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THE EFFECT OF WEATHERING ON SURFACE CHARACTERISTICS OF CHEMICALLY MODIFIED SCOTS PINE (*PINUS SYLVESTRIS*) WOOD

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ABSTRACT

Scots pine (*Pinus sylvestris* L.) sapwood of 200 × 20 × 80 mm^3 (L×R×T) was treated with both cell wall filling and lumen filling chemical agents (low-molecular phenol-formaldehyde, bio-oil, N-methylol/N-methyl compounds, sorbitol-citric acid, polysiloxane), which were fixed inside the wooden structure during heat-curing processes. The present study investigated the impact of the appointed chemical modifications on the surface characteristics of wood, which was addressed by measurements of the surface roughness (Ra), surface free energy (SFE), contact angles, wettability and its bonding quality. Independent of the chemical agents applied, Ra decreased as result of the chemical treatments, while SFE experienced a reduction. The Ra and SFE of both untreated and modified pine specimens increased after weathering processes. The weathering was appointed to cause a decrease in the equilibrium contact angle (θe) and an increase in the constant contact angle change rate (K-value). Increasing K-values after weathering for both untreated and modified pine specimens indicated their better wettability. Increasing wettability after weathering led to better adherence of acrylic paints on the surface of the Scots pine wood. In summary, the chemical modifications decreased the Ra and SFE of the pine sapwood, which may as a consequence affect the wettability and bonding quality of wood during outdoor exposure.

KEYWORDS:Chemical modification, Scots pine wood, surface roughness, surface free energy, wettability.

INTRODUCTION

Chemical modification occurs through the formation of covalent bonds between chemical reagents and wood substrates (Hill 2006).Chemical modification depends on adequate distribution of reacting chemicals on water-accessible regions of the cell wall. Ithas the potential to improve biological resistance and dimensional stability of wood by acetylation (Rowell 2006; Özmen 2007), DMDHEU (Dieste et al. 2008; Verma et al. 2009), resin treatments (PF, MF, UF, and MMF) (Hill 2006), furfurylation (Sejati et al. 2016; Martha et al. 2021a) and glycerol-maleic anhydride (GMA) treatment (Mubarok et al. 2020; Martha et al. 2021b). In addition to chemical modification, the surface coating also can increase the photostability and outdoor service life. Wood photodegradation is usually caused by environmental factors such as sunlight, moisture, and temperature. The most destructive factors are UV irradiation from the sun and the leaching effect of rain. Wood discoloration is caused by lignin degradation, which is the main structural component of wood (Evans et al. 2002; Cogulet et al. 2016; Sahin et al. 2020). UV irradiation leads to degradation of lignin that can absorb 80–95% of the total UV irradiation absorbed by wood (Nzokou and Kamdem 2006).Degradation of lignin causes a change in chemical properties and physical properties of wood (Grelieret al. 2000). Changes in chemical properties and physical properties lead to discoloration, roughening, and decrease in durability (Martha et al. 2023; Rahayu et al. 2023).

The wettability of water and water-based coatings in unmodified and thermally modified beech wood (*Fagus sylvatica* L.) has been reported by Zigon et al. (2023). The decrease in wettability and absorption of water and water-based coating on thermally modified beech wood is attributed to the chemical composition change of the wood. Altgen et al. (2020) reported that a plasma treatment on the fresh wood surface improves wettability and compares favorably with the wettability of unmodified and thermally modified wood surfaces. The contact angle increases faster on plasma-treated wood surfaces than on unmodified wood surfaces, the decrease in contact angle due to plasma treatment is still significant even after 4 weeks of aging. Hanifah et al. (2022) investigated the influence of furfurylation and GMA treatment on coating performance of short rotation teak wood. The results showed that modified wood samples have better results than unmodified samples regarding photostability. Petrič et al. (2007) have studied the wettability of oil heat-treated and DMDHEU-modified Scots pine wood with several commercial water-based coatings. It was found that the high contact angle is caused by the increased hydrophobic properties of the modified wood. It was also found that water-based coating present much better wettability on modified surfaces.

Though the chemical modification of woods has the potential to improve some of their less favorable characteristics, however it is known that the modifications can change hydrophilic character of the woods, which lead to change their wettability behavior with coating or painting processes. Improvement in the lessfavorableproperties (stability, durability, and color) of Scots pine wood has been being made by modification using enborer, softener, citric acid-sorbitol, dimethylol-dihydroxyethylen urea (DMDHEU),DMDHEU-Softener, phenol-formaldehyde

(PF),PF-bio-oil. Based on this fact, the behavior of wettability after chemical modification followed by weathering should be essentially important for well knowing paints adhesion phenomena. Considering the environmental and health concerns, water-based coatings should be used to protect wood from photodegradation. In other hand, water-based coatings are cost-effective and maintain a good permeability (Peng et al. 2021). The investigation of the surface characteristics of chemical modified woods and their effects on the bonding quality of water-based coatings and on the weathering,resistance is not well known. The objective of this study was to determine surface characteristics such as surface roughness, surface free energy (SFE), wettability, and bonding quality of chemically modified Scots pine wood after weathering.

MATERIAL AND METHODS

Sample preparation and chemical modifications

Chemical modifications were performed with enborer, softener, citric acid-sorbitol (CAS), DMDHEU (D),DMDHEU-softener (DS),phenol-formaldehyde (PF),phenol-formaldehyde-bio-oil (PFO). Chemically modified Scots pine woods were air dried to moisture content of 12–15%. Surface characterizations were performed on blocks of 200 x 20 x 8 mm^3, longitudinal (L), radial (R), tangential (T). The modified samples were weathered at a weathering station of Forest Products Technology Department, IPB University for 12 months and evaluated in 4-month intervals. At the end of each interval, the samples were tested to investigate their surface characteristics (surface roughness, surface free energy (SFE), and wettability).

Surface roughness test

The surface roughness measurement was evaluated according to ISO 4287 (1997). Twelvesamples for each chemical treatment were prepared for the un-weathered (3 samples) tested at 0 month and the weathered (9 samples) tested at 4, 8 and 12 months. The surface roughness measurements were conducted on the tangential surface using Mitutoyo type SJ-210 tester before weathering (0 month) and after 4, 8, and 12 weathering. Three points were made perpendicular to the fibre direction at different positions. The arithmetical mean roughness (Ra value) was performed with a diamond tip radius of 5 μm, tracing length of 6 mm, a cut-off of 2.5 mm and speed of 0.5 mm/s.

Contact angle measurement

This measurement was performed at the interval of 4 month. A video measurement system with a high-resolution CCD camera was used to record the dynamic contact angle. The SFE was measured by the dynamic contact angle of standard liquids (water, methanol, toluene, and glycerine), meanwhile the wettability was measured by the dynamic contact angle of acrylic paint. The syringe method with each drop of liquid as a volume of 0.02 ml was used. A CCD camera recorded the drop shape on the wood surface for 180 s. Dynamic contact angle measurements were carried out for five droplets on each surface. The recorded videos were cut into individual images at 10s intervals for a total duration of 180 s. The Image-J 1.46 software with drop-snakes plugin analysis was used to measure the contact angle of the individual image of the drop. The contact angle of each droplet on the surface of the wood specimen was measured by averaging the contact angles on the left and right sides.

Determination of equilibrium contact angle and constant contact angle change rate

The equilibrium contact angle value was determined by the transition point between contact angle and time using a segmented regression model. The determination of the equilibrium contact angle was calculated using the PROC NLIN program in SAS. The contact angle change rate (K-value) in the S/G model (Shi and Gardner 2001) was used to quantitatively determine wettability with Eq. 1:

$$\theta = \frac{\theta_i \times \theta_e}{\theta_i + (\theta_e - \theta_i) exp\left[K\left(\frac{\theta_e}{\theta_e - \theta_i}\right)t\right]} \quad (1)$$

where: θ is the contact angle at a certain time, θ_i is the initial contact angle, θ_e is equilibrium contact angle, K is the constant contact angle change rate and t is wetting time. A non-linear regression model was used to calculate K-value using the defined function to fit S/G equation by XLSTAT Addinsoft.

Determination of SFE

SFE value and its components were determined using a multi-liquid method as proposed by Rabel (1971). The method uses a regression line from the OWRK method (Owen and Wendt 1969) as Eq. 2. The surface tension values of standard liquids were presented in (Tab. 1).

$$(1 + \cos\theta_e)\frac{\gamma_l}{(\gamma_l^d)^{1/2}} = (\gamma_s^d)^{1/2} + (\gamma_s^p)^{1/2}\left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{1/2} \quad (2)$$

where: θ_e is equilibrium contact angle of the standard liquid, γ_l is the total surface tension of liquid, γ_l^d is dispersive surface tension of liquid, γ_s^d is dispersive component of solid wood, γ_s^p is polar component of solid wood, and γ_l^p is polar surface tension of liquid.

Tab.1: Polar, dispersive and total component of surface tensionfor the standard liquids (Yuningsih et al. 2019).

Liquids	γ_l^p (mJ*m^{-2})	γ_l^d(mJ*m^{-2})	γ_l(mJ*m^{-2})
Water	21.8	51.0	72.8
Methanol 50%	12.9	22.7	35.6
Toluene	2.3	26.1	28.4
Glycerin	30.0	34.0	64.0

Bonding quality test

Two coating layers of acrylic paint were applied using a brush on the modified wood surface to achieve a total application of 150 g/m^2 wet film. Before applying the first coat, the wood surface was sanded using sandpaper number 180. After 24 h, the wood surface was sanded using sandpaper number 240, and then the second coat was applied. When the second coat had dried, a crosscut test method was applied to evaluate the resistance of the acrylic paint to separation from wood surfaces. The coating film on each test specimen was then scratched by a cutter of 11 lines with a distance between lines of 2 mm. The tape was then applied over the scratch, and the end of the tape was pulled rapidly at 45° to the surface of the test specimen. Bonding quality was classified according to the ASTM D 3359-02 (2007).

RESULTS AND DISCUSSION

Surface roughness

Fig. 1 shows the effect of enborer, softener, citric acid-sorbitol (CAS), DMDHEU (D),DMDHEU-softener (DS),phenol-formaldehyde (PF),phenol-formaldehyde-bio-oil (PFO) on surface roughness (Ra) of Scots pine wood. The Ra values of untreated, enborer, softener, CAS, D, DS,PF, and PFO for 0-month weathering were 5.14 µm, 7.99 µm, 5.82 µm, 7.53 µm, 6.88 µm, 7.52 µm, 7.36 µm, and 7.21 µm. The Ra value of the untreated Scots pine was the smallest for 0-month weathering, however its Ra value was the highest after 8 and 12 months weathering. The Ra value of the Scots pine increased slightly after the chemical treatments, and the Ra values among the chemical treatments varied slightly.The acidity of the chemical solutions used in this research may have caused the formation of microscopic cracks in the cell wall causing the increase in the roughness. Reinprecht (2016) reported that contact between wood and an acidic solution can increase surface roughness. Dong et al. (2015) reported that chemical treatment with furfuryl improves hydrophobic properties and leads to wavy structures on the vessel walls. Suggested explanations for the more distinct heterogeneity (rougher surfaces) of the thermally modified wood sample are related to chemical changes of the wood substance, which seem to result in certain micromorphological features, and also possible redistribution of the wood extractives (Källbom et al. 2015).

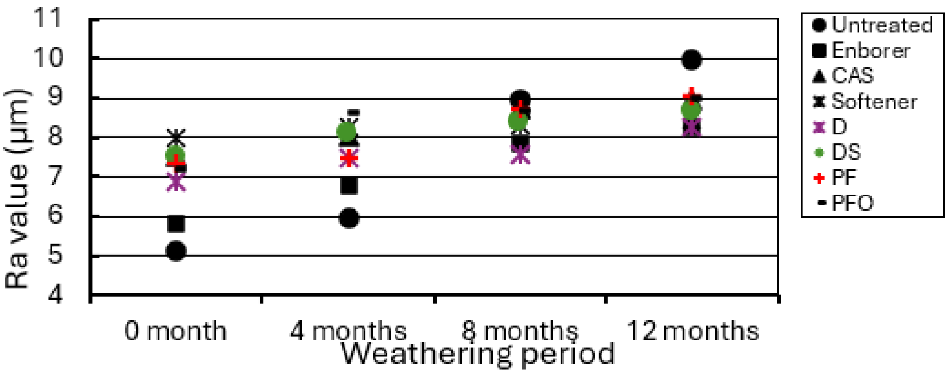

Fig. 1: Surface roughness (Ra value) of untreated and modified Scots pine woods before and after weathering.

The results in Fig. 1 also show that the Ra values increased after weathering treatments. The increase in the Ra values indicates that the surface became rougher due to both the chemical and weathering treatments. A longer period of weathering tended to cause the increase in the roughness. The increase in the surface roughness should be associated with dimensional changes due to swelling during the weathering treatment. The swelling could raise some individual fibres on the surface of the untreated sample. It was reported in another study that cracks have been more likely to form between the cells which cause the increase in the roughness after weathering (Keržič et al. 2021). Comparing the surface degradation due to the weathering, the chemically treated Scots pine provided better surface stability compared to the untreated Scots pine. This might be attributed to the combination of chemicals used and low density of the Scots pine (lower compaction ratio) could produce higher surface stability.

Surface free energy (SFE)

The total SFE for the untreated, enborer, softener, citric acid-sorbitol (CAS), DMDHEU (D),DMDHEU-softener (DS),phenol-formaldehyde (PF),phenol-formaldehyde-bio-oil (PFO) are shown in Fig. 2. Their total SFE values for 0-, 4-, 8-, and 12-months were in the range of 35.67-46.78 mJ·m^{-2}; 44.40-62.17 mJ·m^{-2}; 46.74-65.82 mJ·m^{-2}; and 49.85-67.23 mJ·m^{-2}, resp. Before weathering the total SFE values of chemically treated were lower than untreated wood. Chemical treatment causes a decrease in free hydroxyl groups leading to an increase in hydrophobicity (Martha et al. 2021ab; Basri et al. 2022). The increased hydrophobicity of chemically treated wood might contribute to a decrease in the SFE value. Other studies have also reported that chemical changes due to wood modification treatments lead to a decrease in the total SFE value (Hill 2006; Gérardin 2007; Košelová 2021; Hanifah et al. 2022).

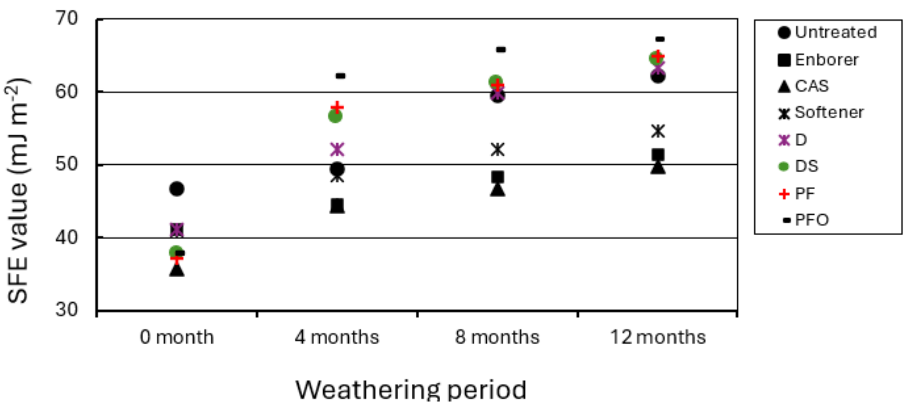

Fig. 2: Total surface free energy (SFE) value of untreated and modified Scots pine woods before and after weathering.

The total SFE of the chemically treated woods increased markedly after weathering treatment (Fig. 2), and their total SFE values were similar as the total SFE of the untreated. This might be due to the chemical component changes in the wood after weathering. During the weathering process, leaching of the chemical agents applied for modification process could reduce water repellence. Rowell (2006) also reported that degradation of lignin during weathering process results in hydrophilic surface. The increase in surface roughness due to weathering treatment could have an effect on increasing the SFE value on the surface of chemically modified Scots pine wood. Other studies reported that the rougher surfaces generate higher SFE values (Yuningsih et al. 2019; Martha et al. 2020). The weathering period affected the ratio of SFE components. Before weathering the polar SFE values were lower than the dispersive SFE both for untreated and chemically treated (Fig. 3). It was also reported in another study that polar SFE (γ_s^p) is lower than dispersive SFE(γ_s^d) for treated Scots pinewood

(Košelová et al. 2021). The dispersive SFE decreased and the polar SFE increased with increasing weathering time (Fig. 3). An increase of moisture content from an air dry to a wet condition for both the unmodified and thermally modified samples during outdoor weathering seemed to have an influence on the decrease of the dispersive component of the surface energy. Another study reported that the influence of exposure to higher RH conditions results in small changes in the dispersive component of the surface energy (Källbom et al. 2015).

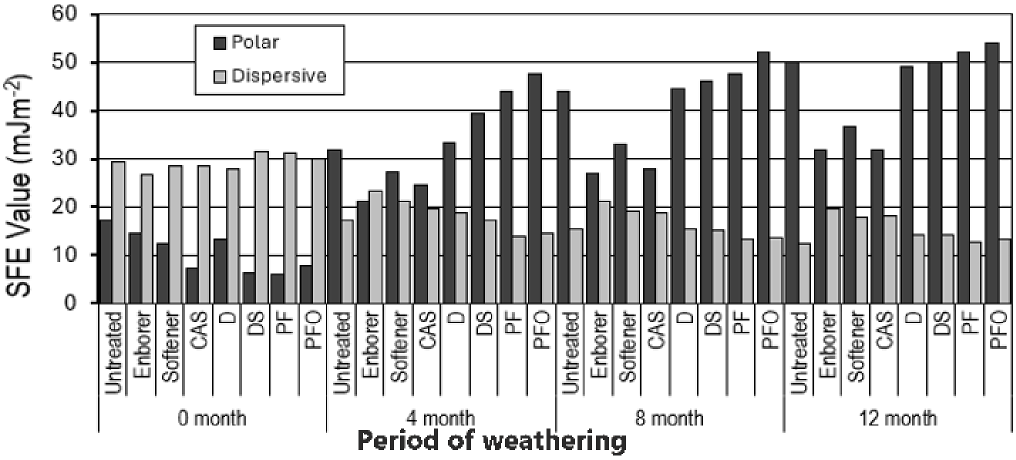

Fig. 3: The value of polar SFE and dispersive SFE of untreated and modified Scots pine woods before and after weathering.

Contact angle and wettability

Fig. 4 presents the value of equilibrium contact angle (θe) before and after weathering for untreated, enborer, softener, citric acid-sorbitol (CAS), DMDHEU (D),DMDHEU-softener (DS),phenol-formaldehyde (PF),phenol-formaldehyde-bio-oil (PFO). The result shows that the equilibrium contact angle on acrylic paint for each treatment decreased in line with the weathering period. The similar result was reported by Basri et al. 2022. The equilibrium contact angle of untreated and modified teak wood decrease after three months of weathering for acrylic varnishes. It was affected by the roughness of the wood surface. Martha et al. (2020) reported that the rougher surfaces tend to provide lower contact angles equilibrium. On the other hand, the increase in the SFE value tended to cause a decrease in the equilibrium contact angel value. It was reported in other studies, a higher SFE generates a lower contact angle, vice versa (Yuningsih et al. 2019; Martha et al. 2020). Wood surface with a lower contact angle equilibrium caused the acrylic paint spread and penetrated easier in its surface. The values of equilibrium contact angle are used to calculate the constant contact angle change rate (K-value) that describes wettability. The greater the K-value generates the faster a liquid spread and penetrates into a wood surface (Shi and Gardner 2001). The mean K-value of acrylic paint of untreated and modified Scots pinewoods after and before weathering are shown in Fig. 5. Chemical modification treatment resulted in decreased wettability of Scots pine wood. Hanifah et al. (2022) reported that an increase in hydrophobicity after chemical modification treatment causes a decrease in surface free energy resulting in a lower K-value. The K-values of acrylic paint for 0-, 4-, 8-, and 12-monthes were in the range 0.025-0.082; 0.047-0.114; 0.057-0.132; and 0.065-0.165, resp. increased as the weathering period increased. This result indicates that weathering treatment increased the wettability of Scots pine wood. The increase in wettability was attributed to the increase in surface roughness and SFE values as the weathering period increased. The determination of the interaction between the wood surface and the paint depends on wettability, which is important in determining the bonding quality (Rathke and Sinn 2013).

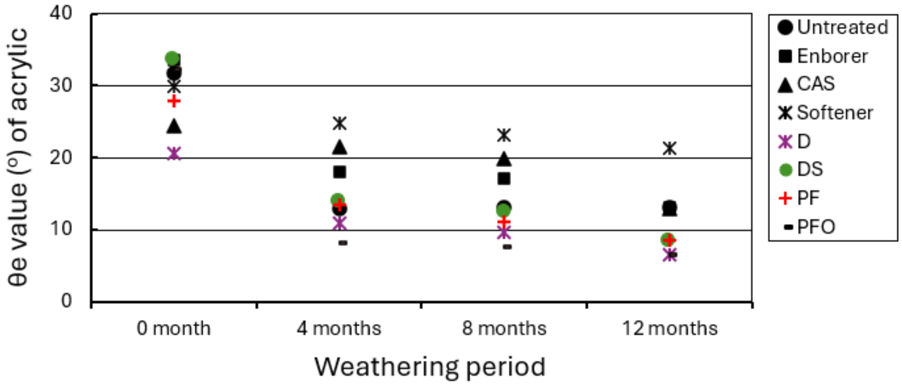

Fig. 4: Contact angle equilibrium of acrylic paint of untreated and modified Scots pine woods before and after weathering.

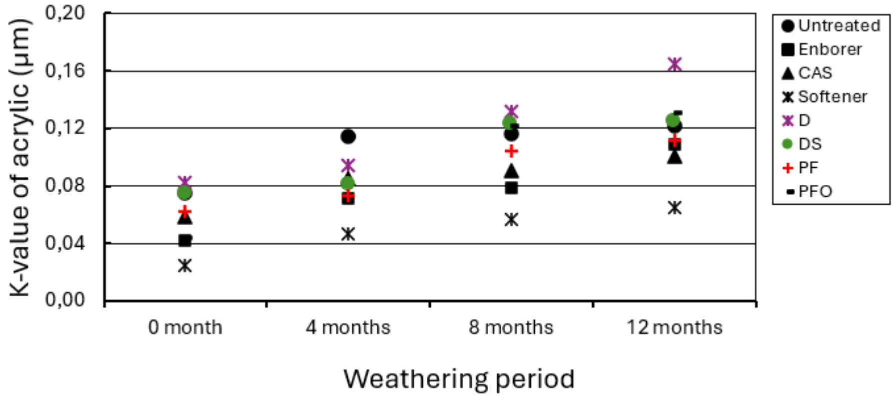

Fig. 5: Wettability (K-value) of acrylic paint of untreated and modified Scots pine woods before and after weathering.

Bonding quality

The test results of bonding quality according to treatments and weathering period are presented in (Tab. 2). The visual appearance of untreated and modified Scots pine woods before and after cross cut test for weathering period of 4 , 8 and 12 months are shown in (Fig. 6).

Tab. 2: The test results of adherence of the coating layer according to ASTM D 3359-02 (2007).

Treatments	Weathering period		
	4-months	8-months	12-months
Untreated	5B	-	-
Enborer	2B	3B	4B
CAS	3B	3B	4B
Softener	0B	1B	1B
D	1B	3B	3B
DS	1B	2B	2B
PF	1B	1B	2B

PFO	2B	2B	2B

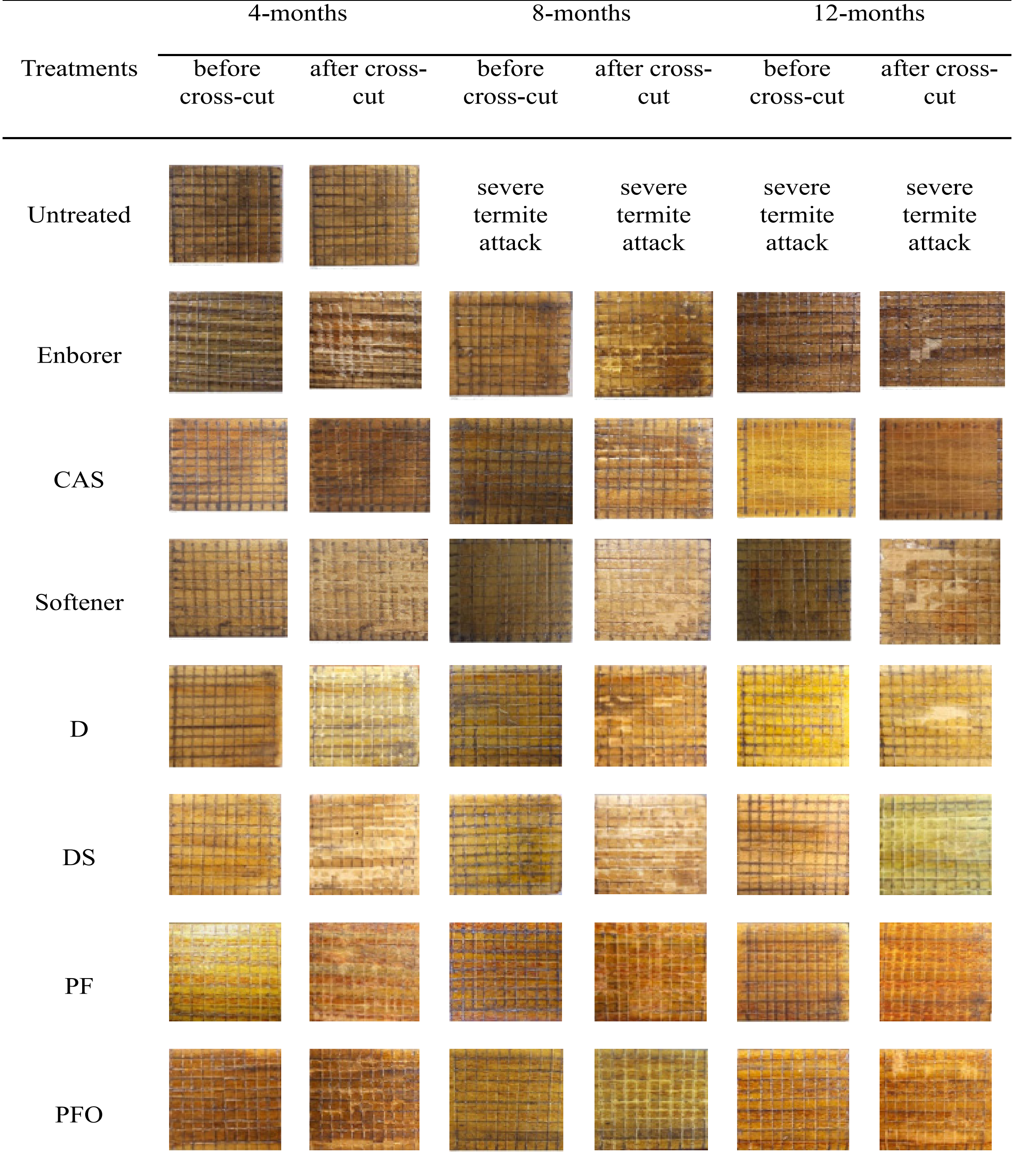

Fig. 6: Visual appearance of untreated and modified Scots pine woods before and after cross cut test for weathering period.

The larger cross-cut test number indicated good bonding quality between the acrylic paint and Scots pine wood. After 4-months weathering, the bonding quality of Scots pine wood was classified as grade 5B. However, the bonding quality of Scots pine wood were classified as less than grade 3B after chemical modification. Chemical modification of Scots pine wood produced lower bonding quality compared to untreated wood. This might be due to the more hydrophobic surface properties of Scots pine modified wood. It caused a decrease in the SFE value and wettability, which could reduce the bonding quality.

In contrast, the results in Tab. 2 show that the bonding quality class increased as the weathering period increased. This could be due to the increase in surface roughness of Scots pine wood after weathering. Weathered wood was frequently exposed to rain and UV light which caused the particles in the wood to loosen, therefore this led to an increase in the surface roughness. It could improve the adhesion bond between the coating and the wood surface. Yuningsih et al. (2019) reported that a rough surface is proposed to improve intrinsic adhesion by providing a larger interfacial area and some mechanical interlocking mechanism. Increasing the interfacial area will increase the total energy of surface interaction. In addition, the bonding quality of acrylic paint increased as the wettability increased. Wettability has a significant influence on bonding quality (Rathke and Sinn 2013). As shown in Fig. 5, the K-value increased as the weathering period increased. Other studies reported that a rough surface can increase the wettability resulting a stronger mechanical bond (Darmawan et al. 2017; Yuningsih et al. 2019).

CONCLUSIONS

Chemical modification by enborer, softener, citric acid-sorbitol (CAS), DMDHEU (D),DMDHEU-softener (DS),phenol-formaldehyde (PF),phenol-formaldehyde-bio-oil (PFO) has an influence on the surface characteristics (surface roughness, total SFE, and wettability) of Scots pine wood. Chemical modification increases the surface roughness up to 7.99 µmand decreases SFE value up to 35.67 $mJ \cdot m^{-2}$. These two factors contribute to a decrease in the wettability and bonding quality of Scots pine. The decrease in K-value (0.065-0.165) and bonding quality (class 1-2) indicates that water-based paint coatings are apparently not suitable for chemically treated Scots pine. In addition, the weathering period also affects the surface characteristics. The surface characteristic increase as the weathering period increase. Surface roughness, SFE value, and wettability increase as the weathering period increases. Application of water-based paint on chemically modified Scots pine wood surfaces may be necessary to maintain periodically for exterior purposes. For improving the utilization of chemically modified wood for exterior purposes needs the selection of suitable coating materials to protect the surface from weathering.

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