



Exposure of *Pinus elliottii* wood treated with titanium dioxide to the fungus *Postia placenta* and photodegradation

Exposición de la madera de *Pinus elliottii* tratada con dióxido de titanio al hongo *Postia placenta* y fotodegradación

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ABSTRACT

It was impregnated commercial titanium dioxide into *Pinus elliottii* wood, aiming to increase its durability against the attack of brown rot fungus *Postia placenta* and photodegradation caused by ultraviolet radiation. The samples were put under 8 bar pressure for 3 hours at different concentrations of TiO₂ (0.5%, 0.25%, 0.124% and 0%-control). To evaluate the effect of the fungus on the wood, the test was carried out according to ASTM D2017-05 and UNE-EN 113:1996 with modifications. The photodegradation was performed by exposing a tangential section to ultraviolet radiation for 400 hours, and the colorimetric parameters were periodically evaluated. Statistically, the treatments with TiO₂ did not differ among themselves, but were much lower than the control, showing the effectiveness of this product in protecting the wood against the attacks of degraders. As for the photodegradation, the treated wood remained practically unchanged, differently from the control that had a darkening accelerated mainly in the first 50 hours. With this information, it can be stated that TiO₂ treated wood is able to hinder the fungus access to the cell wall, to inhibit its growth and to create a barrier that protects the polymers from photodegradation, increasing its durability and emerging as a potential alternative for wood treatment.

KEYWORDS: brown rot, TiO₂, ultraviolet radiation, wood treatment.

RESUMEN

Se impregnó dióxido de titanio comercial en la madera de *Pinus elliottii*, con el objetivo de aumentar su durabilidad contra el ataque del hongo pardo de la *Postia placenta* y la fotodegradación causada por la radiación ultravioleta. Las muestras se pusieron a una presión de 8 bar durante 3 horas a diferentes concentraciones de TiO₂ (0.5%, 0.25%, 0.124% y 0% de control). Para evaluar el efecto del hongo en la madera, la prueba se realizó de acuerdo con la norma ASTM D2017-05 y UNE-EN 113:1996 con modificaciones. La fotodegradación se realizó exponiendo una sección tangencial a radiación ultravioleta durante 400 horas, y los parámetros colorimétricos se evaluaron periódicamente. Estadísticamente, los tratamientos con TiO₂ no diferían entre sí, pero eran mucho más bajos que el control, mostrando la efectividad de este producto en la protección de la madera contra los ataques de los degradadores. En cuanto a la fotodegradación, la madera tratada se mantuvo prácticamente sin cambios, a diferencia del control que tuvo un oscurecimiento acelerado principalmente en las primeras 50 horas. Con esta información, se puede afirmar que la madera tratada con TiO₂ puede dificultar el acceso del hongo a la pared celular, inhibir su crecimiento y crear una barrera que protege a los polímeros de la fotodegradación, aumentando su durabilidad y emergiendo como una alternativa potencial para tratamiento de la madera.

PALABRAS CLAVE: podredumbre parda, TiO₂, radiación ultravioleta, tratamiento de la madera.

INTRODUCTION

According to Vidal et al. (2015), wood is a renewable material whose physico-mechanical and anatomical properties make it versatile for uses like buildings, structures, furniture, and various utensils. In relation to other materials, such as concrete, plastic, steel, and aluminum, wood has several advantages. Among these, it stands out its beauty, high mechanical resistance in relation to the mass, low energy consumption for its processing, good thermal insulation, and easy workability. However, according to the same authors, its disadvantages include being a combustible material when exposed to a heat source, affinity with water which leads to cracking and warping, and susceptibility to xylophagous insects and weathering.

The intensive wood exploitation and the growing concern with environmental issues put the harvesting of timber under certain restrictions. As an alternative to meet the demand for forest and timber products, homogeneous plantations with introduced species, such as those of the genera *Pinus* and *Eucalyptus*, have shown to be viable due to their rapid growth and interesting properties (Paes et al., 2005). However, these species tend to have lower natural durability than native ones traditionally used for lumber, requiring preservative treatments that make them more durable, bringing the quality closer to native ones (Silva, 2012).

According to Vivian (2011), the wood being an organic material is prone to deterioration due to the attack of degrading agents, such as: insects, borers, bacteria, and fungi. The latter are the most worrying biodegrading agents, since they consume wood quickly, and depending on the class, they can decompose it totally or only cause superficial damages (Rocha, 2001; Trevisan et al, 2008).

In this context, when the wood prone to this attack is absent from physicochemical treatments its applications become limited, which leads to the replacement by another material. The most used wood treatments in the Brazilian industry are carried out with CCA (chromated copper arsenate) and CCB (chromated copper borate) by pressure

methods (Vidal et al., 2015). These are effective in protecting the wood against the xylophagous insects, but the presence of toxic elements in their composition requires care in the wood applications and disposal.

Aiming at studies on more environmentally friendly methods and products that may replace the traditional treatments employed, it is believed that the use of nano/micrometric ceramics of low toxicity can be allied to wood preservation, since together demonstrate synergy and effective responses related to material protection (Sun et al. 2012; Filpo et al. 2013; Zheng et al. 2015). Zanatta et al. (2017), when studying the performance of wood treatment with nanoparticles, found that small amounts impregnated with TiO₂ are sufficient to protect the wood against fungal attack, matching the results obtained with CCB treatment. This result is consistent with Filpo et al. (2013), which confirms that this effect comes from the impregnation being deep in the wood material because of the particles' size.

OBJECTIVES

According to this information and with the intention of contributing to new works aimed at more environmentally friendly innovations in wood technology, the objective of this work was to analyze the resistance of *Pinus elliottii* wood impregnated whit commercial TiO₂ against brown rot fungus *Postia placenta* and ultraviolet photodegradation.

MATERIALS AND METHODS

In this study, pine sapwood of *Pinus elliottii* with approximately 22 years old was obtained from trees with a satisfactory phytosanitary appearance, rectilinear stem, and diameter at breast height greater than 30 centimeters. From these, boards were made, and samples were cut in dimensions of 1 cm × 1 cm × 0.9 and 1 cm × 1 cm × 2.5 cm, which were then placed in a room with controlled conditions (65% of relative humidity and temperature of 20 °C) until reaching equilibrium moisture content of 12%.

After reaching the specified condition, the wood samples were treated with Sigma Aldrich (99% purity) commercial TiO₂ (pure rutile phase) in distilled water in different



concentrations (0.125%, 0.25%, and 0.5%). The treatment was carried out in a laboratory autoclave (30 cm in length and 12 cm in diameter), where they received an initial vacuum of approximately 80 kPa to 93 kPa for 30 minutes, a pressure of approximately 790 kPa with the TiO₂ solution for 3 hours and final vacuum of 15 minutes. After this, the samples were exposed in a climatic room to stabilize the moisture content and for subsequent tests.

Postia placenta fungus rotting test

Woods treated at different concentrations and untreated (control), in a total of 158 samples (38 for each treatment + 6 correction blocks), were put in an oven at 50 °C until reaching constant weight. Subsequently, to obtain the initial weight, the samples were weighed using an analytical balance with an accuracy of 0.0001 g. After weighing, the samples were autoclaved at 121 °C for 45 minutes.

The fungus *Postia placenta*, which causes the brown rot disease, was obtained from the Wood Biodegradation and Preservation Sector at the Forest Products Laboratory (LPF, for its acronym in Portuguese) of the Brazilian Institute of Environment and Renewable Natural Resources (IBAMA, for its acronym in Portuguese), located in Brasília, DF. It was placed in Petri dishes containing culture medium (potato dextrose agar prepared with distilled water). After inoculation, the plates were taken to an incubator for mycelial growth, which was used as the inoculum source for the biological degradation test. This

test was conducted based on modifications of ASTM D2017-05 (American Society for Testing and Materials [ASTM], 2005) and European UNE-EN 113:1996 (UNE Normalización Española [UNE], 1996) standard methods.

Thus, in order to verify the treatment effect and compare it with the control, a 5mm mycelial disc was transferred to a petri dish with sterilized and solidified agar, and the wood sample was placed over the mycelial disc (Fig. 1), remaining in this condition for 16 weeks under 12 hours photoperiod. The light exposure was chosen regarding the TiO₂, as it has catalytic properties when absorbing UV radiation (present in fluorescent light) from the incubator chamber.

Every 15 days 4 samples of each treatment were removed from the experiment to verify the kinetics of degradation and the evolution of the fungus attack through mass loss. Likewise, at the end of the 16 weeks, the same procedure was performed with the remainder of the samples (10), i.e., the treated and untreated wood strength was obtained by drying the samples under the same conditions as before, using the results in the equation 1.

$$ML = \frac{(M_i - M_f)}{M_i} \times 100 \quad (1)$$

Wherein:

ML = mass loss

M_i = initial mass

M_f = final mass

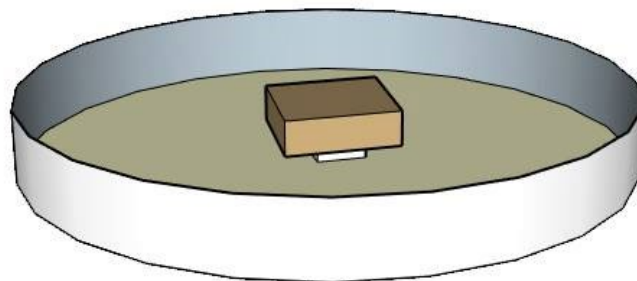


FIGURE 1. Scheme used for the biological test with fungus *Postia placenta*.

Photodegradation

The treated and untreated samples were subjected to accelerated aging (photodegradation) for a period of 400 hours. This experiment was executed in a black box coupled to an ultraviolet lamp (OSRAM-UVA-60Hz/9W/220V) as the radiation source with a wavelength of 315 nm - 400 nm. Color variation analysis was performed every 24 hours until completing approximately 100 hours, and after this color variable was measured every 100 hours.

The colorimetric variation obtained during the test was analyzed using a Konica Minolta portable colorimeter model CR-400. The device was configured to use a D65 light source and 10° viewing angle according to the International Commission on Illumination (CIE-L*a*b*). Two checks were performed under the radial plane, where it had a direct incidence of UV radiation. The colorimetric properties L* (lightness), coordinates a* (green-red color coordinate), b* (blue-yellow color coordinate), C* (saturation), h* (hue angle), and color saturation (C*) were obtained with the equipment software and the color variation (ΔE) by the equation 2.

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

wherein:

ΔE = difference in the test specimens' colors

ΔL , Δa , and Δb = variation of the parameters of lightness, green-red and blue-yellow color coordinates before and after UV radiation exposure

Statistical analysis

The data were analyzed in Statgraphics statistical analysis program, and the treatments were evaluated by analysis of variance, with later mean comparison by the Tukey test with a 5% significance level.

RESULTS AND DISCUSSION

Resistance of treated wood against fungus *Postia placenta*

The treated and untreated wood mass loss obtained at the end of the accelerated rotting test by the fungus *Postia placenta* is shown in Table 1. Statistically, there was no difference between the TiO₂ treatments, only between the treated and untreated woods, changing the resistance classification proposed by the standard ASTM D2017-05 from moderately to highly resistant. This result shows that all the concentrations applied significantly reduce the wood mass loss and that the lowest TiO₂ concentration (0.125%) is considered sufficient for this reduction, not differing from the others. Samples intended for moisture correction did not result in a mass loss at the end of the test, demonstrating that there was no influence of the methodology (modifications of the norms + environment with 12 hours photoperiod) used for the test.

Note that the mass loss for the lowest concentration of TiO₂ is lower than the other concentrations, even without statistical difference. The explanation for this better performance is related to the viscosity of the treatment.

TABLE 1. Mean values of mass loss of *Pinus elliottii* wood treated at different concentrations of commercial TiO₂.

| Treatment | Mass loss (%) | Standard deviation | Variation coefficient (%) | Classification ASTM D2017-05 |
|------------------------------|---------------|--------------------|---------------------------|------------------------------|
| PE + 0,5% TiO ₂ | 4,29 a* | ± 0,50 | 13,72 | Highly resistant |
| PE + 0,25% TiO ₂ | 3,71 a | ± 1,13 | 30,38 | Highly resistant |
| PE + 0,125% TiO ₂ | 3,66 a | ± 0,29 | 6,79 | Highly resistant |
| PE Control | 39,55 b | ± 3,73 | 9,42 | Moderately resistant |
| Value of F | 328,37 | | | |



With the addition of a larger amount of the product dissolved in water, as is the case of the 0.5% concentration, there is an increase in the impregnating solution viscosity, hindering the penetration of the material into the wood pores, resulting in a lower biological resistance. It should also be noted that the increase of the TiO_2 concentration in the solution can cause the formation of a physical barrier, obstructing the anatomical elements, and making it difficult to flow the solution inside the wood.

According to the Institute of Technological Research (2015) *Pinus elliottii* is a low-density wood species (400 kg/m^3) and highly susceptible to attack by xylophagous agents, including rotting fungi. These characteristics explain a deterioration of 39.55% of its total mass when absent from treatment and prove the preservative necessity for its applications, especially when used in outdoor environments. Carvalho et al (2015) also confirms this explanation when finding a mass loss of 54.61% for *Pinus* sp. when subjected to accelerated rotting with the brown rot fungus *Gloeophyllum trabeum*.

In a similar study, Mahr et al (2013) found satisfactory results on the titanium oxide effect on brown rot fungi (*Coniophora puteana* and *Poria = Postia placenta*) on *Pinus sylvestris* wood. For the treated wood, the average loss value was up to 5%, while for the control this loss was 38% to

50%. The efficiency of the treatment, according to the author, can be attributed to the formation of a hydrophobic characteristic on the wood surface by the ceramic material, which has a negative effect on fungal development, since it needs moisture to develop. Zanatta et al. (2017), when comparing the mass loss of the treated wood of *Pinus elliottii* with TiO_2 and with CCB, observed that TiO_2 did not show a significant difference for the CCB.

The treatments tested in this work increased the resistance of the *Pinus elliottii* wood to the fungus *Postia placenta* about 10 times. This shows that the impregnation treatment with TiO_2 is efficient and increases the wood resistance against the fungus. This behavior is similar to that seen by Zanatta et al. (2017), who found an increase in resistance of *Pinus elliottii* wood treated under pressure with TiO_2 particles synthesized by the microwave-assisted thermal method. Moreover, it is evident from studies by Mahr et al. (2013) and Filpo et al. (2013) that products based on TiO_2 demonstrate effectiveness when applied to and submitted to different types of wood and fungi.

A residual mass decrease of the test specimens was observed during the accelerated rotting test with the fungus chosen (Fig. 2). This residual parameter is inversely proportional to the mass loss, therefore, the higher the value, the more efficient the preservative treatment.

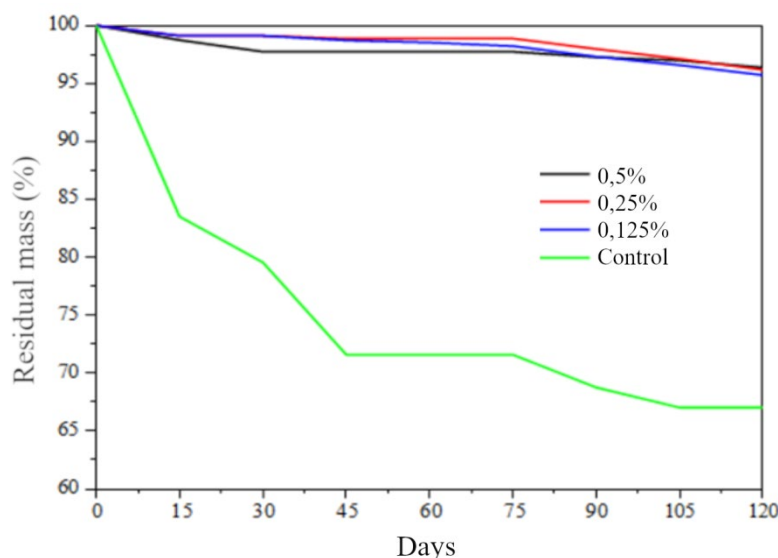


FIGURE 2. Behavior as a function of the residual mass of the *Pinus elliottii* wood during the accelerated rotting test with the fungus *Postia placenta*.

In the first 15 days of exposure to the fungus, the wood presented a significant decrease in its mass (approximately 16.5%), corresponding to the higher rate of degradation. With the treatments, it is possible to verify that such effect was minimized, and this was repeated in other samples, showing stability. For all TiO₂ treatments, a high residual mass was observed, ranging from 95% to 100% in all samples, differently from the control group which oscillates between 85% and 65%. These factors again indicate that treatment with these particles is capable of protecting the wood against the fungus attack.

This protection, coupled with the treatment method (pressure impregnation), occurred due to a physical barrier forced by the particles' presence between the fungus hypha and the wood cellular wall. The barrier prevents the hyphae from penetrating the wood cell wall and consequently does not allow the microorganism digestive enzymes to act on the degradation of the wood cell wall components. Another important characteristic of biological effects is that of photoactivity, i.e. with UV radiation (present in the fluorescent light photoperiod), through oxidative processes, TiO₂ prevents the development and the fungi attack in the wood (Filpo et al., 2013).

In a study on fungi development on wood treated with titanium alkoxides, Filpo et al (2013) visually noted that there was no fungal development. The authors emphasized that the TiO₂ photoactivity is sufficiently high even under daylight because, under these conditions, there is UV radiation that allows the prevention of fungal activity in the wood, as in this experiment.

Mattos and Magalhães (2017) evaluated the wood of *Pinus* sp. treated with Al₂O₃ loaded with carbendazim exposed to the fungi *Trametes versicolor* and *Gloeophyllum trabeum*, finding a 10 and 20-fold improvement in resistance to rotting against the fungi, respectively. Peres (2017), when evaluating the resistance of the *Pinus elliottii* wood to the fungus *Ganoderma applanatum* after treatment with zinc oxide (ZnO) nanoparticles dispersed in water, found a mass loss of 11.9%, 9.3%, and 8.7% for concentrations of 1%,

2.5%, and 5%, while for control this was 25.8%. The author emphasizes that the photocatalytic characteristic of this ceramic is the main response to the antifungal action.

Photodegradation

Table 2 shows the mean values of the colorimetric parameters L*, a*, b*, C* and h° for the treated and untreated *Pinus elliottii* wood before and after 400 hours of UV radiation exposure, which showed difference between treatments.

According to the species description made by the Institute of Technological Research (2017) the *Pinus elliottii* wood is considered yellowish white. For Camargos (1999) the genus *Pinus* is classified as light-colored, as it has L* values higher than 54. In this work, it was noticed that in general, all the treatments surpassed this one, confirming this classification.

Figure 3 shows the graphs corresponding to the colorimetric parameters L*, a*, b*, and ΔE, respectively, in different periods.

The coordinate L* represents the wood lightness and its decreasing behavior corresponds to its darkening. When the wood is exposed to weather, such as UV radiation, it may become greyish, whitish, yellowish, reddish-orange, or brown (Kamdem & Grelier, 2002). In relation to this parameter, a decreasing trend was observed in the L* chart. In the first 50 hours of UV radiation exposure, the decrease in L* values was more pronounced, especially for the untreated samples. Those treated with TiO₂ showed resistance to darkening, even at the beginning of the test, where it did not decline linearly.

The photodegradation, according to Ayadi et al. (2003) and George et al. (2005), occurred mainly due to lignin degradation, a phenolic compound that has chromophore groups that are oxidized and absorbed by ultraviolet rays. The conifers, having a greater presence of lignin in their structure when compared to hardwoods, have a faster colorimetric variation, tending to yellow or brown in the first hours of exposure (POUBEL et al., 2017).



TABLE 2. Mean values of the colorimetric parameters of *Pinus elliottii* wood.

| Treatments | | L* | a* | b* | C | h° |
|------------------------------|---------|-------------------|------------------|-------------------|-------------------|-------------------|
| PE + 0,5% TiO ₂ | Initial | 80,75b* (2,24) | 5,02a (0,70) | 21,13a (3,06) | 21,20a (3,12) | 76,25a (1,07) |
| | Final | 77,55BC (1,89) | 6,36A (0,57) | 21,37A (1,59) | 22,29A (1,66) | 73,43A (0,75) |
| PE + 0,25% TiO ₂ | Initial | 77,93a (2,84) | 5,94b (2,25) | 26,00c (3,39) | 26,65c (3,74) | 77,44ab (3,59) |
| | Final | 73,34A (3,60) | 8,17B (2,61) | 26,18B (2,97) | 27,48C (3,53) | 72,9A (3,69) |
| PE + 0,125% TiO ₂ | Initial | 83,34c (2,67) | 3,79ab (1,13) | 23,98b (2,70) | 23,76b (2,79) | 80,91c (2,10) |
| | Final | 79,00C (2,50) | 5,52A (1,18) | 24,33B (2,70) | 24,96B (2,85) | 77,3B (1,71) |
| PE Control | Initial | 81,44bc (3,98) | 4,90ab (2,20) | 24,77bc (2,07) | 25,11bc (2,44) | 79,01bc (3,98) |
| | Final | 76,32BC (3,90) | 6,72A (1,78) | 26,03B (1,49) | 26,92BC (1,81) | 75,6B (3,05) |

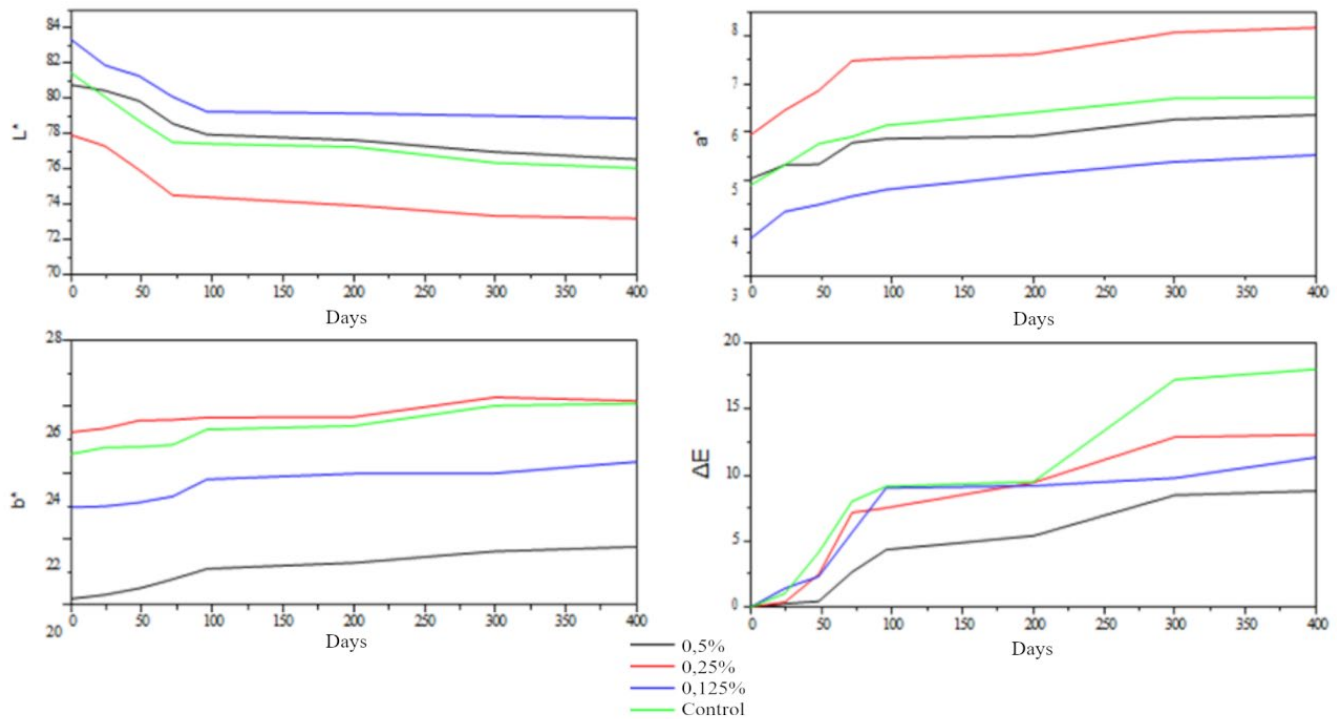


FIGURE 3. Graphs of the colorimetric parameters L*, a*, b*, and ΔE, respectively, during exposure to UV radiation

For a^* (green-red color coordinate) the values showed an increasing tendency to stabilize after about 48 hours of exposure. This behavior was observed by Martins et al. (2011), who evaluated the surface color photodegradation of the *Pinus caribaea* var. *bondurensis* and *Eucalyptus benthamii* to ultraviolet radiation. Poubel et al. (2017) found a direct and significant correlation between the extractive content and this coordinate, indicating that the higher the wood extractive content, the greater the value of this coordinate, giving the wood a reddish color.

In relation to the control, in the coordinate a^* chart the treatment with 0.125% of TiO_2 began the color stabilization before the 50 hours with a more or less abrupt reduction in the period of the colorimetric modification observed for the control at the same time interval.

The b^* values (blue-yellow color coordinate) increased during the initial period of UV radiation exposure, showing that there is a predominance of yellow color. This is a *Pinus* wood property and is directly related to the photochemistry of wood components, mainly lignin (Gierlinger et al., 2004; Pincelli et al., 2012). This explains the observed in the control, which increases to a greater degree its values when compared to those treated with TiO_2 that had a reduced variation by the oxide application.

As for C (saturation), there was an increase in values characterizing a higher color saturation, while for h° (hue angle) the values showed a decreasing behavior. The h parameter, in general, had a reduction of the average value during the UV radiation exposure, demonstrating that the wood had darkened. According to Pincelli et al. (2012), the reduction of h° implies the homogenization of the wood color after the thermal rectification, while in this work occurred after the UV radiation exposure with TiO_2 treatment.

It can be seen from the ΔE graph (color variation) that the total colorimetric variation for the control is higher than the treated wood at any concentration tested, with a double variation in the treatment at 0.5% TiO_2 . It is observed that the control has enormous variation in the first 50 hours of

UV radiation exposure, which is not prominent for the treated test specimens.

According to Tolvaj and Faix (1995), the colorimetric variation (ΔE) occurs with greater intensity in the first 50 hours of UV exposure, and in this period, the variation corresponds to 70% of the total colorimetric variation that happens during all the exposure time, which explains the behavior, especially of the control, during the experiment.

Regarding the difference in total color variation for the treated wood and the control, Poubel et al. (2017) explains that due to the strong shielding capacity against the UV rays by the metallic nanoparticles, these have a great impact on the wood photoprotection, thus reducing the total color variation values (ΔE).

Compared with existing treatments, the heat treatment is widely used to modify the wood color making it similar to valuable dark wood (Weiland & Guyonnet, 2003). However, this type of treatment, in some cases, affects some wood properties, physical and mechanical, due to the high temperatures, which does not occur in this treatment.

According to Blanchard and Blanchet (2011) ultraviolet radiation, in general, tends to darken the wood. But in their study when impregnating zinc oxide, the authors found that this protects the cell wall from deterioration by UV radiation and reduces the darkening.

Poubel et al. (2017), when impregnating zinc oxide nanoparticles in *Pinus* sp. wood evaluated its resistance to UV radiation and found values similar to this work for all colorimetric coordinates and characterized the oxide as a good preservative for this purpose. Cai (2007) noticed that the durability of an outdoor wood was increased after the TiO_2 nanoparticles application. These results showed that TiO_2 protects the wood against deterioration caused by UV radiation at all concentrations used, mainly at 0.5%.

Thus, it is observed the need for research aiming at obtaining wood preservatives that also prevent the degradation of its cellular component by ultraviolet radiation, being the treatment with TiO_2 a promising alternative, due to its protection property and low toxicity reported in the literature.



CONCLUSIONS

The wood treatment with TiO₂ particles is efficient for the preservation of the *Pinus elliottii* wood against the brown rot fungus *Postia placenta* attack and to the photodegradation, without altering its natural colorimetric characteristics.

Minimal concentrations of titanium dioxide are sufficient to protect the *Pinus elliottii* wood from the fungus *Postia placenta*, but higher concentrations are required to protect it against ultraviolet radiation.

With the demand for developing of new products with a lower environmental impact, TiO₂ is a promising product to be used in the wood technology area, standing out as a possible alternative to the existing ones.

ACKNOWLEDGEMENTS

The authors thank CAPES, CNPq [151833 / 2018-9] and FAPERGS

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Received: 21 November 2018

Accepted: 14 March 2019

Published: 26 July 2022

This paper must be cited as:

Zanatta, P., Mattoso, T., Gallio, E., Lazarotto, M., Gatto, D. A., Cunha, A. B., & Beltrame, R. (2022). Exposure of *Pinus elliottii* wood treated with titanium dioxide to the fungus *Postia placenta* and photodegradation. *Madera y Bosques*, 28(1), e2811895. <https://doi.org/10.21829/myb.2022.2811895>



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