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DURABILITY IN OUTDOOR CONDITIONS OF COATING SYSTEMS BASED ON WATERBORNE ACRYLIC RESIN WITH COMMERCIAL UV ABSORBER AND TREE BARK EXTRACT

Environmentally friendly new wood preservative surface materials were developed to protect the wood surface against outdoor conditions. The durability performance of different types of waterborne acrylic resin with bark extract used as coatings on a wood surface exposed to outdoor conditions was investigated. Scots pine and Oriental beech surfaces were coated with three different waterborne acrylic resin coatings. Bark extracts from two different trees (fir and black pine) and a commercial UV absorber were used in the acrylic resin coating formulations. The durability of the coatings containing these bark extracts were compared with that of the coating with a commercial UV absorber. The wood samples coated with the test and control coatings were exposed to the QUV test in laboratory conditions. The surface colour and roughness changes on the wood samples coated with the test coatings and exposed to the accelerated weathering test were compared with the control samples. Microscopic images were taken, and the coating thicknesses of the control and test samples prepared before the outdoor test were determined. The results showed that the protective effect of the acrylic coating system containing fir bark extracts in QUV test conditions over 2016 h was similar to that of the coating containing commercial UV absorbers. However, while the fir bark extract's antioxidant effect positively affected the beech wood to increase durability in outdoor conditions, it had a lower preservative effect on the pine wood.

Keywords: artificial weathering test, acrylic coating, black pine bark extract, colour change, fir bark extract, surface roughness

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Introduction

As one of the methods used to protect the surface of wood material against outdoor conditions, UV light and water contact with the wood surface are prevented by surface treatments. Today, different wood surface protective products used in outdoor conditions are offered on the market [Decker et al. 2004; Evans et al. 2005; Penga et al. 2020]. In order to protect the wood surface from photodegradation, acrylic resin coatings and UV absorbers used in the car industry are being investigated extensively. According to these studies, high protection is provided on the surface against photodegradation by an acrylic resin containing organic or inorganic UV absorbers [George et al. 2005; Custódio and Eusébio 2006; Aloui et al. 2007; Deka and Petrič 2008]. While colour stabilisation is provided on the surface, lignification is also prevented by protecting the wood surface from washing. An acrylic resin containing UV absorber provides more extended protection against outdoor conditions in comparison to older generation surface treatments [Hayoz et al. 2003; Forsthuber and Grüll 2010; Özgenç and Yıldız 2016a]. Several studies have been carried out on the effects of some bark extracts, high phenol content, or antioxidant effects. Chang et al. [2010] investigated the antioxidant activity of extracts obtained from the bark and heartwood of Taiwan's acacia tree. It was determined that the acacia heartwood extract is a more effective antioxidant than bark extract. Douf et al. [2006] tested extraction of hazelnut, sumac, *Shinopsis lorentzii* L. leaf, and pine and mimosa barks by three different methods. It was determined that the antioxidant capacity of the extracts was very high. Jerez et al. [2007] compared the antioxidant activity and procyanidin components of extracts obtained from maritime pine and red pine barks. Vázquez et al. [2008] investigated the potential antioxidant components of chestnut and eucalyptus bark extracts. The chestnut bark extract was shown to have a high molecular weight, and eucalyptus bark a lower molecular weight. The phenol content and antioxidant activity of the bark extracts were found to have a positive linear correlation. The results of FTIR spectroscopy analysis revealed that the phenol content of the chestnut bark extract was higher than that of the eucalyptus bark extract. Salem et al. [2014] achieved positive results for antibacterial, antifungal, and antioxidant properties of extracts from *Delonix regia* L. bark, commonly used as firewood. There are some studies in the literature on the use of tree bark extracts as organic UV absorbers due to their high antioxidant effect. One of these studies compared the photostabilisation effect of bark extract, lignin stabiliser, and UV absorber and inhibitor (amine light stabiliser) on acrylic polyurethane coating on a wood surface. The highest photostabilisation effect after the accelerated weathering test was found in heat-treated wood surfaces with applied acrylic polyurethane containing bark extract and lignin stabiliser [Kocaefe and Saha 2012]. According to the results of another study, natural antioxidant (bark extract) and lignin stabiliser alone or combined were mixed into an acrylic polyurethane material to increase the durability of the coating material in outdoor conditions. The colour stabilisation of the wood surfaces coated with acrylic polyurethane containing bark extract was improved [Saha et al. 2011]. The colour stability of wood coatings based on a combination of epoxy functionalized soybean oil and UV-absorber 2-hydroxy-4(2,3--epoxypropoxy)-benzophenone (HEPBP) was very high compared with wood coatings containing HEPBP only. Additionally, the epoxy-based soybean oil combination with UV absorber (HEPBP) provided substantial protection against physical deformation of wood coatings caused by external factors such as UV radiation and rain [Olsson et al. 2012]. Saha et al. [2013] examined the outdoor strength of acrylic polyurethane coatings prepared with three different UV stabilisers (UV absorber, HALS, antioxidant). It was determined that the strength of wooden surfaces with applied acrylic polyurethane coating modified with coniferous tree bark and leaf extracts increased in outdoor conditions. It was concluded that the antioxidant effect of coniferous tree bark and leaf extracts provides high UV absorption. In a study by Grigsby and Steward [2018], condensed grain extracted from tree barks was added to an acrylic resin-based coating as a functional additive. Acrylic coatings modified with tannin with a high antioxidant effect provided very high protection on the surface of wood against outdoor conditions.

In studies in the literature, existing coatings have been modified using bark extracts. This study examined the durability of waterborne acrylic coating systems containing tree bark extracts in outdoor conditions. Bark extracts obtained from two different tree species (fir and black pine) and commercial UV absorber (Tinuvin DW 400) were used as the test and control groups respectively. The surface colour and roughness changes of the test and control samples exposed to artificial weathering testing were analysed. Additionally, the coating thicknesses of the test and control samples were determined by light microscopy. UV absorber materials used in wood surface coating systems are imported and expensive products. For this reason, the study will investigate the availability of tree bark extracts as alternatives to UV absorbers.

Materials and methods

Wood and bark samples

The barks were peeled from 20- to 30-year-old fir (*Abies nordmanniana* L.) and black pine (*Pinus nigra* L.) trees felled in the Black Sea Region in the north of Turkey. The TAPPI T 257 cm-12 and TAPPI T 264 cm-07 standard methods were used for preparation and chopping of tree barks for extraction analysis. Scots pine (*Pinus sylvestris* L.) and Oriental beech (*Fagus orientalis* L.) were used in the study as defect-free samples. The wood samples were prepared with dimensions of 150 mm (longitudinal) by 70 mm (tangential) by 20 mm (radial) for artificial weathering.

Bark extraction process

All tree barks were air-dried at room temperature and then ground using a laboratory-scale Wiley mill to obtain 40- to 60-mesh bark powder. To obtain extractives, the bark powders were extracted in a Soxhlet extractor. The bark powder (25 g for each) was soaked in 300 mL of ethyl alcohol:benzene (1:2 v/v). The solvents from each extract were removed using a rotary evaporator at 50°C and stored in sealed flasks at 4°C until use.

The extracts' antioxidant activities were determined by the method of 2,2--diphenyl-1-picryl hydroxyl (DPPH) radical capture activity. Determination of total phenolic matter also sheds light on the extract's antioxidant capacity, since it is based on oxidation–reduction reactions. There is a linear relationship between the bark extract's total phenolic content (BE) and its antioxidant activity (Table 1).

Table 1. Total phenol content and antioxidant activity of tree bark extracts (BE)

BE species	Total phenol content (TPC) (mg-GAE/g-DB)	Antioxidant activity (AA) (%)
Fir	49.09	75.47
Black pine	191.47	90.31

Preparation and application of coating systems

The waterborne acrylic-based coating system was formulated from the commercial acrylic resin, a poly-(methyl acrylate/methyl methacrylate/butyl acrylate) copolymer dispersion. In order to increase the effects of other additives on photostabilisation, only small amounts of defoamer and 2,2,4-trimethyl-1,3--pentandiolemonoisobutyrate as coalescent agents were used in the formulation. Acrylic-based coating systems containing bark extracts were compared with the control coating containing commercial UV absorber. Tinuvin DW 400, known as a commercial UV absorber containing a hydroxyphenyl-striazine class (HPT) is a BASF product.

For the artificial weathering test, the back, end-grain, and side faces of wood samples were covered with 2-epoxy white paint with a brush, and the front side was left uncoated for the experiments. The commercial waterborne impregnation product, with the active ingredients 1.20% propiconazole and 0.30% iodopropynyl butylcarbamate, was used as a primer to protect the samples against biological deterioration, including soft rot and blue stain. The primer was applied to the samples in an amount of 120 g/m^2 using a brush. The first layers of topcoats were also applied to each sample at a spread rate of 100 g/m^2 with a brush. Later, the specimens were sanded with 240-grit size sandpaper and kept at room temperature for two days before the second layer of topcoat was applied.

After the top coating, the samples were conditioned at 20°C and 65% RH for two weeks.

Artificial weathering test

All wood samples with applied coating systems comprising bark extracts and mineral UV absorber were subjected to an artificial weathering test by exposure to fluorescent UV lamps (340 nm) in a QUV/spray accelerated weathering tester (Q-Panel Lab Products, Cleveland, USA) for 2016 h according to the EN 927-6 standard. The weathering experiment was conducted using cycles of UV-light irradiation for 2.5 h at 60°C followed by a water spray for 0.5 h, followed by condensation for 24 h at 45°C in an accelerated weathering test cycle chamber. Four samples were used for each variation in the artificial weathering test. The samples were removed from the test chamber at 24 h intervals over 48 h, and subsequently, colour measurements were performed on the exposed surfaces coated with acrylic and alkyd systems. After the measurements, the samples were placed again into the chamber, and then, at every 224 h interval over 2016 h, the samples were removed, and the measurements were repeated on the surfaces. During the 2016 h test period, 11 periodic measurements were performed on the exposed surfaces.

Colour measurement

The colour measurements were carried out using a Minolta CM-600d spectrophotometer (Konica Minolta, Japan) equipped with an integrating sphere according to the CIE L* a* b* system (ISO/DIS 7724-2 standard). The Konica Minolta CM-600d instrument's reflection spectrum was acquired from an area measuring 8 mm in diameter with 10^0 in the 400-700 nm wavelength range. Five measurements were recorded for each sample.

Surface roughness measurement

A TR100 Surface Roughness Tester was employed for the measurement of surface roughness. The *Ra* and *Rz* roughness parameters were measured to evaluate the surface roughness of unweathered and weathered (treated and untreated) samples according to the ISO E 4287 standard. *Ra* is the arithmetic mean of absolute values for profile departures within the reference length, and *Rz* is the arithmetic mean of the 5-point height of irregularities. The cut-off length was 2.5 mm, the sampling length was 12.5 mm, and the detecting tip radius was 5 mm for the surface roughness measurements.

Ra = the arithmetic mean of the absolute values for profile departures within the reference length;

Rz = the arithmetic mean of the 5-point height of irregularities.

The variable called the Roughness Index (*RI*) is defined according to equation (1) [Nzokou, 2004]:

$$RI = \frac{Rz_{(w)} - Rz_{(i)}}{Rz_{(i)}} \tag{1}$$

Determination of coating thickness using the light microscope

The coating thickness was determined using a ZEISS Stemi 305 light microscope and ZEISS AxiocAM erC 5s camera at 2X magnification according to ASTM D5235-18 [2018]. Dry film thickness was calculated as the average of the measurements taken from two samples: five measurements from each sample surface. The viscosities of the three different coatings applied in this study were determined using DIN cup/4mm/20 0 C ASTM D5235-18 [2018]. The coating viscosity value was calculated as the average of five measurements taken from each varnish type.

Results and discussion

Colour change of wood surfaces finished with coating systems

The colour stabilisation of acrylic resin containing tree bark extractives and UV absorbent was evaluated in artificial weathering conditions for 2016 h. The changes in the colour as a result of intensive weathering conditions are clearly seen in Figure 1. The greatest colour change (*E**) was found with the coating systems containing black pine extract, and the smallest with the fir extract on pine. In this study, it was determined that the effect of colour stabilisation of the fir extract may rival that of commercial products such as that used in the control samples.

A positive value of Δb indicates increasing yellowish colour on the wood surface, while a negative value refers to a bluish colour. As seen in Figure 1, the test samples' colour changed from yellowish to bluish, compared with the beginning of the artificial weathering test. The greatest change was found in black pine bark extractives, while the lowest Δb value was obtained from the control samples. Moreover, the Δa value refers to a reddish colour for positive values, and greenish for negative values [Ozgenc and Yildiz 2016b]. The test coating samples' general trend, except for the fir bark extract and control coatings, was a change from reddish to greenish over the 2016 h. After that time, the test samples' surfaces tended to be greenish, except for samples with the fir bark extract coating. However, the control coating samples had a positive value, that is, reddish. The lowest Δa values were obtained from the fir bark extractives for beech, compared with the control samples as a commercial product. The ΔL values give clues about the surface quality and lightness [Penga et al. 2020]. Polymerisation of lignin during a weathering test causes the wood surface to be

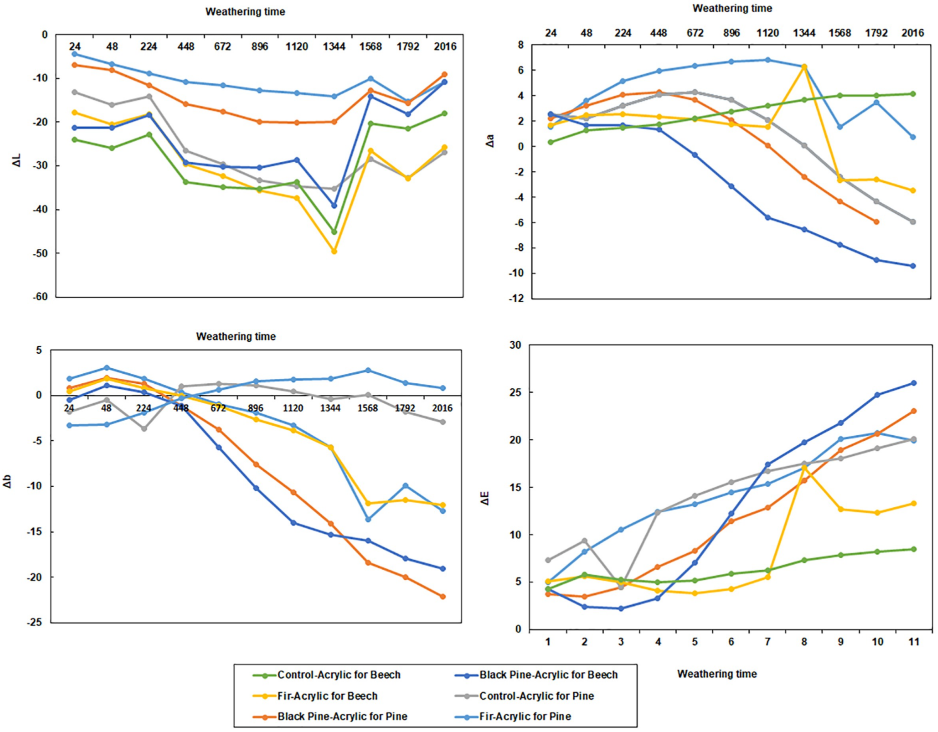

Fig. 1. Changes in colour coordinates of wood surfaces with applied acrylic coating systems

dark [Kocaefe and Saha 2012]. The Δ*L* values of coatings for beech tended towards negative during the 2016 hours. Therefore, the beech wood surface became rougher and darker during the artificial weathering test. It was found that, in both types of wood, the lowest Δ*L* value was observed in the coating systems containing black pine extract.

In the literature, tree bark extractives have provided the best colour stabilisation based on Δ*E* values representing the colour change on the wood surface under artificial weathering conditions. The high antioxidant activity of tree bark extractives inhibits the oxidation reaction which occurs in cell wall components, especially in lignin [Evans et al. 1987; Saha et al. 2011, 2013]. Therefore, tree bark extractives improve weathering resistance significantly. However, in this study, the Δ*E* values of the control and test samples with the acrylic coating systems were different. The least colour change was obtained from the commercial UV absorber (control) for both pine and beech. While the colour changes increased gradually during the 2016 h for the bark extracts, the colour change in the coating systems containing bark extracts was generally higher than in the control coatings containing the commercial UV absorber. The oxidation reactions induced by weathering conditions (the combination of UV radiation, high humidity, temperature, etc.) cause deformation in the resin structure influencing coating adhesion and cohesion [Singh et al. 2001; Perera 2003]. Therefore, UV rays reach the wood surface, and degradation occurs. Nevertheless, the colour change of the coating system containing fir extract was close to that of the control coating applied on a pine wood surface.

Surface roughness change for coating systems

The surface roughness values (*Ra* and *Rz*) of waterborne acrylic-based systems containing bark extract or commercial UV absorber are given in Figure 2. It is seen that there was no significant change in the *Ra* and *Rz* values of the control samples after weathering. On the other hand, considerable changes arose after weathering in the coating systems containing bark extracts. Additionally, except for the test coating systems applied to pine wood, the *Ra* values of other test coating systems showed an increase in the weathering test. There was a significant change in the *Rz* values of all coating systems. While the *Rz* values of the coating systems containing black pine extract applied to beech wood increased, a decrease was recorded in the samples' *Rz* values for the other test and control coating systems.

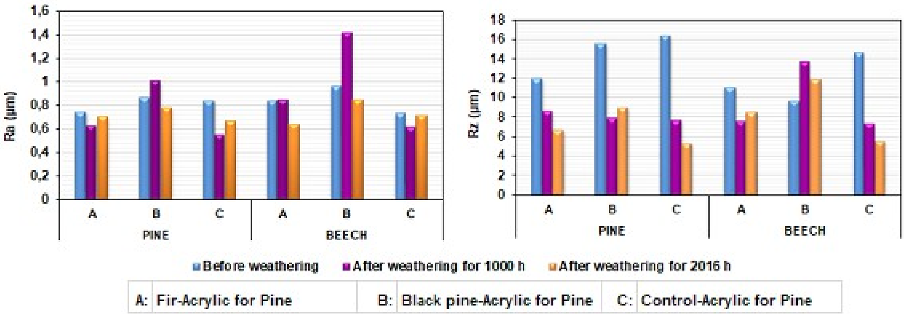

Fig. 2. Changes in surface roughness values of wood surfaces with applied acrylic coating systems

Since the surface roughness change parameters (*RI*) of acrylic coatings applied on the Scots pine and beech woods after the weathering test did not exhibit normality, a comparison was made with the Mann-Whitney U-test. As seen in Tables 2 and 3, no significant difference was found between the control coating on both wood species' surfaces, since $p > 0.05$. In contrast, a significant difference was found in other test coatings containing bark extract, as $p < 0.05$.

The surface roughness of wood materials is not induced only by the anatomical wood structure, as it is an anisotropic and heterogeneous material. Therefore, some factors must be taken into consideration to evaluate surface roughness, for example, earlywood, and latewood content in the annual ring,

Table 2. Comparison of surface roughness change (RI) parameters of acrylic coatings on Scots pine samples

Scots pine	N	Average rank	*Sum rank*	U	p
Coating with fir extract	20	10.50	210	0.00	0.00
Coating with black pine extract	20	11.50	230	20.00	0.00
Coating with UV absorber (control)	20	19.45	389	179.00	0.570

Table 3. Comparison of surface roughness change (RI) parameters of acrylic coatings on beech samples

Beech	N	Average rank	*Sum rank*	U	p
Coating with fir extract	20	10.50	210	0.00	0.00
Coating with black pine extract	20	12.35	247	37.00	0.00
Coating with UV absorber (control)	20	21.25	425	185.00	0.685

natural growth characteristics (knot, fibre curl, etc.), annual ring width, drying temperature, shear direction and angle [Kilic et al. 2006; Aydın and Çolakoğlu 2005]. Extensive weathering conditions influence the wood surface, and some chemical changes in both the coating and wood structures result in diminishing coating adhesion and cohesion [Perera 2003]. The film thickness decreases depending on the intensity of weathering conditions due to surface erosion during the exposure period [Decker et al. 2004]. Therefore, the wood surface deteriorates, which leads to the creation of hollows and bumps on its surface. Chemical changes occur in the polymer structure because of different changes on the surface [Perera 1995], resulting in variation for resin types.

Coating thickness

This study investigated the effects of coating thickness and viscosity of coating systems on durability in outdoor conditions. The coating thickness measurements were made at ten locations along the coating using a light microscope equipped with a scale calibrated with a micrometer. A summary of coating thickness and coating viscosity is presented in Table 4. According to the results of the study, there was a significant relationship between coating thickness and viscosity.

As seen in Figure 3, microscope observations revealed that the penetration behaviour was different in pine and beech wood. It was seen that the colour change of pine wood was higher than that of beech wood because the coating penetration of beech wood was lower than in pine wood. Thus, the high coating thickness on the beech wood increased the acrylic coating systems' colour stability in outdoor conditions.

Table 4. Coating thicknesses and viscosity values of acrylic coating systems

Coating systems	Coating thickness for pine (μm)	Coating thickness for beech (μm)	Viscosity (s)
Fir–Acrylic	45.36 ±5.18	71.24 ±9.89	100 ±10
Black pine–Acrylic	53.73 ±8.72	87.96 ±11.85	120 ±15
Control–Acrylic	37.23 ±4.32	65.37 ±9.15	70 ±8

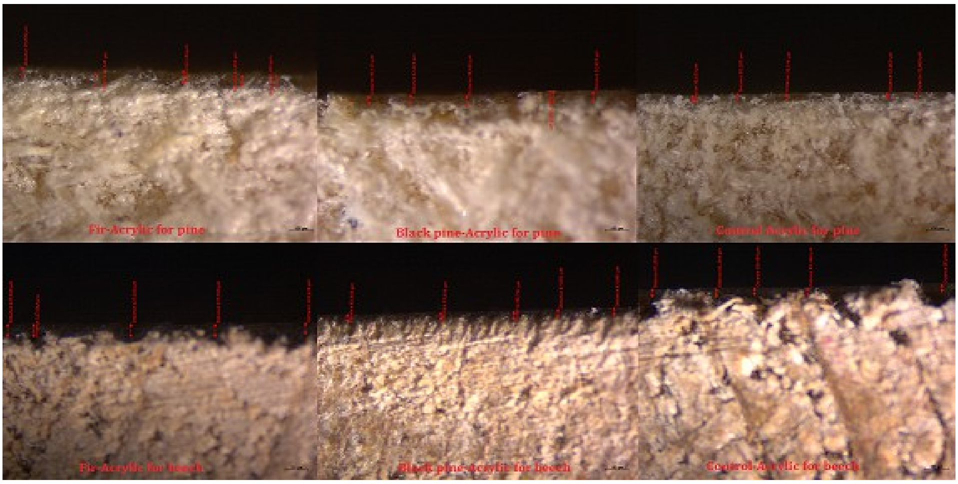

Fig. 3. Microscopic screening of test and control coating systems applied to beech and Scots pine surfaces

In this study, there was a significant relationship between coating thickness and coating viscosity. Low viscosity of the coating led to low coating thickness on the surface of the wood. Compared with high-viscosity substances, low-viscosity coatings have a higher penetration in the wood [Toker et al. 2012]. In all instances, the same amount of coating systems was applied to the wood. However, depending on the coating viscosity and the variability of wood types, the coating penetration also varied. Because of coating thickness, different colour changes in wood samples after weathering are seen. The low penetration of acrylic coating systems on the wood surface increases the coating thickness. When the thickness of coating systems decreases with increasing penetration, the colour change on the wood surface decreases after weathering [Bulcke et al. 2008; Dawson et al. 2008]. As seen in Figure 3, microscope observations revealed that the penetration behaviour was different in pine and beech wood. It is seen that the colour change of pine wood was higher than that of beech wood because the coating penetration of beech wood was lower than that of pine wood. Thus, the high coating thickness on the beech wood increased the acrylic coating systems' colour stability in outdoor conditions.

Conclusions

The bark constitutes a significant part of the weight of trees (10-15%) which offers vast quantities of biomass to industry. However, it is mainly used as fuel or left to rot in the forest. Indeed, bark has potential due to its high antioxidant capacity. In this study, tree bark extracts were evaluated as UV absorbers against artificial weathering conditions. For this purpose, two different tree barks (fir and black pine) were obtained by alcohol:benzene extraction and compared with commercial UV absorber on the surface of two different wood species (pine and beech) under weathering conditions. The viscosity and penetration are essential factors to determine the weathering performance of coating systems. Light microscopy images showed that the coating thickness is higher on the beech wood surface than on pine wood. Therefore, when the viscosity of coatings increases, the penetration decreases, which results in a high coating thickness. As the coating thickness on the wood surface increases, the colour stabilisation of the coating system increases, and the variation of roughness decreases in outdoor conditions. However, according to the results obtained, colour changes on the wood surface due to photodegradation are inevitable. Meanwhile, the bark extracts show significant resistance against extensive weathering conditions. The fir bark extract restricted colour changes as much as a commercial UV absorber. Similarly, the changes in the surface roughness are close to those obtained using the commercial UV absorber after the weathering test. Consequently, fir bark extracts with high antioxidant capacity and high phenol content provide photostabilisation and protect the wood surface against outdoor conditions. Such tree bark extracts have the potential to compete with commercial UV absorbents. In future studies it is recommended to study the development of acrylic coating systems with various tree bark extracts.

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