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Investigation of Roughness and Adhesion Strength Properties of Pine and Poplar Wood Heat Treated in Air and under Vacuum after Artificial Aging

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Abstract: Heat treatment is an eco-friendly and efficient way to improve the defective properties of wood, such as its hygroscopic nature, the lack of dimensional stability, and low resistance against biological degradation, and to produce a green and sustainable wood material for construction and buildings. However, these treatments alter the substrates and could influence the performance of the coating products necessary to maintain the surface features in certain end-use sectors. In this study, the effects of heat treatment in air (HT) and under vacuum (VHT) on the surface properties of Scots pine (*Pinus sylvestris* L.) and poplar (*Populus euramericana*) wood were investigated. For this purpose, the samples were exposed to an artificial aging process. After the aging process, surface roughness and the adhesion strength behavior of the samples were measured. After the HT and VHT, poplar and pine wood samples showed different roughness. While the roughness value parallel to the fibers decreased in poplar wood, it increased in pine wood. The roughness value perpendicular to the fibers increased in both tree species. The maximum roughness value after UV aging was 62,622 in the VHT-200 group. Although with the UV ageing treatment, the adhesion strength was decreased in the samples heat treated in air, it was increased in the samples subjected to vacuum heat treatment. A lower loss of adhesion strength was observed in the heat-treated samples processed under vacuum compared to the heat-treated samples processed in air.

Keywords: adhesion strength; artificial aging; heat treatment; roughness

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1. Introduction

Wood, as a renewable lignocellulosic material, is a very suitable material for building construction due to a myriad of applications. However, wood has a lower dimensional stability than non-recyclable synthetic materials. This issue limits the use of wood because dimensional stability is an important criterion in many fields of application. There are some techniques in which the negative characteristics of wood is decreased and over the past decades, some very effective materials and methods have been developed; the search for environmentally friendly materials and methods is still in progress [1,2]. Wood modification reduces the hygroscopic behavior of wood and is therefore necessary in order to improve its dimensional stability, as well as to boost its resistance to biological organisms such as fungi and insects. Thermal (heat) treatment is performed by heating the wood without the use of any chemicals. This feature makes it one of the most common commercialized wood modification methods [3]. According to Hill [4], thermal modification should be performed between 180 and 260 °C. Although lower temperatures (below 140 °C) caused no significant changes in the properties of wood, the structure of the wood was severely degraded at higher temperatures, which caused the physical, chemical, and mechanical properties of the wood to change [5–7]. One of the important physical properties that changes at high temperatures is the color of the wood [8]. Lignin and the chromophores contained in the extractives are responsible for the color of wood. The degradation of hemicelluloses during heat treatment leads to the formation of new chromophores, which as a consequence, cause the color of the wood to change [8]. The darkening of the wood surface increases in parallel with the increase in the temperature of the heat treatment. The higher temperatures used in heat treatment methods to reach physical stability and bio-durability result in the undesirable loss of mechanical properties [9]. Although the effect of heat treatment temperature and duration on the properties of wood material has been evaluated in various studies, research on the effect of the atmosphere used during heat treatment is insufficient. Some studies have experimented with the use of vacuum as a heat treatment medium [10–12]. The boiling point of water is reduced under vacuum, which causes the water to evaporate at low heat treatment temperatures. The effect of water is reduced in wood hydrolyzed during heat treatment. Therefore, heat treatment under vacuum results in lower weight losses [12,13].

Silvaprodukt [10] developed the development of an industrial kiln for thermal wood modification with an initial vacuuming step. In this process, oxygen, which causes wood combustion, is removed from the reactor by means of a vacuum pump [14]. Lin et al. [15] found that the crystallinity of cellulose was increased by increasing in treatment temperature under vacuum. According to Xue-hua et al. [16], vacuum heat treatment was found promising to improve the dimensional stability of wood and in keeping mechanical properties at 160 and 200 °C. Norway spruce and fir treated with the vacuum-heat process showed higher dimensional stability and durability against brown-rot and white-rot fungi, as compared to untreated samples [12]. In addition, the weight loss of the wood samples treated with the vacuum-heat systems was considerably lower than those treated according to the Thermowood process [17]. Most recently, Sivrikaya et al. [18] quoted that Scots pine wood samples treated with the vacuum-heat process showed lower weight loss, higher lightness, and lower color changes in comparison with that from solely a heat treatment method. The equilibrium moisture content (EMC) of wood treated with the vacuum-heat process was considerably reduced as a result of the degradation of the carbonyl groups in xylan, and the loss of carbonyl group linked to the aromatic skeleton in lignin due to the treatment [19].

There are some studies on the heat treatment of wood samples under vacuum. However, there is no study on the UV aging of vacuum heat treated samples. Therefore, in this study, the effect of UV aging on pine and poplar wood heat treated under vacuum (VHT) was determined by measuring surface roughness and adhesion strength resistance.

2. Experiments

2.1. Materials

Scots pine (*Pinus sylvestris* L.) and poplar (*Populus euramericana*) specimens were prepared from sapwood blocks in dimensions of 75 (R) $\times$ 15 (T) $\times$ 150 (L) mm^3. The specimens were free of macroscopic defects such as knots and splits. The oven-dry density of the pine and poplar samples used was 660 kg·m^{-3} and 280 kg·m^{-3}, respectively.

2.2. Heat (HT) and Vacuum Heat Treatment (VHT)

Prior to heat treatment, all the specimens were oven-dried at 103 °C. The samples placed in the oven and in the pressure chamber were subjected to heat treatment for 2 h at 180–200 °C for the poplar samples and 190–212 °C for the Scots pine samples. After treatment, the samples have about 0-2% humidity.

The heat treatment in the air medium (HT) was carried out in an oven (Memmert INB200) and no water vapor or other gases were present in the environment.

For the vacuum heat treatment (VHT), the oven-dried samples were placed in a vacuum pressure chamber (Jeiotech OV-11), where a vacuum of 675 mmHg was achieved. The samples were placed in the oven until the target temperature was reached.

The mass loss as a result of the heat treatments was then determined by oven drying. Finally, the modified samples were stored for two weeks in a controlled environment at

20 °C and 65% relative humidity (RH). The weight loss values in the samples after heat treatment were calculated according to the formula below [4]:

$$\text{Mass loss} = ((m_1 - m_2)/m_1) \times 100 \quad (1)$$

2.3. UV-IR Aging

Six treated wood and control samples were subjected to an ultraviolet light combined with infrared radiation (UV + IR) aging test. This test was performed using an ultraviolet and infrared radiation quartz lamp (VT 800, FAMED Łódź S.A., Łódź, Poland) with a radiation energy of 740 W. During the measurement, the wood samples were arranged at an angle of 45° and 40 cm from the lamp. The wood samples were irradiated for 4 h.

2.4. Surface Roughness Measurements

Measurements of the surface quality were performed using a MicroProf FRT instrument (Fries Research and Technology GmbH, Bergisch Gladbach, Germany). The roughness measurements were taken for all samples prior to and after the UV-IR aging treatment. Surface roughness measurements were made according to the relevant literature [20].

2.5. Adhesion Strength Measurements

The adhesion strength of the coatings was evaluated by means of pull-off testing according to EN ISO 4624 standard [21]. A PosiTest-AT adhesion strength tester (DeFelsko Corporation, Ogdensburg, NY, USA) was employed for the adhesion strength evaluation of the specimens coated with the abovementioned lacquer. Three random measurements were taken from each sample by gluing small 20 mm-diameter steel dollies to the film surface using a two-component silane-epoxy resin (Jowat 690.00). Tests were performed under ambient conditions (23 ± 2 °C and 60 ± 5% RH). After seven days of curing, incisions were made around the glued dollies to prevent failure damages near the tested area. The adhesion strength was measured using a hand-operated PosiTest device. The force in MPa required to delaminate the sample was recorded and the surface was evaluated visually. The adhesion strength value of the finishing was also recorded in MPa on the display of the pull-off testing unit and the delamination was evaluated visually for each specimen.

3. Result and Discussion

3.1. Weight Loss of HT and VHT Samples

The weight loss values of the poplar and Scots pine samples, which were heat treated after reaching full dry weight, are given in Figure 1. In addition, standard deviation values are given in parentheses and Duncan test results are given as letters next to the mean values.

All wood samples were brought to oven dry weight before heat treatment. For this reason, the weight losses of wood samples after heat treatment were found to be low. Weight losses of 2.24%–6.35% were observed in the HT Scots pine samples treated at 190–212 °C and losses of 0.65%–2.4% in the poplar samples treated at 180–200 °C. However, lower weight losses were obtained after heat treatment under vacuum. Weight loss in the range of 1.2%–2.1% was observed in the VHT samples of Scots pine and in the range of 0.6%–0.9% in the poplar samples. Weight loss is one of the most important features that change during heat treatment. The wood types varied depending on the heat treatment medium, temperature, and duration [22,23]. For Scots pine heat treated at 210 °C, a mass loss of 13.6% was reported [18]. However, in the study presented here, heat treatment at 212 °C resulted in a mass loss of only 6.32% and at temperatures below 190 °C, loss was quite low (Figure 1). The weight loss values of the poplar samples after heat treatment in air and under vacuum were found to be lower than in the Scots pine samples. MacLean [24] reported that after heat treatment in water, steam, and air environments, the weight loss in wood of deciduous trees was higher than in that of coniferous trees. However, it was revealed that the wood of coniferous trees is more sensitive to heat treatment in a dry environment. The lack of oxygen during the heat treatment in a vacuum environment reduces the weight loss values. In the study of Ferrari et al. [25], weight loss values increased as an effect of a reduced vacuum medium, but at low temperatures, oxygen in the environment was not important. The results of our study showed that at 180 °C, there was no statistically significant difference between poplar wood subjected to heat treatment under vacuum or subjected to heat treatment in air.

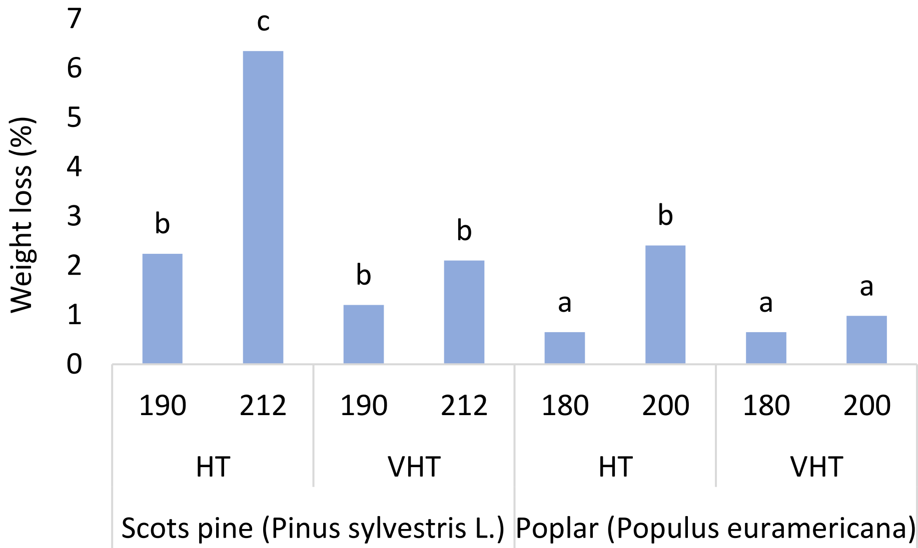

Figure 1. Weight loss (%) of heat and vacuum heat treated Scots pine and poplar wood. (The same letters on the columns denote no statistically significant differences between the groups.)

3.2. Surface Roughness

The average roughness values of the poplar and Scots pine samples subjected to heat treatment are shown in Table 1, of samples exposed to UV-IR treatment after heat treatment in Table 2. The surface roughness was measured parallel to the fibers (||) and perpendicular to the fibers (⊥). The three measurement parameters (Ra, Rz and Rmax) important for surface roughness are given in the tables.

The roughness values of the poplar wood measured parallel to the fibers decreased compared to the post-treatment controls. The roughness continued to decrease with the increase in processing temperature to HT. Roughness obtained in the samples after VHT showed similar behavior to those subjected to HT. However, roughness values increased with the increase in vacuum heat treatment temperature. Roughness values measured perpendicular to the fibers decreased with increasing temperature. In the VHT samples, roughness increased with increasing temperature. In both HT and VHT samples the roughness values parallel to the fibers were lower than in the control samples, whereas the roughness values measured perpendicular to the fibers were found to be higher than the control samples.

In the Scots pine samples, the roughness values measured parallel to the fibers increased after the heat treatment. With the increase in duration of the heat treatment, roughness decreased in the HT samples, while surface roughness increased in the VHT samples. Maximum roughness values were obtained in the samples heat treated at VHT-212 °C. The results obtained with the measurements parallel to the fibers also applied to measurements perpendicular to the fibers.

In our study, it was observed that the roughness values of the poplar samples exposed to heat treatment decreased, while those of the Scots pine samples increased. Bakar et al. [26] suggested that the improvement of surface quality, such as smoothness with heat treatment, may be due to biochemical changes in the cell wall, possibly at elevated temperatures. In the literature studies, it was stated that the surface quality of the samples increased after heat treatment [27–29]. It was also reported that the roughness values were reduced with the increase of heat treatment temperature [30,31]. It was stated that surface roughness had decreased by 10%–25% after heat treatment at 190 and 212 °C [27,28]. Another study found that the roughness values of Scots pine samples had increased after heat treatment. It was stressed that after heat treatment at 190 and 212 °C, Ra and Rz values were increased. However, there were no statistically significant differences between the heat treatment temperatures [32].

Table 1. Average surface roughness values of poplar and Scots pine after heat (HT) and vacuum heat (VHT) treatment.

Wood Species	Modification Parameters	Along (‖) the Grain			Across (⊥) the Grain		
		Ra	Rz	Rmax	Ra	Rz	Rmax
		Roughness [μm]					
Poplar	Control	3.821 (1.12)	23.479 (6.10)	40.338 (9.47)	4.048 (0.91)	27.261 (4.15)	44.183 (10.56)
	HT-180 °C	2.754 (0.96)	17.466 (6.37)	29.012 (11.04)	5.741 (1.79)	30.309 (9.19)	47.659 (16.91)
	HT-200 °C	2.517 (0.96)	16.703 (5.83)	27.667 (12.48)	4.952 (1.52)	31.570 (8.40)	43.090 (11.64)
	VHT-180 °C	2.423 (0.86)	17.333 (6.75)	19.080 (10.09)	4.853 (1.18)	27.838 (6.21)	38.011 (10.65)
	VHT-200 °C	3.171 (1.11)	19.375 (5.12)	36.098 (11.96)	5.226 (1.77)	33.026 (7.08)	50.719 (11.11)
Scots Pine	Control	3.677 (0.92)	23.266 (5.98)	30.662 (7.83)	4.498 (0.78)	29.370 (4.64)	38.360 (8.43)
	HT-190 °C	4.923 (0.72)	30.914 (4.21)	42.435 (4.63)	5.101 (0.78)	33.348 (3.50)	45.263 (5.35)
	HT-212 °C	3.712 (0.99)	23.626 (5.58)	31.461 (8.89)	4.243 (0.82)	27.024 (4.25)	35.997 (6.73)
	VHT-190 °C	3.686 (0.77)	22.788 (4.95)	29.903 (5.25)	5.462 (1.58)	32.502 (7.46)	39.987 (8.52)
	VHT-212 °C	4.198 (0.59)	27.174 (3.90)	36.738 (5.31)	5.425 (0.75)	33.746 (4.52)	47.268 (6.80)

Table 2. Average surface roughness values of poplar and Scots pine after UV-IR exposure.

Wood Species	Modification Parameters	Along (‖) the Grain			Across (⊥) the Grain		
		Ra	Rz	Rmax	Ra	Rz	Rmax
Poplar	Control	2.404 (0.21)	14.856 (1.40)	21.526 (5.47)	5.201 (1.38)	31.579 (1.31)	44.097 (6.02)
	HT-180 °C	3.007 (0.42)	18.914 (0.61)	30.023 (4.47)	4.547 (1.34)	29.673 (6.12)	38.207 (4.42)
	HT-200 °C	2.491 (0.69)	12.662 (1.45)	27.014 (7.23)	6.558 (1.10)	36.892 (2.57)	57.753 (6.73)
	VHT-180 °C	4.972 (1.25)	27.098 (6.00)	48.254 (7.30)	5.357 (1.11)	28.556 (0.71)	42.675 (6.01)
	VHT-200 °C	2.566 (1.8)	14.305 (2.36)	21.038 (3.81)	6.088 (1.25)	32.362 (2.00)	62.622 (0.55)
Scots Pine	Control	5.970 (1.21)	35.940 (1.97)	57.670 (1.71)	5.230 (0.85)	36.489 (10.75)	47.012 (14.06)
	HT-190 °C	5.091 (0.37)	33.717 (2.78)	46.675 (6.88)	4.174 (0.60)	26.077 (2.39)	31.655 (3.36)
	HT-212 °C	3.644 (0.43)	22.965 (3.37)	28.106 (4.65)	3.556 (0.57)	22.568 (2.97)	32.537 (3.24)
	VHT-190 °C	2.620 (0.49)	19.403 (3.46)	24.890 (1.61)	4.073 (1.24)	27.152 (6.63)	36.438 (12.26)
	VHT-212 °C	3.533 (1.02)	22.803 (11.84)	30.917 (21.58)	4.726 (0.75)	26.860 (0.32)	33.610 (5.48)

The UV-IR process led to a decrease in the roughness values of the poplar control samples parallel to the fibers, while the roughness values perpendicular to the fibers increased. The resistance in related to the influence on roughness against UV rays was exhibited in both HT and VHT samples because the samples treated and untreated by UV had similar roughness values. The roughness of VHT-180 increased after the UV probably due to surface cracks, and changes of surface composition. The increase in temperature in both HT and VHT samples increased their resistance to UV rays. Although in the case of the direction perpendicular to the fibers, this situation was the opposite. Minimum roughness values were obtained at HT-200 °C. Surface roughness increased with weathering time [33–35] because of the surface erosion [36]. Wood surface loses consistency and becomes friable, and splinters and fragments break off the surface with the combination of UV, water, temperate and cold factors in outdoor [37].

With the UV process, all roughness parameter values increased significantly in the Scots pine samples. The roughness values of both the control and test samples increased after UV treatment. The results are similar to those found in the literature [38–40]. With the increase of the heat treatment temperature, resistance to UV rays increased in the HT-samples, whereas it decreased in the VHT-samples. The roughness of the wood surfaces after the weathering process was increased with the increase in duration.

The increase of the wood surface roughness can be explained by the degradation of the wood polymers [41]. Although in the Scots pine samples all roughness values of heat-treated sample surfaces decreased, there was no significant change in the poplar samples, except for VHT-180 °C. After UV treatment, the roughness values (Ra) decreased by 56% in the VHT-190 °C samples, while they increased by 106% in the VHT-180 °C samples compared to the controls.

3.3. Adhesion Strength Values

The adhesion strength of the heat and vacuum heattreated samples were measured. In this study, the heat treatment methods, heat treatment temperatures and the UV process all acted upon the adhesion strength. The adhesion values obtained in the poplar and Scots pine samples are given in Figures 2 and 3.

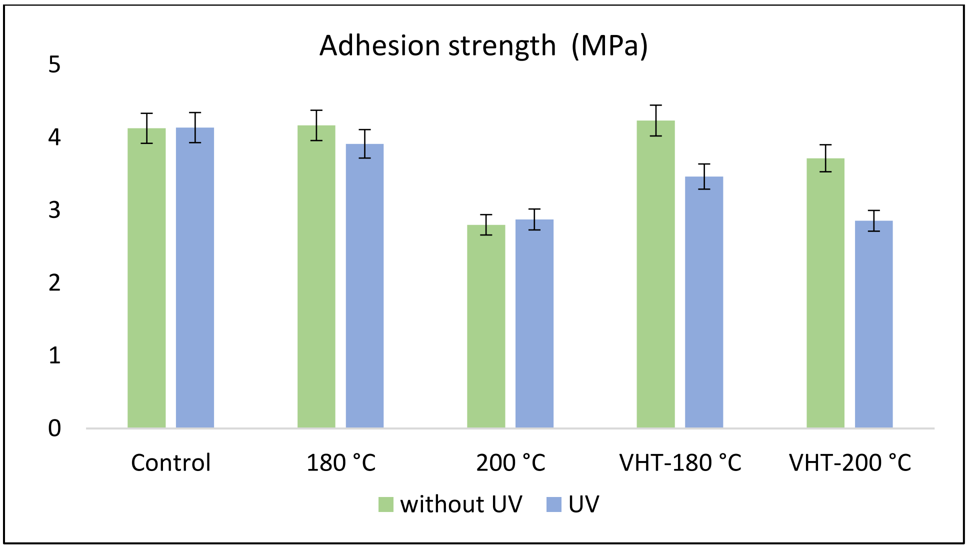

Figure 2. Average adhesion strength values of modified poplar wood samples.

The adhesion strength obtained were 4.13 MPa in the non-UV treated poplar control samples and 4.80 MPa in the Scots pine samples. The roughness of the surface creates a non-homogeneous contact area between the surface and the coating, resulting in poor adhesion strength. In our study, the Ra/Rz/Rmax roughness values of the poplar samples were 3.821/23.479/40.338, respectively, while the Ra/Rz/Rmax roughness values of the Scots pine samples were 3.677/23.266/30.662. A rough wood surface prevents strong bonding between the surface and the coating [42]. Therefore, the adhesion strength decreases. In a previous study, the samples with the smoothest surfaces exhibited maximum adhesion strength with polyurethane-based varnish [43].

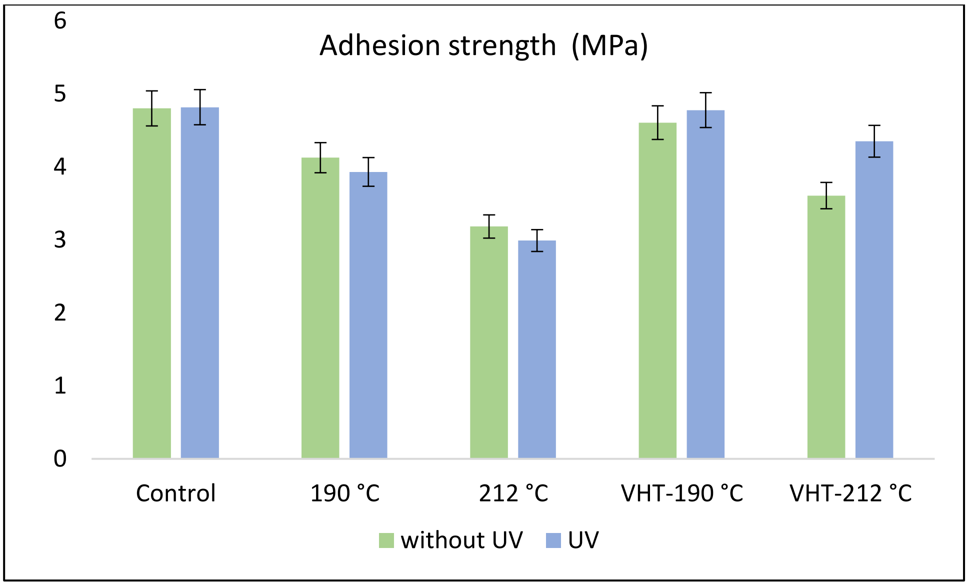

Figure 3. Average adhesion strength values of modified Scots pine wood samples.

The adhesion strength values of the samples decreased with heat treatment application. In addition, the decrease in the adhesion strength increased with the increase in heat treatment duration. A similar situation was observed with both poplar and pine samples. The Scots pine was more affected by heat treatment than the poplar. After heat treatment in air, the adhesion strength decreased by 14%–33%, while after heat treatment under vacuum it decreased by 4%–24%. Samples subjected to heat treatment under vacuum and those subjected to heat treatment in air exhibited similar behaviours. However, higher adhesion strength were obtained in heat treatment samples processed under vacuum compared to those heat treated in air. In the poplar samples at VHT-180 °C, the adhesion strength was higher than in the control samples (4.23).

After heat treatment, water molecules move away from wood surfaces and irreversible bonds are formed between the molecules in the cell walls. Therefore, the adhesion strength of wood surfaces is thought to be increased [44–46]. However, with the increase of heat treatment temperature, the losses in wood cell components increase, resulting in increased losses in physical and mechanical strength [4,47].

The cellulose content of wood samples is reduced after heat treatment. In the literature studies, it is seen that polyurethane-based lacquers bond with cellulose in the wood (C=C, C=O, C=H) and adhesion strength increase due to this bond [48–51].

Both UV-treated and non-UV-treated control samples exhibited the same adhesion strength. The negative effect of both the heat treatment and the UV treatment caused a further decrease in the adhesion strength. However, the case was the opposite with the heat treatment of the Scots pine samples under vacuum. There was a significant increase in adhesion strength after VHT. After UV treatment, the adhesion strength of the VHT-212 °C-treated samples increased by 20% compared to the HT-212 °C samples. However, in general, the adhesion strength value decreased after UV aging. Micro surface cracks and fiber lifts occur in wood samples with UV treatment. This situation causes the adhesion strength value to decrease. Similar results have been demonstrated in the literature study [52,53].

4. Conclusions

After the heat treatment, the surface roughness values measured in the parallel direction were always lower than the values measured perpendicular to the fibers. The roughness values decreased with the increase in the duration of the heat treatment in air. The surface roughness values of the poplar and Scots pine samples heat treated under vacuum increased with the increase of the heat treatment temperature. It can be said that the chemical structure of the samples deteriorated with the increase of heat treatment temperature and structural damages increased with the effect of the vacuum. The same behavior was exhibited by HT-200 and 212 °C and VHT-180 and 200 °C samples. Smoother surfaces were obtained in the poplar samples after heat treatment. Poplar and Scots pine samples exhibited different behaviors against UV rays. Adhesion strength decreased in both the poplar and the Scots pine samples with the increase of heat treatment temperature. Heat treatment affected the adhesion strength of the Scots pine samples more than the poplar samples. The adhesion strength decreased in all variations of the poplar samples with UV-IR exposure, whereas the adhesion strength values of the Scots pine samples increased. However, higher adhesion values were obtained in VHT samples. It is recommended to use VHT applied samples where high adhesion resistance is required.

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Conflicts of Interest: The authors declare no conflict of interest.

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