distance, the between-group linkage method, and standardization by the number of Z-scores.

All statistical analyses were performed by the Statgraphics Centurion XVI statistical package (Statgraphics Technologies Inc., The Plains, VA, USA).

RESULTS

Surface analysis

The addition of nanocellulose had no significant effect on the varnish adherence (Kruskal-Wallis p-value>0.05) (Table 3) or impact resistance (Kruskal-Wallis p-value>0.05) (Table 4), suggesting that the nanomaterial had good dispersion in the varnish matrix [36] and good chemical compatibility with the varnishes [37]. Considering these two surface tests, there was no difference between the types of varnishes used (Kruskal-Wallis p-value>0.05) (Tables 3 and 4). All adherence values were considered low by the ABNT NBR 14535:2008 standard [33]; however, all the showed good performance in the impact resistance test.

Table 3. Results and statistical analysis of the adherence test.

Factor	Treatment	Medium values	Kruskal-Wallis p-value
Cellulosic nanomaterial content (% in w/w)	0	1.25	0.810
	5	1.87	
	10	1.25	
Type of vanish	WBV	1.07	0.411
	NWBV	1.78	

WBV - waterborne vanish, NWBV - non-waterborne vanish.

Table 4. Results and statistical analysis of the impact resistance test.

Factor	Treatment	Medium values	Kruskal-Wallis p-value
Cellulosic nanomaterial content (% in w/w)	0	4.80	0.513
	5	5.00	
	10	5.00	
Type of vanish	WBV	5.00	0.317
	NWBV	4.80	

WBV - waterborne vanish, NWBV - non-waterborne vanish.

Colorimetric analysis

The Table 5 presents the results of the colorimetric parameters throughout the natural weathering exposure test, accompanied by the statistical analysis.

Table 5. Mean of the colorimetric parameters of *Pinus taeda* wood, without and with coatings, for each period of exposure to natural weathering.

L*								
Month	**Treatment**							**Tukey p-value**
	NV	**WBV**	**WBV-5**	**WBV-10**	**NWBV**	**NWBV-5**	**NWBV-10**	
0	74.50a	68.78b	68.71b	70.17b	67.57b	67.96b	69.46b	0.000
February	73.56a	63.16b	64.20b	65.85b	61.34b	62.03b	61.23b	0.001
March	71.81a	64.23b	65.16b	65.18b	59.38b	61.16b	61.14b	0.000
April	70.53a	62.81b	63.24b	62.27b	55.06b	57.08b	58.22b	0.002
May	64.14a	63.03a	62.10ab	60.12ab	56.25bc	56.64b	56.17b	0.001
June	64.73a	57.38ab	60.62ab	57.62ab	52.41b	54.37b	51.96b	0.002
a*								
Month	**Treatment**							**Tukey p-value**
	NV	**WBV**	**WBV-5**	**WBV-10**	**NWBV**	**NWBV-5**	**NWBV-10**	
0	4.64e	6.59c	6.36c	6.31c	8.05a	7.23b	7.09bc	0.023
February	6.09d	11.63bc	11.23bc	10.72c	14.88a	13.44ab	12.96ab	0.008
March	4.78c	10.72b	10.53b	9.65b	14.18a	12.29ab	13.01a	0.000
April	3.28d	11.64ab	9.15bc	6.89cd	15.48a	11.38ab	10.80ab	0.000
May	2.94c	11.88b	12.62ab	15.48a	14.73a	14.51a	13.84ab	0.003
June	2.27c	10.38ab	7.26b	11.38ab	14.52a	9.58ab	9.97ab	0.000
b*								
Month	**Treatment**							**Tukey p-value**
	NV	**WBV**	**WBV-5**	**WBV-10**	**NWBV**	**NWBV-5**	**NWBV-10**	
0	12.73c	17.64b	16.70b	16.73b	16.52b	16.52b	20.15a	0.000
February	14.68c	34.35b	31.38b	29.33b	41.31a	35.17ab	42.31a	0.002
March	11.83c	30.62b	29.71b	25.74b	40.39a	30.57b	40.60a	0.000
April	9.08d	32.31ab	24.73bc	19.93c	39.75a	28.48bc	34.198ab	0.000
May	4.77e	32.02d	33.77cd	32.17d	39.14ab	36.22bc	41.55a	0.034
June	6.98d	26.49ab	18.62bc	13.89c	33.92a	22.95ab	29.76ab	0.000

NV - no varnish, WBV - waterborne vanish, NWBV - non-waterborne vanish, L*: brightness, a*: red-green chromatic coordinate, b*: yellow-red chromatic coordinate. In the same line different letters indicate a significant difference in the Tukey test at 5% of probability.

The color of *Pinus taeda* wood is considered light because it has a L* value higher than 56 [38], and is predominantly yellow because it contains a higher proportion of b* chromophores than a* chromophores.

The application of varnishes significantly decreased the brightness and increased the proportion of the a* and b* chromophores, which is a common effect of these coatings [39]. Considering the luminosity, there was no significant difference between the varnish type and the presence of nanocellulose. Whereas the chromatic coordinate a* was increased by the application of both WBV and NWBV, the addition of nanocellulose only significantly increased yellowing in the NWBV. The chromatic coordinate b* had an effect similar to that of the chromatic coordinate a*, with a significant increase with the addition of nanocellulose only in the NWBV reinforced with 10% nanocellulose.

Exposure to weathering has caused the untreated wood to become darker, less yellowish, and less reddish, making the wood color grayish [40,41], which is a typical behavior of the wood after exposure to natural weathering. The change in wood color is a result of the loss of extractives present in the wood [42], as well as the degradation of the chemical components of the cell wall, such as lignin, cellulose, and hemicelluloses [43].

The reduction in the chromatic coordinate L* also occurred in the varnished wood; however, it was more intense in the NWBV, and there was no significant effect from the addition of nanocellulose. Unlike the unvarnished wood (NV), the treated wood showed yellowing and reddening. Yellowing and reddening were more intense in wood that received WBV without the addition of nanocellulose and in those that received NWBV (NWBV, NWBV-5, and NWBV-10). This difference in the color-change behavior suggests that varnishes underwent pigmentation, which changed the color of the wood surface over time [44].

Table 6 shows the total color variation caused by exposure of the woods to natural weathering.

Table 6. Total color variation after exposure to natural weathering.

Treatment	**ΔE**
NV	12.19
WBV	15.35
WBV-5	13.79
WBV-10	14.31
NWBV	23.24
NWBV-5	19.05
NWBV-10	23.63

Where: ΔE: total color variation, NV-no varnish, WBV-waterborne vanish, NWBV-non-waterborne vanish.

The total color difference becomes very appreciable to the human eye when it is greater than six [45]. In this case, even though it was a short exposure time, all the samples suffered appreciable color variations. The addition of 5 and 10% nanocellulose to the WBV and 5% nanocellulose to the NWBV decreased this change; however, the changes were still visible.

The grouping of the colorimetric data by similarity at the end of exposure to natural weathering (Figure 1) showed that the samples that received varnishes differed greatly from those that did not. The difference between NV and the other treatments was also observed in the reflectance graph (Figure 2), with the NV having the highest reflectance at all wavelengths of the electromagnetic spectrum. These differences were due to the darkening effect caused by the application of the varnishes [46], observed in Table 5.

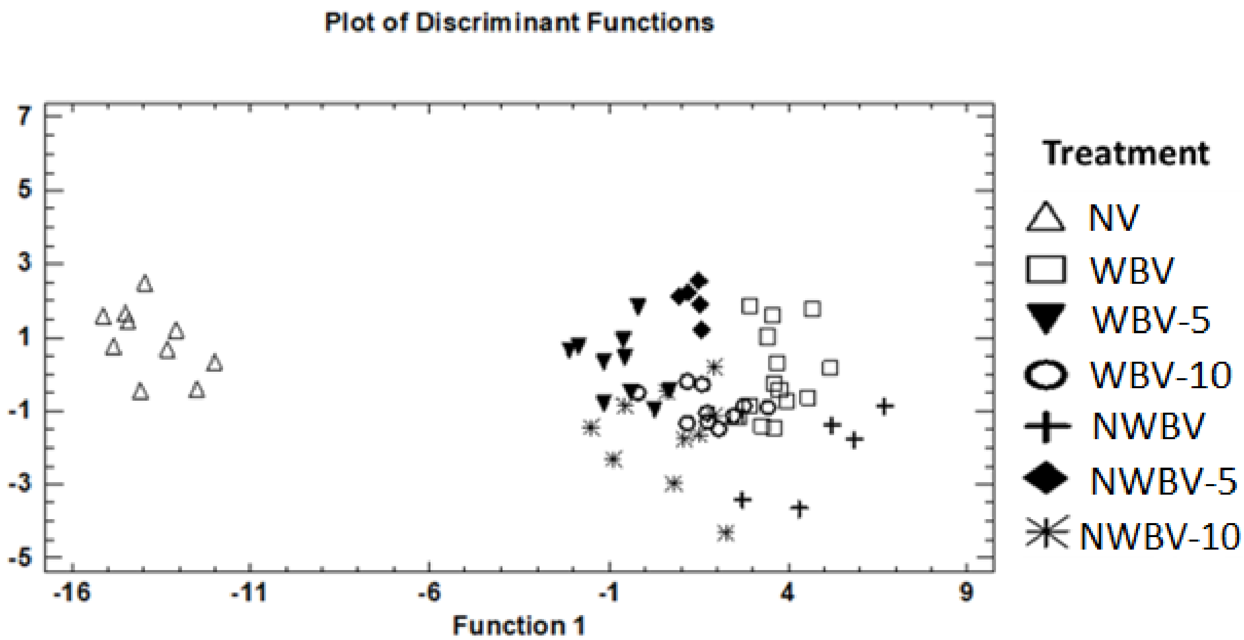

Figure 1. Grouping of the colorimetric data by similarity.

Where: NV - no varnish, WBV - waterborne vanish, NWBV - non-waterborne vanish.

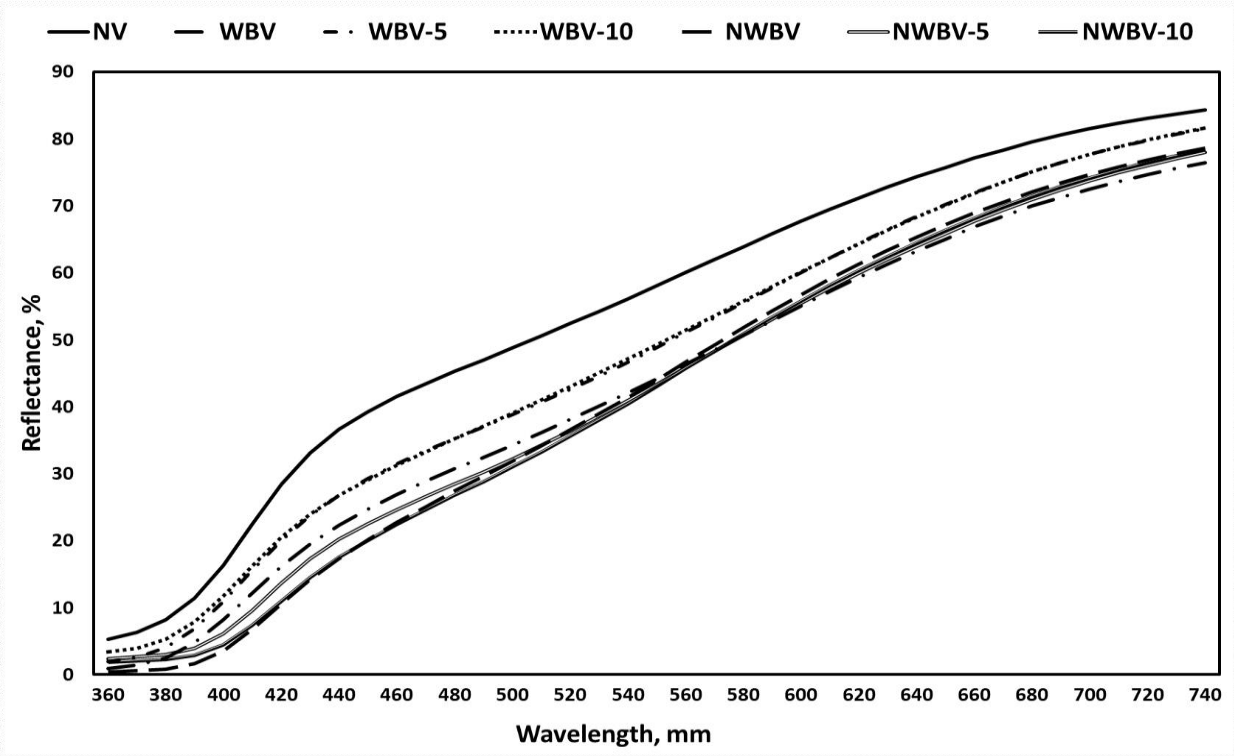

Figure 2. Reflectance of treatments in the frequencies of the visible range of the electromagnetic spectrum.

Where: NV - no varnish, WBV - waterborne vanish, NWBV - non-waterborne vanish.

CONCLUSION

The addition of nanocellulose showed no significant effect on the adherence of varnishes to wood or on the impact resistance of these coatings; therefore, it is possible to add up to 5% nanocellulose without harming the characteristics of the varnishes.

The application of the varnishes darkened, yellowed, and reddened the wood surface color. The addition of nanocellulose did not affect the darkening of the wood; however, the addition of 5 and 10% nanocellulose to the NWBV significantly increased the yellowing effect, and the addition of 10% nanocellulose to the NWBV significantly increased the reddening change.

All color changes caused by natural weathering are visible to the human eye. In the wood without varnish, the color tended towards grayish, whereas the application of varnish caused an increase in yellow (a*) and red (b*) pigments over time. The varnishes reinforced with 5% nanocellulose exhibited lower wood surface color variation at the end of the experiment.

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