Surface Activation of Wood by Corona Treatment and NaOH Soaking for Improved Bond Performance in Plywood

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In plywood manufacturing, the surface characteristics of veneers play a critical role in achieving appropriate bonding performance. An inactivated wood surface caused by oxidation or migration of wood extractives has been shown to lead to an insufficient bonding quality. In this study, inactivated birch and spruce veneer surfaces were activated with corona and chemical NaOH treatments. The effects of the treatments were determined by contact angle measurements and bond quality tests conducted with Automated Bonding Evaluation System (ABES). In addition, the mechanical properties of the plywood produced from the treated veneers were evaluated. The results showed that the corona treatment remarkably increased the wettability of the veneer surface and bond quality of both the spruce and birch veneers evaluated by ABES. The corona treatment also improved the mechanical properties of the birch plywood, but the spruce plywood properties were not affected as much. Soaking veneers in NaOH improved the wettability, but the bond strength was lower than that of the references.

Keywords: Plywood; Corona; Plasma activation; Chemical activation; Wood veneers; Wettability

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INTRODUCTION

In adhesive bonding, the surface characteristics of wood play a critical role in achieving appropriate bonding quality. An inactivated wood surface caused by oxidation or migration of wood extractives has been shown to contaminate the surface and lead to an insufficient bond quality. For a good adhesion performance, close contact on a molecular level is required. Surface activity can be evaluated through contact angle measurements. The surface energy of a veneer depends on numerous characteristics, such as the wood species, surface treatment, whether the veneer is made of heartwood or sapwood, cleanliness of the surface, drying method, and age (Kalnins and Knaebe 1992; Gindl *et al.* 2004). Freshness of the wood surface is one of the most critical factors for adhesion (Nussbaum 1999; Gindl *et al.* 2004). However, this property diminishes rapidly when the surface is exposed to air (Christiansen 1990; Nussbaum 1999; Wålinder 2000). Other mechanisms that contribute to a loss of surface activity are high drying temperatures, long drying times, low dryer humidity, and low moisture content (Christiansen 1990).

Surface activation with chemicals can improve the wettability and bond performance. However, many chemicals have environmental or cost-related issues.

Sodium hydroxide (NaOH) is widely used in processing of cellulosic materials. It activates wood surfaces by degrading wood extractives (Hse and Kuo 1988). NaOH swells the wood structure for better permeability, but hydrogen bonds can be broken, which diminishes the moisture tolerance of the glue line (Balfas 1994). According to Christiansen (1991), soaking veneers in a NaOH solution is more effective than dipping.

Wood surfaces can also be activated with plasma (Sakata *et al*. 1993). This method possesses many favourable aspects. The modification of surface characteristics occurs only at the very surface of the material. Hence, plasma treatment does not change the bulk properties of the material, as can happen when using chemical activation (Aydin and Demirkir 2010; Acda *et al.* 2012). Plasma treatment, especially when using the low-pressure option, is shown to be a cost-effective and environmentally friendly method with electricity as the only requirement and ozone produced as an emission (Aydin and Demirkir 2010; Rowell 2013). A point-to-plane corona discharge can be used as an alternative to plasma. The discharge is created at atmospheric pressure between a point-like tip and a planar electrode (Schütze *et al.* 1998). The energy density of the discharge is low; therefore, the intensity of the treatment also remains quite low as the density of charged particles decreases drastically as the distance from the electrode tip increases (Schütze *et al.* 1998). Increasing the energy density would also increase the temperature, which is undesired when treating wood (Rowell 2013). The effect of plasma activation on the wettability is mainly positive, although it is also dependent on the wood species and gas mix used, which can lead to an increased hydrophobicity (Avramidis *et al.* 2009; Wascher *et al.* 2014). According to Tino and Smatko (2014), the hydrophobicity increases as the distance from the electrode increases, and it was also concluded that using air as a gas mix creates the best results for industrial applications when compared with CO_2 and N_2. At its best, activation can be implemented in the process without compromising the production capacity.

The bonding quality is an important parameter because it determines the suitability of plywood for a certain environment and end use. Several variables contribute to the bonding quality, with the wood species and adhesive properties being the most crucial variables. The standards EN 314-1:2004 and EN 314-2:1993 define the limits within which the quality must be so that the plywood can withstand the conditions of the intended use. The automated bonding evaluation system (ABES) has successfully been used to determine the bonding performance of veneers with a high accuracy and reliability (Ferra *et al.* 2011; Costa *et al.* 2012; Rohumaa *et al.* 2013, 2014; Yamamoto *et al*. 2017). It facilitates easy comparison of different veneer treatments and materials. The aim of this study was to determine how corona and NaOH treatments affect the properties of birch and spruce veneer surfaces, which were inactivated during storage. The impacts of the activation treatments were studied with contact angle measurements, veneer glue line shear strength tests, as well as bending strength and bonding quality tests for plywood made from the treated veneers.

EXPERIMENTAL

Sample Preparation

The materials used in this study were new and old 0.8 mm thick birch (*Betula pendula* Roth) and spruce (*Picea abies* L.) veneers, which were the suitable thickness for the bonding performance test. The 0.8 mm thick veneers were produced from logs soaked at either 20 °C or 70 °C. The logs were peeled with a rotary lathe, and the veneers were prepared as described in Rohumaa *et al.* (2016a). For the panel tests, 1.5 mm birch and spruce veneers were produced. The trees for the 0.8 and 1.5 mm veneers were felled in Southern Finland (Vihti, Finland) in the winter of 2014. The old 0.8 mm thick veneers were stored at Aalto University for five years. The logs were stored outside for 1 to 4 months. All of the veneers were stored at 20 °C and 35% relative humidity (RH) until they were used.

Corona Treatment

The corona treatment was applied to the veneers produced from the logs soaked at 20 °C using a LabTEC corona system (Tantec A/S, Lunderskov, Denmark). The veneers were cut to the dimensions 120 x 250 mm before the treatment. The intensity of the corona treatment was set to 0.1 kWh/m^2. The voltage setpoint was 28 kV. However, the true value was around 24 to 25 kV because the wood acted as an insulator. The required treatment time (t) was calculated based on the surface area (A) and true power (P),

$$t = A\ (\text{m}^2) \times 360{,}000\ (\text{J m}^2) / P\ (\text{W}) \tag{1}$$

The appropriate power was determined to be 125 W, which led to a required treatment time of 1 min and 26 s. In that time, the veneer was treated 84 times from end to end of the veneer. After the treatment, the samples were cut to the dimensions 117 x 20 mm. Similar sized samples were prepared for the bonding strength test.

Contact Angle Measurements

Contact angle measurements were conducted to determine the hydrophobicity of the veneer surface. The tests were done using the sessile drop method on a CAM 200 contact angle meter (KSV instruments Ltd., Helsinki, Finland). Distilled water was used as a test liquid. The measurements were conducted in a normal uncontrolled room atmosphere. The water droplet was 6.7 µL. The motion and absorption of the droplet were recorded every 200 ms for the first five seconds, and after that, every five seconds for up to 70 s (from the droplet release). For the contact angle measurements, ten veneer samples (20 x 117 mm) were measured per series. Three contact angle measurements were done on each veneer sample, which resulted in a total of 30 measurements per series. All of the series were stored at 20 °C and 35% RH. The corona-treated and reference samples were first measured 6 h after the corona treatment, and the final measurement took place 42 days later. In the beginning, measurements were made every few days, and then later once a week.

Determination of the Bonding Performance by ABES

Automated Bonding Evaluation Systems (ABES; Adhesive Evaluation Systems, Inc., Corvallis, OR, USA) was used to determine the bonding performance of the veneers. The thickness of the ABES samples was 0.8 mm and the moisture content (MC) was 7%. In order to determine the effects from the corona treatment, the veneer samples from the logs soaked at 20 °C were treated with corona in the same manner mentioned above and with a treatment time of 71 s.

Liquid phenol formaldehyde (PF) resin (Prefere 14J021, Prefere Resins Holding GmbH, Erkner, Germany) was dropped onto the specimen and spread over an area of 5 x 20 mm using other specimens, which led to a glue spreading extent of 100 g/m^2. The press temperature was 130 °C, and the pressure was 20 PSI (2.21 MPa) for birch and 13 PSI

(1.44 MPa) for spruce. Immediately after pressing, the device automatically determined the shear strength.

The effect of the open time was also tested in order to determine whether an increased absorption time improves the bond quality. The effect was tested on the new, old, and corona-treated 0.8 mm thick birch veneers every five minutes for 30 min, and then every ten minutes for up to 60 min. The open time was followed by ABES testing with a cure time of 160 s at 130 °C and a surface pressure of 2.21 MPa.

Chemical Surface Activation

Sodium hydroxide (NaOH) (Merck & Co., Inc., Branchburg, NJ, USA) was used for the chemical surface activation. The new 0.8 mm birch veneers were chemically treated in the following ways: 1) soaking in 1 M NaOH solution, 2) soaking in the NaOH solution followed by rinsing with tap water, and 3) soaking in tap water. The duration of soaking was 5 min. The veneers were air dried and stored at 20 °C and 35% RH until an equilibrium moisture content (EMC) was reached.

Plywood Preparation

Seven-ply plywood samples with the approximate dimensions of 400 x 400 mm were made from the 1.5 mm birch and spruce veneers. The average thickness of the plywood was 9.5 mm. Both the birch and spruce veneers were treated on both sides with corona, except for the plywood surface sheets. The plywood was prepared with the PF adhesive, and the glue spread ratio was 160 g/m^2 for birch and 130 g/m^2 for spruce, the ratio adjusted to different surface characteristics of the two wood species. Pre-pressing was carried out for 8 min with a surface pressure of 0.8 MPa. Hot pressing was conducted for 8 min at 128 °C with a pressure of 1.8 MPa for birch and 1.1 MPa for spruce. The plywood was conditioned at 20 °C and 65% RH for a minimum of four weeks. The mechanical properties of the plywood were tested according to EN 310 (1993) and EN 314 (2004). For the modulus of rupture (MOR), 18 samples were tested parallel to the grain and 17 samples were tested perpendicular to the grain in all of the test groups. For the glue line shear strength, the samples were divided into two groups, those that were tested in dry conditions (33 pieces per test group) and those soaked in water for 24 h at 20 °C (40 pieces per test group). The one-way independent samples t-test (parametric) and Mann-Whitney U-test (non-parametric) for comparing means were used based on the normal distribution of the samples in order to analyse the effect of the veneer treatment on the plywood strength, and was performed with IBM SPSS Statistics 23 (IBM Corp., Armonk, NY, USA).

RESULTS AND DISCUSSION

Effect of Log Soaking Temperatures on the Contact Angle of the Veneer Surface

The results of the contact angle measurement of the veneers produced from logs soaked at a specific temperature (20 and 70 °C) are summarized in Fig. 1. The contact angle measurements revealed that the higher soaking temperature led to lower contact angles for both the birch and spruce veneers, which was in agreement with the results previously obtained by Rohumaa *et al*. (2014). Higher soaking temperatures, especially over 70 °C, affect the surface characteristics of veneers, including the roughness, colour, and even chemical characteristics: the roughness increases, the surface changes lighter in colour, and some thermolabile chemical compounds, such as flavonoids, might be degraded (Rohumaa *et al.* 2014, 2016a,b; Yamamoto *et al.* 2015).

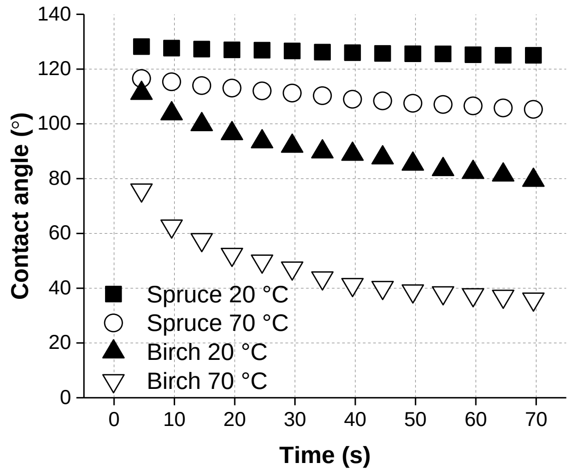

Fig. 1. Contact angles of the birch and spruce veneers prepared from logs soaked at different temperatures

Effect of the Corona Treatment on the Veneer Surface Activity

The contact angles of the reference and corona-treated veneers, as well as the old and new veneers, are compared in Fig 2. The differences in the wettability were insignificant between the old and new reference veneers. The new veneers had lower contact angles than the old ones, which suggested inactivation took place during storage. A notable reduction in the contact angle was observed in the corona-treated veneers. The contact angle decreased by over 100° immediately after releasing the droplet.

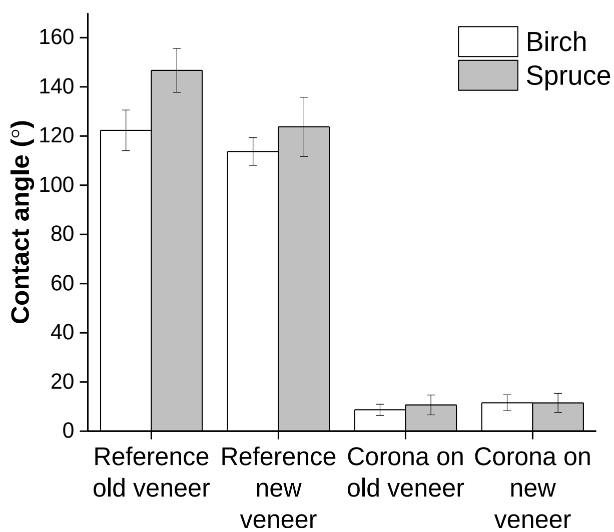

Fig. 2. Contact angles of the reference and corona-treated birch and spruce veneers, and new and old veneers; Contact angles of the reference veneers were measured 0.4 s after contact with the surface, and after 0.04 s for the corona-treated veneers

The veneer surface became more hydrophilic because of the corona treatment, which most likely improved the wettability of the veneer surface. The differences between the reference and treated veneers were quite clear, and showed that the corona treatment improved the water spreading and absorption on the surface, as well as eliminated differences between the new and old veneer surfaces. Aydin and Demirkir (2010) presented similar results when activating old spruce veneers with plasma treatment. Despite the fact that a corona discharge has a restricted area and rather low energy density (Schütze *et al.* 1998), the corona treatment remarkably improved the veneer surface characteristics, such as wettability.

Stability of the Corona Treatment Effect

The stability of the corona treatment over a duration of 42 d was assessed with the contact angle measurements (Fig. 3). The measurement series included old birch veneers from ten different stacks that were measured on different days. The contact angle was measured directly after releasing the water droplet. The contact angle and absorption time increased almost linearly during the experimental period. The contact angle and absorption time correlated well, which proved that the effect of the corona treatment was not permanent and diminished over time. Novak *et al.* (2011) came to a similar conclusion, and assumed that the activity diminished fastest directly after the treatment. Despite the reduction in the effects of the corona treatment, the surfaces of the corona-treated veneers were notably more active 42 d after the treatment than the reference veneer surfaces.

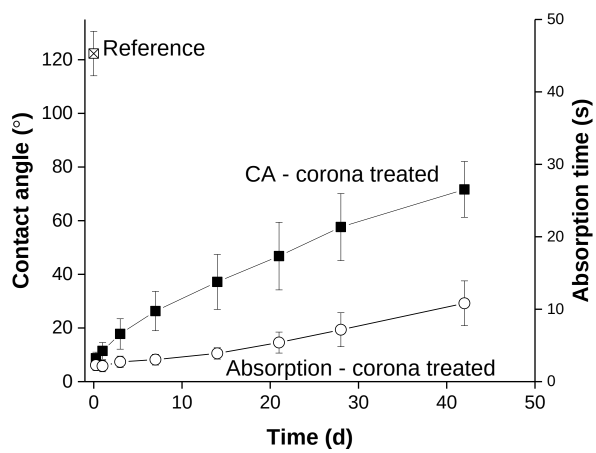

Fig. 3. Contact angles (CA) and drop absorption times of the corona-treated veneer samples over the period of 42 days

Effect of the Corona Treatment on the Bonding Strength

The results from the ABES measurements conducted on the old veneers showed a consistent improvement in the bond strength compared with the reference veneers, which was independent of the press time (Fig. 4). The maximum strength was obtained with a press time of approximately 90 s for the treated veneers, whereas the maximum strength was achieved with a press time of approximately 120 and 160 s for the spruce and birch veneer references, respectively. This finding has an important implication for the reduction of the pressing time in industrial processes when the treatment is applied to full-scale production.

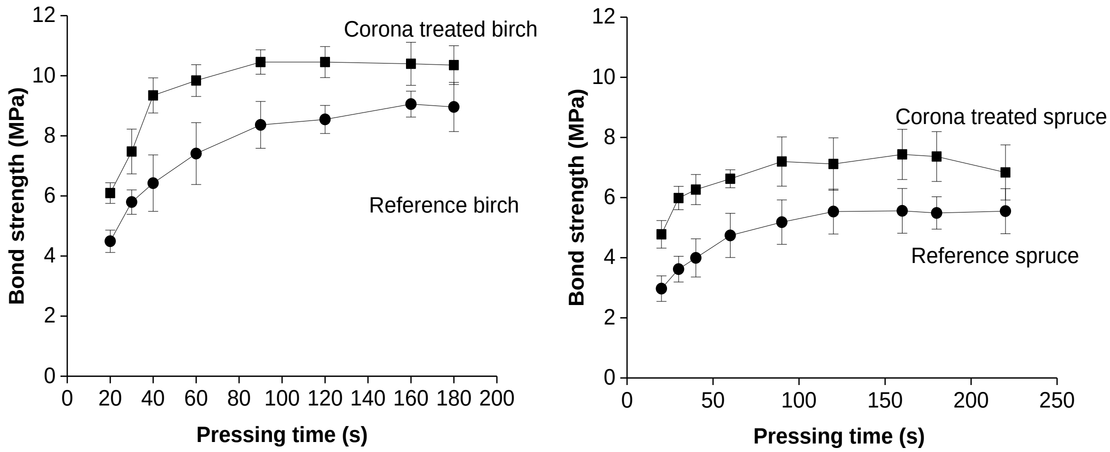

Fig. 4. Bond strength of the reference and treated birch and spruce veneers; Note the different time scales on the X-axes

The corona-treated veneers had higher bonding qualities when hot pressing was conducted immediately after adhesive spreading. However, after increasing the open time, the untreated veneers had similar strengths after 5 min, and the bonding strength of the corona-treated veneer did not increase with time (Fig. 5). The old untreated veneer had a maximum value after 10 min, but the maximum was below the bond strengths of the new and corona-treated veneers. Because the strength did not increase after a certain time, it seemed that a maximum level of activation was reached for that surface. However, with higher adhesive amounts, the corona-treated veneers could show even more improvement.

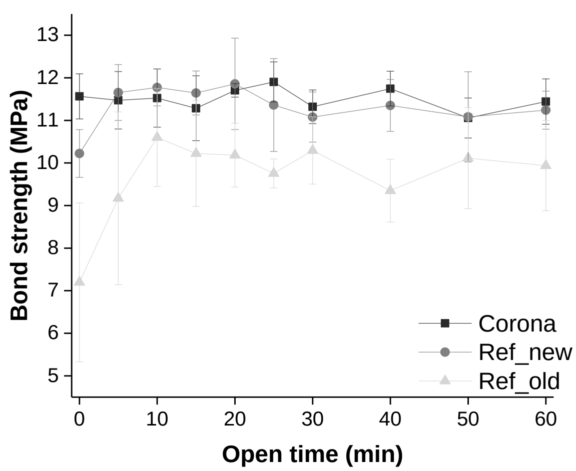

Fig. 5. Bond strength of the corona-treated birch, and new and old reference birch veneers for different open times

Effect of the Chemical Treatment on the Veneer Properties

The surface activity of the chemically-treated veneers increased after soaking in NaOH. Soaking in water had no effect, but rinsing after NaOH soaking increased the contact angle. Compared with the corona-treated veneers, the results were modest. The bond strength results (Fig. 6) indicated that soaking in water had no clear effect on the bonding performance of the veneers. However, after soaking in NaOH, there was a significant reduction in the bonding performance and the lowest bonding strengths were obtained after soaking in NaOH only. Veneers that were soaked in NaOH and rinsed with water had similar bond strengths compared with those of the references. The bond strengths of the veneers soaked in NaOH were lower than those of the references. In comparison with these results, treatment with corona resulted in a remarkable improvement to the bond strength. The NaOH on the veneer surface might have partially interfered with the curing of the PF adhesive after the treatments, which could explain the lower bonding performance if the NaOH-treated veneers. Balfas (1994) suggests that while NaOH is useful in increasing the surface activity, it swells the wood and breaks hydrogen bonds, which may also affect the strength and surface wettability.

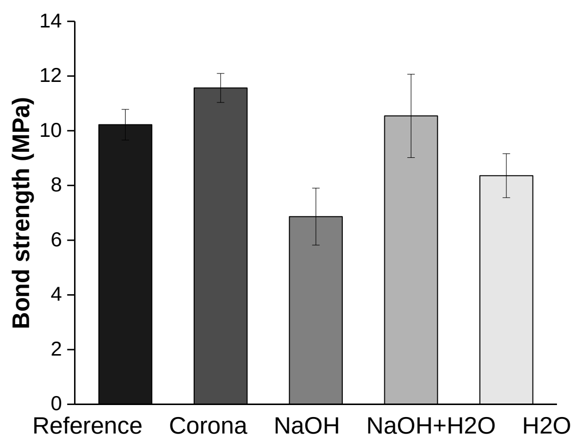

Fig. 6. Bond strength of the variously treated new birch veneers

A remarkable colour change was observed in the veneer samples treated with the NaOH solution, which would be an undesired side effect in the industrial process. A possible explanation for this might have been that chromophores in the wood matrix reacted with NaOH during the treatment. NaOH may also have partially degraded some wood extractives on the veneer surface, such as fatty acids and wax, which generally makes the veneer surface more hydrophobic, and thereby aids adhesive penetration (Christiansen 1991). However, the effect from the degradation of hydrophobic compounds by NaOH was limited in this study, and was also mentioned in the work done by Šernek *et al.* (2004). Wood species and the preparation procedures may also have an effect on the noted results.

Effect of the Corona Treatment on the Mechanical Properties of the Plywood

Mechanical tests were conducted according to EN 310 (1993) on the plywood made from the treated veneers (Fig. 7). The MOR and modulus of elasticity (MOE) of the reference and corona-treated birch veneers were similar. However, the corona-treated plywood had a slightly higher strength for all of the series. The largest difference (3.2%) was in the MOR perpendicular to the grain, which was higher for the corona-treated samples, while the smallest difference (0.9%) was in the MOR parallel to the grain. The MOE difference was the same (2.9%) in both directions and was slightly higher in the corona-treated samples.

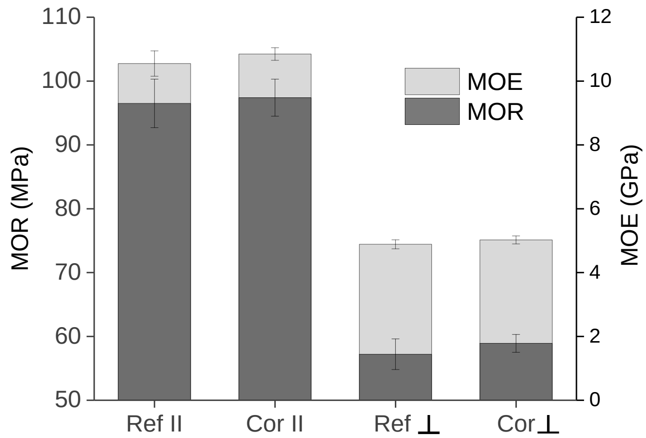

Fig. 7. MOR and MOE of the plywood prepared from the reference (Ref) and corona (Cor) treated birch veneers; II = parallel to the face veneer grain; ⊥ = perpendicular to the face veneer grain

The results of the statistical analysis are summarised in Table 1. The largest difference (3.2%) was in MOR in perpendicular to grain, being higher in corona-treated samples, while the smallest difference (0.9%) was in MOR in parallel to grain. MOE difference was the same (2.9%) in both directions, and slightly higher in corona-treated samples.

Although the differences between the test results were minor, statistically the results were significant. Except for the results of the MOR parallel to the grain, the results were notably different (reference *vs.* corona) for every group. However, differences this small could have also stemmed from the natural variation in the wood structures. The results from the bending test of the spruce plywood were similar to those of the birch plywood (data not shown). However, there was no statistical significance between the results from the treatments. There was more variation in the results from the spruce plywood, which was most likely because of the characteristics of spruce. In general, spruce has a less homogeneous wood surface than birch.

Table 1. Statistical Analysis Based on the Results from the Bending tests of the Birch Plywood

Test	Group	N	Average	difference	SD	p-value
MOR ǁ	Corona	18	97.4	0.9%	2.93	0.226
	Reference	18	96.5		3.81	
MOE ǁ	Corona	18	10.8	2.9%	0.20	0.004
	Reference	18	10.5		0.40	
MOR ⊥	Corona	17	58.9	3.2%	1.40	0.006
	Reference	17	57.1		2.43	
MOE ⊥	Corona	17	5.0	2.9%	0.13	0.003
	Reference	17	4.9		0.14	

Effect of the Corona Treatment on the Bonding Strength Quality of the Birch and Spruce Plywood

The results of the bonding quality test are shown in Fig. 8. **Fig.** Because the orientation of the lathe checks has a large impact on the bonding quality test results (Rohumaa *et al*. 2013), the samples were divided into two groups, one where the lathe check was pulled open and one where it was pulled closed. In general, regardless of the orientation of the lathe checks or pre-treatment, the corona-treated birch veneers showed 5% to 11% higher results for all of the groups. For the closed group, a larger percent difference was obtained compared with the open group. The corona treatment was statistically significant in terms of the shear strength. The wood failure percentage of the reference samples was higher than that of the corona-treated samples.

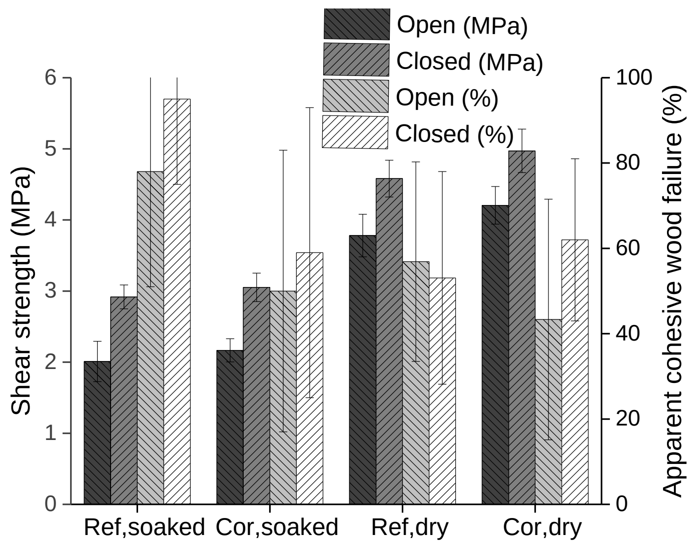

Fig. 8. Bond strength and apparent cohesive wood failure percentage of the birch plywood prepared from the treated veneers; Open = lathe checks pulled open; closed = lathe checks pulled closed

There was no clear difference between the reference and corona-treated spruce plywood (data not shown). The corona treatment slightly improved the birch veneer bonding quality measured with the ABES, but there were no statistical differences between the corona-treated and reference samples (not shown). The soaked corona-treated samples in the group where the lathe checks were pulled open had a slightly higher strength than the reference samples. However, the standard deviation (SD) of the average results of the soaked corona-treated samples was higher than that of the reference samples. The SD of the spruce plywood shear strength was higher than that of the birch plywood. No statistical significance was found for the spruce plywood. For the birch plywood, there was a statistically significant difference between the reference and corona-treated plywood (Table 2).

Overall, based on these results, corona treatment of wood veneer shows promise in improving the surface characteristics and bonding performance of the veneers and plywood. The process of corona treatment is simple, and compared to NaOH soaking, it is rather cost-effective and presents a green option. Re-drying after treatment, storage and handling of chemicals, and final disposal of chemicals are not needed. Plasma also affects only the near surface of the material; this is unlike chemical modification, which alters the bulk properties (Demirkir *et al.* 2014). The cost-effectiveness of corona depends on the local cost for energy and is a viable option for practical use in the industry compared to other surface modification techniques, such as chemical and thermal treatments.

Table 2. Statistical Analysis on the Results from the Bond Strength Tests of Birch Plywood

Test	Group	pre-treatment	N	Average (MPa)	Difference (%)	SD	p-value
Closed	Corona	Soaked	17	3.1	4.5%	0.21	0.022
	Reference		18	2.9		0.17	
Open	Corona	Soaked	19	2.2	5.9%	0.17	0.033
	Reference		17	2.1		0.28	
Closed	Corona	Dry	17	5.0	8.5%	0.32	0.001
	Reference		13	4.6		0.27	
Open	Corona	Dry	15	4.2	11.4%	0.27	0
	Reference		16	3.8		0.31	

CONCLUSIONS

1. The contact angle results showed that the higher soaking temperature (70 °C) remarkably improved the surface wettability of the birch veneer, but the effect of the soaking temperature on the spruce veneer was modest. In general, the results were consistent with an expectation that softwoods are often more susceptible to wood surface inactivation, which could have been the reason for the spruce showing less promising results.

2. The corona treatment demonstrated a great potential to improve the wettability of the veneer surface, which was shown by the reduced water contact angle values for both birch and spruce. This was also shown by the mechanical tests, where the corona-treated birch plywood exhibited a slightly improved bending strength and bond strength compared with the reference samples. In contrast, there were no clear differences in the properties of the corona-treated spruce plywood compared with the reference samples. Although the effect of the contact angle diminished over time, the improvement to the surface activity of the corona-treated samples remained higher than that of the references even after 42 d.

3. Chemical surface activation with NaOH did not result in a significant improvement of the wettability of the veneer surfaces, and it also degraded the quality of the veneers through colour changes and dimensional distortion.

4. Although the bonding quality test, conducted with ABES, showed remarkable improvement to the bonding quality of the corona-treated veneers, the effect of the corona treatment on the plywood bonding quality was modest. This was most likely because of the longer assembly times in plywood preparation, where the adhesive had more time to wet the surface. Testing the increased open time with the ABES also supported this finding. These findings suggested that by increasing the open time, the bonding strength of the reference veneers can achieve strength values similar to that of the corona-treated veneers.

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