Artificial Weathering of Heat-treated Pines from the Iberian Peninsula

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Samples from the two most common pines grown in Portugal (*Pinus pinaster* Ait) and Spain (*Pinus radiata,* D. Don) were heat-treated in industrial facilities in accordance with ThermoWood ® class D. For both species, the variation in surface properties, of untreated and heat-treated wood after artificial weathering from 75 to 750 h, is presented. The analysis included the determination of color, roughness, gloss, and wettability before exposure and after each artificial weathering period. Untreated woods became darker faster, while in heat-treated woods, lightness remained approximately constant until 750 h of artificial weathering. Both untreated and heat-treated wood became more reddish in the beginning of the weathering process, turning greener for longer exposure times. Untreated woods became yellower in the beginning, turning into blueish tones later. Heat-treated wood turned slightly yellower until 750 h of weathering. Gloss decreased for untreated wood with no significant changes in heat-treated wood. Despite the changes, the gloss of both untreated and heat-treated wood converged to similar values. Roughness increased for both untreated and heat-treated woods. Artificial weathering increased the wettability of heat-treated wood.

Keywords: Artificial weathering; Heat treatment; Pinus pinaster; Pinus radiata

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INTRODUCTION

Heat treatment is a well-known modification procedure that increases wood stability and durability (Esteves and Pereira 2009). One of the most successful commercial processes is the Thermowood® process, which started in Finland but is now applied in several countries including Sweden, Turkey, Japan, Spain, and Portugal. The treatment is done with steam, with less than 3 to 5% oxygen, without pressure, and with a minimum air speed of 10 m/s. The process begins with a rapid increase in temperature of the oven with heat and steam up to 100 °C, followed by a gradual increase up to 130 °C to near zero humidity. Then, heat treatment is made at a chosen temperature between 185 °C to 230 °C for 2 to 3 h. Finally, the temperature decreases to 80 to 90 °C (Mayes and Oksanen 2002).

Deterioration by abiotic agents such as solar radiation, rain, snow and sleet, wind and humidity changes are generally linked to weather exposure conditions (Feist *et al.* 2007). These atmospheric agents can cause color changes as well as chemical, physical, mechanical, and anatomical changes on the surface of the wood. These changes occur on the surface to a depth of 0.05 to 2.5 mm during the initial period.

Solar radiation affects wood due mainly to the UV component, which causes photochemical damage in lignin and extractives. UV degradation is driven by free radicals formed by the oxidation of phenolic hydroxides (Buchner *et al.* 2019; Derbyshire and Miller 1981; Feist *et al.* 2007)

Mechanical damage on the surface is a result of retraction and swelling of the wood, which may also lead to cracks. For instance, Xing *et al.* (2015) stated that small cracks in untreated and heat-treated wood could be observed by SEM analysis. In accordance to Yildiz *et al.* (2013), effects relative to mechanical properties depend on the species, and generally hardwood species behave better than softwood with respect to weathering.

With the leaching of soluble compounds, the most photo-resistant compounds are exposed in the surface, leading to their degradation. With time, the wood acquires a greyish tone due to the residual cellulose and blue stain fungal growth (Feist *et al.* 2007; Oberhofnerová and Pánek 2016).

There have been several attempts to predict service life of wood above ground, such as the work of Meyer-Veltrup *et al.* (2017), taking into account wood species, their durability classes, wetting abilities, temperature, and moisture content, as also mentioned by Oberhofnerová and Pánek (2016). One difficulty in estimating wood service life in Europe is the variability of climates between regions, particularly the average number of sunshine hours per year. Cities such as Lisbon, Madrid, or Athens show an average well above 2500 h of sunshine per year, far superior to the values measured on Northern European countries, which are typically bellow 1800 h per year. The expected level of degradation by UV radiation is thus significantly higher in Southern Europe, though most wood modification developments were initially targeted to Nordic countries. On the other hand, the precipitation levels are higher in Northern, compared to Southern Europe, and the differences are increasing (Van den Besselaar *et al.* 2013).

Thermally treated wood has a very different chemical composition than the original wood. The percentage of hemicelluloses is much lower, but lignin levels increase significantly (Alén *et al*. 2002; Windeisen *et al*. 2007; Esteves *et al*. 2008). Because UV radiation mainly attacks lignin, heat-treated wood might be more affected by UV radiation. However, the color variations of treated wood subject to UV are smaller than the variation in untreated samples (Ayadi *et al*. 2003; Deka *et al*. 2008; Miklecic *et al*. 2011), which according to Ayadi *et al.* (2003) is due to the stabilization of lignin during heat treatment. Nuopponen *et al*. (2005) reported that treated wood is more resistant when exposed to weather conditions because the degradation products of lignin are less leached than those of untreated wood. This might be due to the fact that during heat treatment, low molecular weight water extractable components react to produce insoluble high molecular weight compounds. The color variation depends heavily on the initial color of the wood. Typically, darker woods (such as heat-treated wood) become lighter, while lighter woods become darker initially and then lighter when they acquire a gray/silver tone. This phenomenon has been described by several authors (Temiz *et al*. 2006; Olărescu *et al*. 2014; Kucuktuvek *et al*. 2017). Xing *et al.* (2015) stated that the color changes of the wood surface are due to the removal of extractives and the modification of lignin.

One of the great advantages of thermally treated wood with regard to resistance to atmospheric agents is that they suffer less swelling and shrinkage with variations in wood moisture content. In accordance to Humar *et al.* (2020), thermally modified wood during the course of weathering has lower moisture content in comparison to untreated Norway spruce.

This work was done in the framework of the project PROJ/CI&DETS/2016/0010-Determination of resistance of thermally treated wood to weather conditions in different countries (HTW). The goal of this project was to study the degradation of thermally modified wood by Thermowood® process in countries where solar radiation is higher, *i.e.*, Portugal and Spain, and compare it to accelerated weathering. The objective of this part of the work was to determine the changes on the surface of the most used heat-treated pines from the Iberian Peninsula after artificial weathering.

EXPERIMENTAL

Materials

Pine samples from the two most common pines grown in Portugal (*Pinus pinaster* Ait) and in Spain (*Pinus radiata* D. Don) were treated in industrial facilities at 212 °C in accordance to the Thermowood ® process using Thermo D class specifications. Boards were cut into samples of approximately 150 mm x 50 mm x 10 mm.

Methods

Artificial weathering

Seven specimens (150 mm x 50 mm x 10 mm) for each treated/untreated wood were positioned in a QUV accelerated weathering tester (Q-LAB, Homestead, FL, USA) from 75 h to 750 h. The measurements were made at 0 h, 75 h, 150 h, 300 h, 450 h, 600 h, and 750 h of artificial weathering. This chamber replicates the damage caused by sunlight and dew. The exposure to UVA lamps followed the conditions of the cycle n°1 (method A) of EN ISO 16474-3 (2013). The specimens were cycled through periods of UV radiation exposure followed by periods of no radiation, during which temperature changes occur. The cycle consisted of 4 h of dry UV exposure at a black-standard temperature of (60 ± 3) °C followed by 4 h of condensation exposure, without radiation, at a black-standard temperature of (50 ± 3) °C.

Color determination

The color was analyzed in a portable spectrophotometer COLOREYE® XTH (Gretag Macbeth, Grand Rapids, MI USA), before and after being exposed to accelerated weathering. Color parameters were determined using an average of three measurements made in the weathered surface (Fig. 1) by the CIELAB system. This system is composed of three parameters: L^* that represents lightness and varies from 100 (white) to zero (black), and two color tones (a^* and b^*), where a^* goes from red (+*a*) to green (-*a*), while b^* goes from yellow (+*b*) to blue (-*b*).

Gloss determination

Gloss was determined parallel to the wood fibers with a gloss meter REFO-3D (DR Lange, Düsseldorf, Germany) with three-angle geometry 20°, 60°, and 85°. Gloss was determined for untreated and heat-treated maritime pine and radiata pine woods before and after artificial weathering using an average of three measurements made in the surface.

Roughness determination

Roughness was measured in a Surftest SJ-400 (Mitutoyo, Illinois, United States). Average roughness (R_a) and the maximum height of the roughness profile (R_z), calculated as the average distance between the highest peak and lowest valley in each sampling length, were determined for both untreated and heat-treated woods before and after artificial weathering.

Wettability

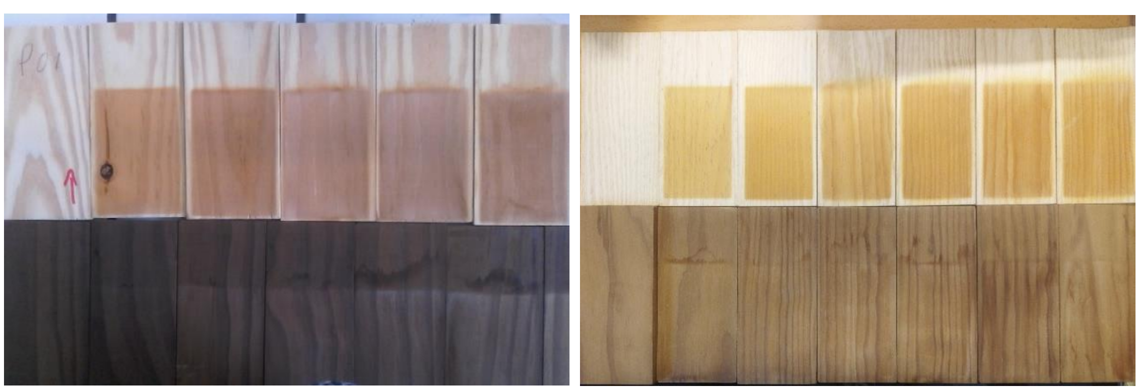
The wettability was determined by measuring the contact angles using the sessile drop method in a Contact Angle System OCA20 (DataPhysics Instruments, Filderstadt, Germany), a video-based measuring device equipped with software for image analysis. The measurements were performed parallel to wood fibers, and distilled water was used as probe liquid. The droplet volume was 4 μL. Contact angles were measured during approximately 50 s, allowing the droplets to reach equilibrium. The initial value was used.

Statistical analysis

Statistical analysis was performed using IBM® SPSS® V26 edition. Two-way ANOVA was made to test if there was a difference between heat treatment and weathering for color (L^*, a^*, and b^*), gloss (20º, 60º, and 85º) and roughness (R_a and R_z) for *Pinus pinaster* and *Pinus radiata* woods. One-way ANOVA was done for each untreated and heat-treated wood during weathering. Average and standard deviations were determined whenever possible.

RESULTS AND DISCUSSION

Figure 1 presents the surface changes of untreated and heat-treated maritime and radiata pine in the course of the artificial weathering process. As expected, untreated woods become darker, even with only 75 h of weathering (second sample), during the weathering process. The differences, for heat-treated samples, were more difficult to track visually. There was a slight color change along the treatment, which was clearly more pronounced for heat-treated maritime pine, probably due to its initial darker color.



(a) (b)

Fig. 1. Changes due to artificial weathering in the surface of (a) *P. pinaster* (b) *P. radiata*. Untreated (first row) and heat-treated (second row).

Almost no differences were seen with respect to the color of heat-treated radiata pine. As expected, the color variation during the weathering process depends on the initial color of the wood. The darkest wood—maritime pine heat-treated wood—became lighter, whereas the lighter untreated pine woods became darker. Similar results were presented previously (Temiz *et al*. 2006; Olărescu *et al*. 2014; Kucuktuvek *et al*. 2017).

Figure 2 presents the average lightness in the course of the weathering process for untreated and heat-treated maritime and radiata pine. Standard deviation is shown as error bars in the figure. For untreated maritime pine, lightness (*L**) decreased in the first 150 h of weathering from 76 to about 65, staying approximately constant until about 600 h, decreasing afterwards to about 54. The behavior of untreated radiata pine *L** was similar, although there was a higher decrease between 450 and 600 h of weathering, reaching a minimum of 50. With 750 h, the *L** value was very similar to that of maritime pine, 52. Standard deviation for untreated and heat-treated woods *L** is very small showing low dispersion of values. ANOVA results presented in Table 2 show that *L** is significantly different during weathering for untreated woods.

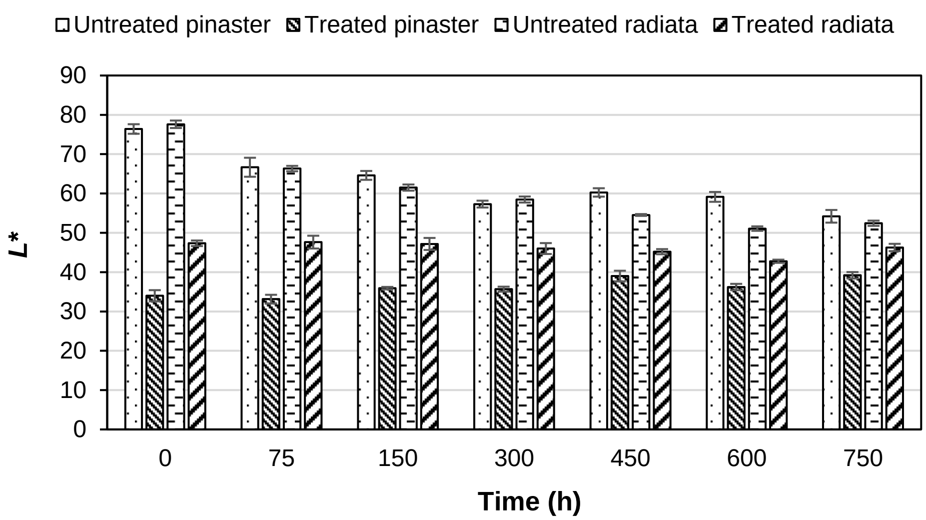
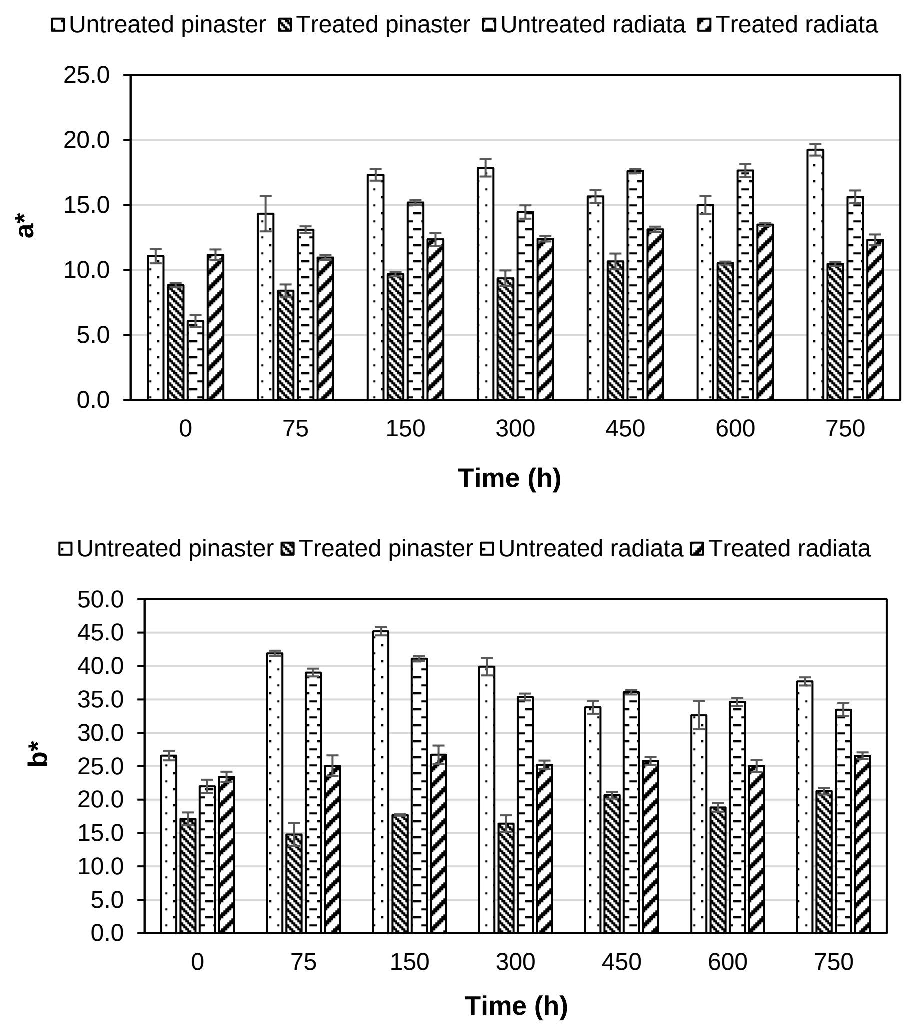


Fig. 2. *L** variation in the course of the weathering process for untreated and heat-treated maritime and radiata pines

Heat-treated radiata pine *L** was almost the same during the weathering process, ranging from 48 to 43. Nevertheless, there is a statistically significant difference during weathering, which only shows that is at least one average that differs significantly from the remaining (Table 2. In relation to heat-treated maritime pine, there was an increase in lightness in the course of the weathering process from the initial 34 to the final 39. This difference can be seen in Fig. 1. Initial heat-treated radiata pine was lighter than heat-treated maritime pine with lightness 47 and 34, respectively. However, the difference between the lightness of both woods decreased as a consequence of the weathering process. There was a similar trend between untreated and heat-treated pines, where the differences in lightness decreased with weathering. The results show that heat-treated wood lightness is more stable with the weathering process than that of untreated wood, as described before (Ayadi *et al*. 2003). Nevertheless, this higher stability might be due also to the initial lightness of heat-treated wood, which is much darker than untreated woods. The increase in lightness for heat-treated wood was described for jack pine (Kocaefe *et al*. 2013), Oriental beech (Turkoglu *et al*. 2015), Scots pine, spruce, iroko, and ash (Yildiz *et al*. 2013). However, Gonzalez de Cademartori *et al.* (2015) exposed three fast growing eucalyptus to natural weathering for 360 days and concluded that lightness decreased.

Artificial weathering mostly affects lignin. Even though heat-treated wood has more lignin than untreated wood, the higher resistance of heat-treated wood may be due to lignin stabilization during heat treatments (Ayadi *et al*. 2003). Polyphenols (lignin and tannins) react at high temperature, by self-condensation or by copolymerization with other compounds that are produced in the heat treatment. It is expected that, if the weathering is prolonged, lightness will be very similar regardless of the initial values (Huang *et al*. 2012a), and that was verified in the present study.

The parameter *a** increased for both untreated pines in the beginning of the weathering process until about 300 h for maritime pine and 400 h for radiata pine, decreasing later on until 750 h. The values for *a** after 750 h were higher than the initial ones, indicating some reddening of the samples. In relation to heat-treated woods, *a** increased until 450 h of weathering, staying approximately constant afterwards (Fig. 3).



(a) (b)

Fig. 3. Parameters *a** and *b** variation in the course of the weathering process for untreated and heat-treated maritime and radiata pine

The *b** parameter followed a similar pattern, with an increase followed by a decrease with weathering for untreated woods and a slight increase with weathering for both heat-treated woods. All final *b** values were higher than initial ones, showing a yellowing of the surface (Fig. 3). All *a** and *b**, for untreated and heat-treated wood, were considered to be statistically different with weathering, as can be seen in Table 2. Similar results were presented for heat-treated Scots pine aged for 500 h with an increase in both *a** and *b** (Kucuktuvek *et al*. 2017).

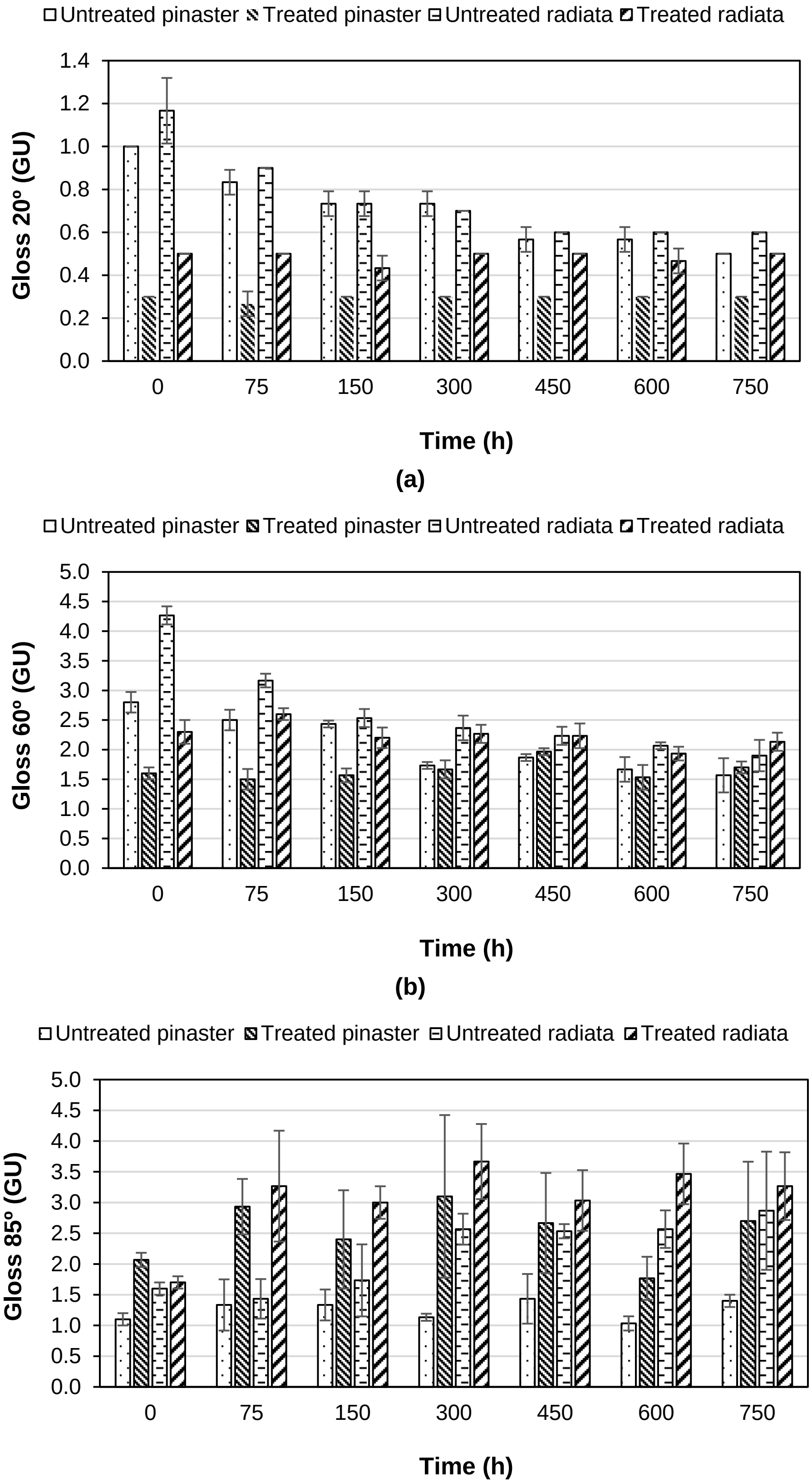
Different results were reported for heat-treated Oriental beech (Turkoglu *et al*. 2015), jack pine (Kocaefe *et al*. 2013), and for three different eucalyptus woods exposed to natural weathering, which all showed a decrease in both *a** and *b** (Gonzalez de Cademartori *et al.* 2015). According to Yildiz *et al* (2013) artificial weathering for 400 h, 600 h, and 1200 h produced greenish and bluish surfaces on heat-treated Scots pine, spruce, iroko, and ash wood. The effect was higher for heat-treated pine and iroko samples. These different results might be due to the initial color of untreated woods but also to the number of hours of weathering.

Figure 4 presents the glossiness variation along the weathering process for untreated and heat-treated wood measured at 20°, 60°, and 85° angles. At 20° and 60°, the gloss of heat-treated wood was lower than untreated woods, as reported before (Ayata *et al*. 2017a,b; Esteves *et al*. 2019). For untreated wood, glossiness decreased with the weathering process until about 400 h of weathering, staying approximately constant afterwards.

Although radiata pine had a higher initial gloss than maritime pine, both pines had similar gloss after 400 h of weathering. This decrease was seen on the 20° and 60° angles. The statistical significative difference was confirmed by one-way ANOVA in Table 2. The correct angle for glossiness determination should be 85° due to the matte surface presented by the untreated and heat-treated samples with glossiness lower than 30 GU. However, the values for 85° have a very high dispersion, as can be seen by the high standard deviations on Fig. 4c. This can be due to surface irregularities that usually decrease the measured value of gloss, especially at higher angles (Bekhta *et al*. 2014).

Regarding heat-treated wood at 20°, there were few changes along the weathering process, which is in accordance with ANOVA tests that show no significant differences in the course of weathering (Table 2). At 60°, there was a small decrease of gloss for heat-treated radiata pine and a little increase for heat-treated maritime pine, which was enough, however, to consider the differences during weathering significantly different (Table 2). Despite of all the changes, the gloss of both untreated and heat-treated wood converged to similar values in the course of the weathering process, mainly at 20° and 60°.

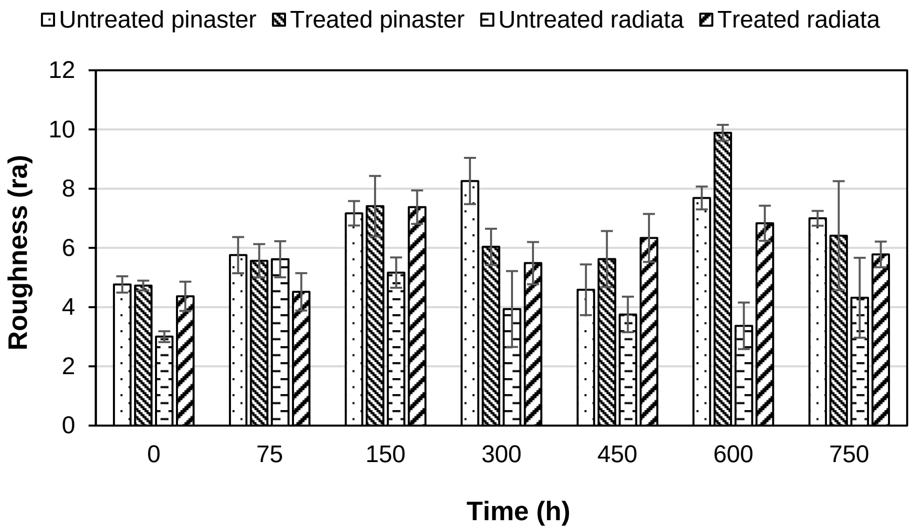
There are conflicting reports on the gloss changes due to artificial or natural weathering. Kucuktuvek *et al*. (2017) noted that heat treatments at 210 °C and 220 °C increase the surface gloss of Scots pine after weathering. Turkoglu *et al*. (2015) exposed Oriental beech to natural weathering for three months, demonstrating that gloss decreases for aged heat-treated wood and that this decrease is higher than in untreated wood. Similar results were presented for heat-treated Scots pine after accelerated weathering (Baysal *et al*. 2014); the changes in gloss were lower for heat-treated wood, similar to the results presented here.



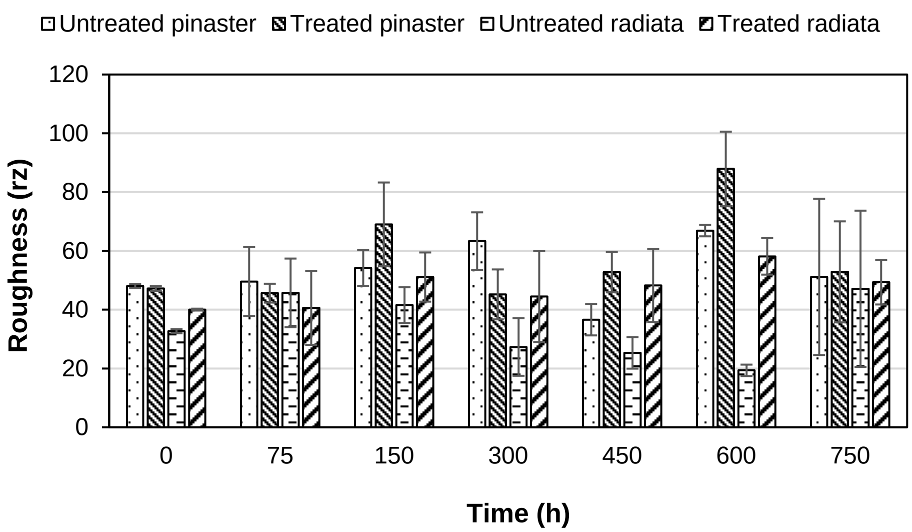
(c)

Fig. 4. Gloss at 20° (a), 60° (b) and 85° (c) variation in the course of the weathering process for untreated and heat-treated maritime and radiata pine

The roughness variation during the weathering process is shown in Fig. 5. For untreated wood, R_a increased in the beginning until about 300 h of exposure, decreasing slightly after that though with some small variations. Overall, roughness (R_a) increased for untreated wood. For heat-treated wood, there was a general increase in roughness for both heat-treated maritime and radiata pines. All the differences of R_a in the course of weathering were considered to be statistically different during weathering (Table 2).



(a)



(b)

Fig. 5. Changes due to artificial weathering in the surface roughness of untreated and heat-treated maritime and radiata pine. R_a values (a) and R_z values (b).

Since R_z values represent the maximum peak to valley height of the profile, their values can be influence by several irregularities in the samples. Nonetheless R_z exhibited a trend similar to R_a values with the exception of treated radiata pine, where the difference during weathering was not statistically significant (Table 2). The higher standard deviations of R_z values (Fig. 5b) in relation to R_a values (Fig. 5a) show that R_z values have a higher dispersion of values than R_a. Similarly, Turkoglu *et al.* (2015) stated that roughness increased for both untreated and heat-treated Oriental beech. Kucuktuvek *et al.* (2017) tested untreated and heat-treated Scots pine at temperatures between 210 to 230 °C before and after natural weathering for 6 months, concluding that roughness increases with weathering for both untreated and heat-treated wood nevertheless the increase was higher for untreated wood. Baysal *et al.* (2014) reported an increase in R_z but a decrease in R_a parameter for Scots pine roughness after artificial weathering. Yildiz *et al*. (2013) determined the R_z roughness value for heat-treated Scots pine, spruce, iroko, and ash and concluded that roughness depended on the species. While R_z values tended to decrease after weathering for Scots pine, in spruce there was a decrease followed by an increase. For iroko and ash, there was a clear increase along the weathering process.

Figure 6 presents the changes due to artificial weathering in the contact angle (wettability) of untreated and heat-treated woods. In untreated woods, there were some oscillations in the contact angle with weathering, but there were no observable changes in relation to initial values. Therefore, weathering did not greatly change the wettability of the untreated wood surface. In treated woods, there was a clear decrease in the contact angle measured in the surface, which corresponded to an increase in wettability. This increase was observed for both heat-treated pines. For 750 h the contact angle decreased for under 20° for radiata pine and for about 50° for maritime pine wood. The initial contact angle was higher for heat-treated maritime pine wood. Parallel results were reported in three different eucalypt species where wettability increased for all heat-treated samples (Gonzalez de Cademartori *et al*. 2015) or with jack pine heat-treated at 210 °C (Kocaefe *et al*. 2013). The reasons for this increase in wettability were mentioned by Gindl *et al.* (2006) to be due to the cleaning of wood surface done by UV irradiation that increased both the wettability and surface free energy. However, Huang *et al.* (2012b) noted that this increase is possible due to the formation of cracks during weathering, which they confirmed by scanning electron microscopic analysis.

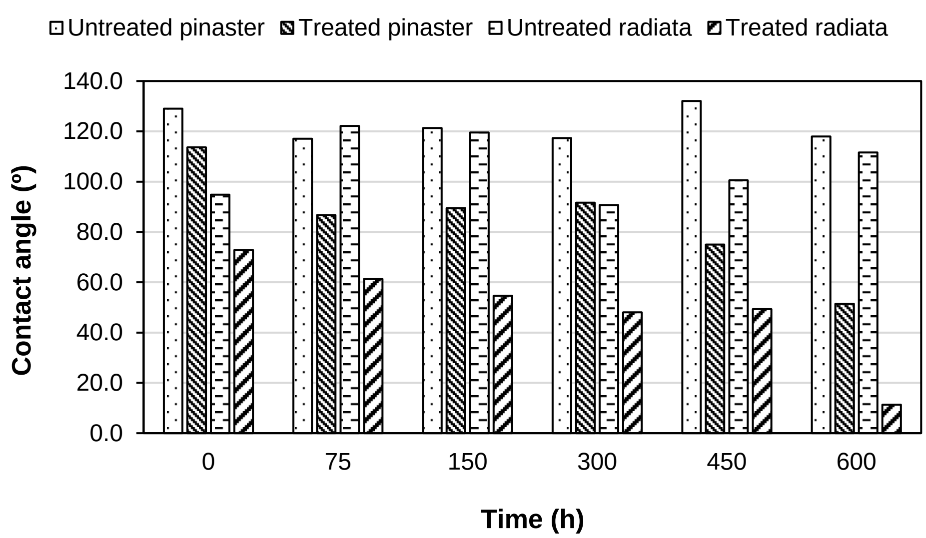


Fig. 6. Changes due to artificial weathering in the contact angle (wettability) of untreated and heat-treated maritime and radiata pine

Table 1 presents the results for an analysis of variance (ANOVA) of color, gloss and roughness parameters with heat treatment and weathering fixed factors (only interaction significance level is presented). Results show that interaction between both factors is significant for all the variables with the exception of 85º Gloss for *Pinus pinaster*. The non-significance of 85º gloss is most probably due to the high dispersion of results for this angle, as can be seen by the high standard deviation on Fig. 4. Therefore, and because there was a high significance level for the cross-effects (heat treatment x weathering level), single effects must be evaluated. These effects are presented in Table 2.

Table 1. Two-way ANOVA for Color, Gloss, and Roughness Parameters with Heat Treatment and Weathering Fixed Factors for Pinaster and Radiata Woods – Interaction Significance Level (p-value)

Test	*L**	*a**	*b**	Roughness (R_a)	Roughness (R_z)	Gloss 20º	Gloss 60º	Gloss 85º
Source	Treat * Weathering time							
P. pinaster	0.000	0.000	0.000	0.002	0.008	0.000	0.000	0.616
P. radiata	0.000	0.000	0.000	0.001	0.042	0.000	0.000	0.050

Table 2 presents the results for a one-way analysis of variance (ANOVA) to study the effects of weathering on color, gloss and roughness parameters. The color parameters were considered statistically significantly different for all untreated and heat-treated woods during weathering. Nevertheless, although statistically significant, the differences for heat treated woods for *L** were very small, as can be seen in Fig. 2. Gloss at 20º was statistically different for the untreated samples, but not for the heat-treated ones. This reinforces what was said before. Untreated wood gloss at 20º remained approximately constant in the course of the weathering process. Although gloss at 60º was significantly different during weathering, the significance values show that this was probably due to one or two means that are different from the others (Fig. 4b). In relation to the 85º, results are most likely influenced by the high dispersion of values as seen in Fig. 4c. Roughness was significantly different for untreated and heat-treated samples during weathering, except for R_z values of untreated and treated *Pinus radiata* woods.

Table 2. One-way ANOVA for Color, Gloss, and Roughness Parameters with Weathering for *Pinus pinaster* and *radiata* Woods

Weathering	Significance level (p-value)			
Test	Untreated *P. pinaster*	Untreated *P. radiata*	Treated *P. pinaster*	Treated *P. radiata*
*L**	0.000	0.000	0.000	0.002
*a**	0.000	0.000	0.000	0.000
*b**	0.000	0.000	0.000	0.017
Gloss 20º	0.000	0.000	0.463	0.109
Gloss 60º	0.000	0.000	0.017	0.009
Gloss 85º	0.350	0.001	0.414	0.013
Roughness (R_a)	0.000	0.021	0.000	0.000
Roughness (R_z)	0.001	0.080	0.002	0.370

CONCLUSIONS

1. With artificial weathering, lightness (L^*) decreased, staying approximately constant afterwards for untreated wood, while smaller changes were observed for heat-treated wood until 750 h.
2. Both untreated and heat-treated wood became more reddish (higher a^*) in the beginning of the weathering process, turning greener (lower a^*) for more severe treatments. At the same time, untreated woods became yellower (higher b^*) in the beginning turning into blueish tones later on (lower b^*). Heat-treated wood generally became yellower.
3. In general, roughness seems to increase with artificial weathering for both untreated and heat-treated wood.
4. Untreated wood glossiness seems to decrease with weathering, while heat-treated wood glossiness remained approximately constant.
5. There was an increase in wettability with artificial weathering, mainly for heat-treated woods.
6. Heat treatment gave some protection to artificial weathering in relation to color and glossiness but not in relation to wettability in the first 750 hours.

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