SURFACE ENERGY MODIFICATION BY RADIOFREQUENCY INDUCTIVE AND CAPACITIVE PLASMAS AT LOW PRESSURES ON SUGAR MAPLE: AN EXPLORATORY STUDY

*Vincent Blanchard**

Research Scientist

Pierre Blanchet†

Research Scientist and Group Leader
FPInnovations–Forintek Division
319, rue Franquet
Québec (QC), Canada, G1P 4R4
and
Adjunct Professor
Département des Sciences du Bois et de la Forêt
Université Laval
Québec, Canada, G1K 7P4

Bernard Riedl†

Professor
Centre de Recherche sur le Bois, Université Laval
Pavillon G.-H. Kruger
Québec (QC), Canada, G1K 7PE

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Abstract. The wood products industry is going through hard times in both Canada and the US. It is faced with competition from emerging economies and substitution products. The North American economy is slowing down with decreasing demand for wood products. Under these conditions, the industry should be innovative and develop the next generation of wood products. Plasma technology could be used to improve wood surface properties and compensate for the variations to be expected from an organic living material, which is sensitive to its environment (moisture, water, temperature, ultraviolet light). In recent years, the plastic and textile industries have begun experimenting with plasma technology to activate surfaces, mainly to improve coating/substrate adhesion. The literature on potential applications of plasma treatment to wood surfaces is very limited. This report describes the results of an exploratory study on the effect of plasma treatments on sugar maple wood using different gases and mixtures (N_2, H_2, O_2, and Ar) at different pressures (13.3 – 665 Pa). Water wettability and adhesion between surface and waterborne polyurethane acrylate coatings were also studied. The results show that it was possible, under certain conditions, to significantly increase wood/coating adhesion by 30 – 100%. This improvement is correlated with improvements in wood surface energy and coating penetration depth. In addition, chemical analyses showed that, with some plasma types, the treatment led to new atoms being grafted.

Keywords: Surface energy surface properties, plasma, adhesion, sugar maple, wettability, ICP, CCP, mechanical properties, wood, confocal Raman spectroscopy, XPS

INTRODUCTION

In physical and chemical science, plasma can be defined as an ionized gas with a neutral global charge. It is often considered the fourth state of matter and constitutes more than 99% of the universe. In contrast to space and astrophysical plasmas, laboratory plasmas are generally created in closed discharge vessels. Application of an electric and/or magnetic field using, for example, coils or electrodes, accelerates primary electrons

* Corresponding author: vincent.blanchard@fpinnovations.ca
† SWST member

(as a result of ultraviolet and/or cosmic radiation) to energies sufficient to produce ionization of the gas constituents (Lieberman and Lichtenberg 1994; Conrads and Schmidt 2000; Tendro et al 2006). The characteristics of the resulting plasma depend on the applied voltage or current, frequency of alternating current, and a number of other parameters such as nature and pressure of the gas, dimension of the discharge vessel, and so on. Because plasmas are made up of electrons, radicals, ions, and photons, they are extremely reactive and can thus be used for materials processing. The wide variety of operating conditions of laboratory plasmas allows for various laboratory and industrial applications such as hospital tool sterilization, surface energy modification, thin film deposition, submicrometer etching, chemical synthesis, chemical surface analysis, lighting, and so on.

Plasma technology can be used to alter wood surfaces to modify its surface energy (Chen and Zavarin 1990; Lipska-Quinn 1994; Denez et al 2005). Podgorski et al (2000, 2002) studied the influence of various plasma treatments on several fir species. They showed that coating adhesion could be improved under specific conditions. Rehn et al (2003) showed that the fracture strength of glued black locust (*Robinia pseudoacacia*) can be increased and coating delamination reduced. Yang et al (2004) studied the influence of microwave plasma treatment on wood surfaces and observed that the oxygen/carbon ratio was modified after treatment. Several studies investigated plasma polymerization of wood surfaces (Magalhães and Souza 2002; Manolache et al 2008; Kim et al 2009). The fire and moisture resistance of different Philippine wood species was improved after hydrogenous plasma treatment by Blantocas et al (2007). Evans et al (2007) investigated the impact of plasma treatment on wettability and glue-bond strength of four eucalypt wood species. Finally, Wolkenhauer et al (2007, 2008, 2009) studied several properties of wood after plasma treatments. In 2009, they demonstrated that a plasma treatment of wood surfaces was superior to sanding for increasing surface energy.

This survey indicates that plasma processing is a promising approach for treating wood surfaces. This technology is used in the plastic and glass industries to improve adhesion between coating and substrate by modifying surface energy and/or grafting new chemical functions. The textile industry has also been developing new techniques based on plasmas to obtain fibers with better resistance to industrial washing cycles (Tessier D, CTT Group, personal communication, 2008). Despite its great potential, the use of laboratory plasmas in the wood industry remains at an embryonic state and more work is clearly needed.

The main objective of the present study was to assess the potential application of low-temperature plasmas for treating sugar maple wood surfaces. We investigated the influence of different plasma treatments on wettability and wood-coating adhesion and penetration. The coatings used were waterborne polyurethane/polyacrylate, because these are increasingly popular with the wood industry.

EXPERIMENTAL DETAILS AND DIAGNOSTICS

The substrate investigated was sugar maple (*Acer saccharum*) lumber. For each parameter series tested (see Table 1), all specimens came from the same board. The specimens were conditioned at 20°C and 65% RH for 1 wk and sanded at 150 grit before treatment. Each series consisted of eight specimens, each $4 \times 85 \times 100$ mm in thickness, length, and width, respectively.

Two plasma sources were used to functionalize sugar maple surfaces, the schematics of which are shown in Figs 1 – 3. The first was a remote inductively coupled plasma (ICP) (PLASMIONIQUE's PLUME series ICPRS-300; Figs 1 and 3A), whereas the second was a capacitively coupled plasma (CCP) (PLASMIONIQUE's FLARION series CCP-300; Figs 2 and 3B). Both systems used 13.56-MHz radiofrequency (RF) generators. The experimental chamber was the same for both plasma sources and con-

Table 1. *List of parameters and types of plasma studied for radiofrequency (RF) capacitive and inductive plasma treatments.*

	Vacuum level (Pa)			Exposure time (min)			
	13.3	133	665	0.5	10	20	60
RF capacitive plasma							
Ar – 150 W		Yes		Yes	Yes	Yes	Yes
Ar – 180 W		Yes		Yes	Yes	Yes	
Ar – 200 W		Yes			Yes	Yes	
N2 – 150 W		Yes				Yes	
RF inductive plasma							
Ar – 150 W	Yes	Yes	Yes	Yes	Yes	Yes	
N2 – 150 W	Yes	Yes	Yes	Yes	Yes	Yes	
Ar/N2 – 150 W	Yes	Yes	Yes	Yes	Yes	Yes	
N2/O2 – 150 W	Yes	Yes	Yes	Yes	Yes	Yes	
N2/H2 – 150 W	Yes	Yes	Yes	Yes	Yes	Yes	

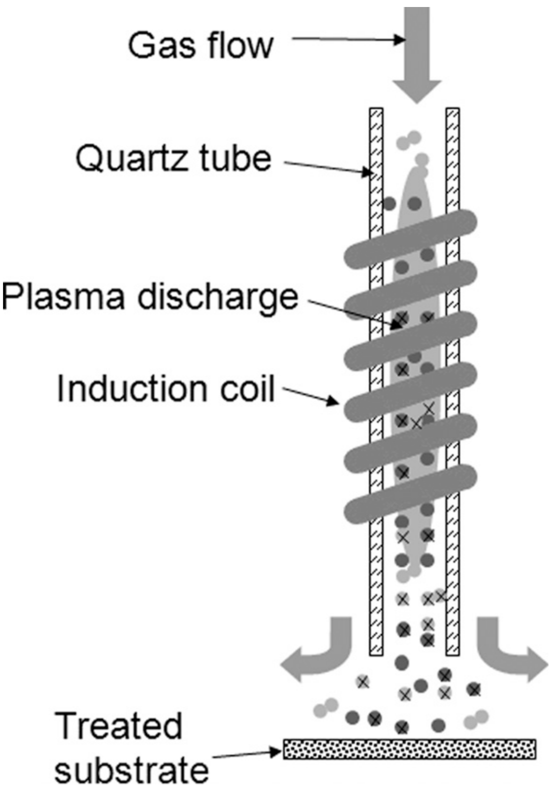

Figure 1. Schematic view of inductive plasma apparatus.

sisted of a stainless steel chamber evacuated by a turbomolecular pump (450 L/s nominal pumping capacity) backed up by a two-stage mechanical pump. For the tests involving the ICP source, the gas mixture was injected from the top of the quartz tube. Gas flow rates were con-

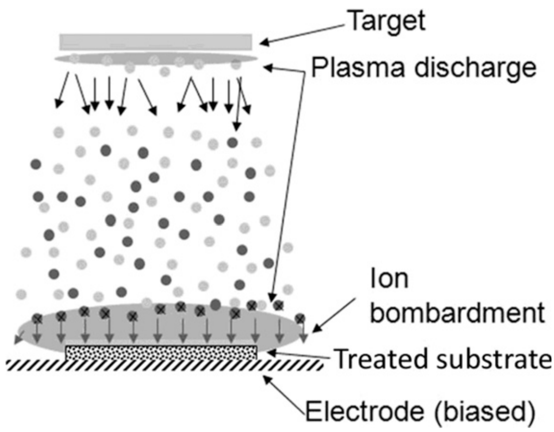

Figure 2. Schematic view of radiofrequency capacitive plasma apparatus. This equipment also allows for sputtering tests but none were conducted in this study.

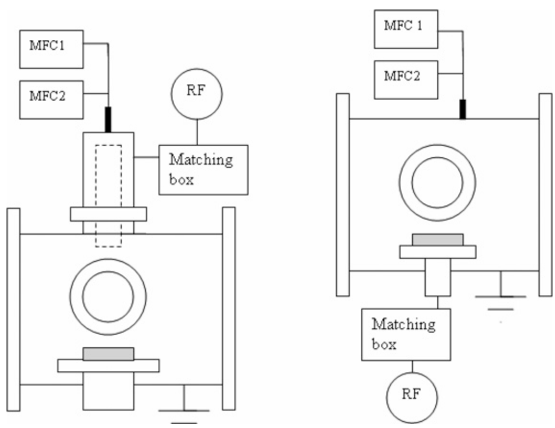

Figure 3. Shematic view of inductively coupled plasma and capacitively coupled plasma reactors used for this experiment (Plasmionique–Montréal). P_w and P are the plasma power and pressure, respectively, in the closed vessel.

trolled by two electronic mass flow controllers. The ICP source was configured to operate with its Faraday filter to avoid capacitive coupling and erosion of the quartz discharge tube. At high flow rates, pumping was done by the mechanical pump, whereas at lower flow rates, the pressure control was conductance-controlled. The distance between the specimens and the exit of the ICP source was a few millimeters. For the test with the CCP source, the specimens were placed on the substrate holder, which was RF-biased. The substrate holder had a diameter of 76 mm. The gas mixture was injected directly into the chamber from a port at the top of the reactor. The experimental conditions are shown in Table 1.

Several diagnostics were used to investigate the influence of the plasma treatment on the sugar maple surface. First, substrate wettability with water was quantified before and after plasma treatment with a First Ten Ångstroms 200 Dynamic Contact Angle Analyzer. Because this equipment was not located close to the plasma chamber, the treated specimens were conditioned under partial nitrogen or inert atmosphere vacuum to facilitate transportation. Contact angles were measured immediately after droplet deposition using the sessile drop method. Droplet volume was 70 μL with 10 droplets of distilled water used for each test.

The chemical composition of selected substrates was investigated by X-ray Photoelectron Spectroscopy (XPS) using a PHI 5600-ci spectrometer (Physical Electronics, Eden Prairie, MN). A monochromatic aluminum X-ray source (1486.6 eV) at 300 W with a neutralizer was used to record the survey spectra while high-resolution spectra were obtained with a monochromatic magnesium X-ray source (1253.6 eV) at 300 W without charge neutralization. Detection was performed at 45° with respect to the surface.

After each treatment, a waterborne ultraviolet (UV)-curable polyurethane/polyacrylate resin was deposited on the substrate. The components and their formulations were supplied by an industrial partner. The coating was applied with a square applicator to a thickness of 60 μm and then dried in two steps. It was first dried at 60°C for 10 min for water evaporation and polyacrylate crosslinkage. In the second step, it was exposed to UV radiation to crosslink the polyurethane (Sunkist mercury lamp/ UVA = 53 J/m^2) and complete the curing process. Coating adhesion was measured in accordance with the ASTM D4541-02 test method (ASTM 2002). The technique involves gluing aluminum dollies using a 24-h curable epoxy resin. The load required to pull off the coatings was measured with a Positest AT adhesion tester. Three pull-off tests were performed for each treated and untreated specimen. Coating penetration was estimated by confocal Raman spectroscopy after each treatment. Each specimen had to be cut perpendicularly to the coated face, because wood thickness and opacity limited signal backscattering. Spectra were recorded at 22 ± 0.5°C and 20 ± 5% RH using a LABRAM 800HR Raman spectrometer (Horiba Jobin Yvon, Villeneuve d'Ascq, France) coupled to an Olympus BX 30 fixed-stage microscope equipped with a high-precision XY microscope table. The 514.5-nm line of an argon (Ar)+ laser (INNOVA 70C Series Ion Laser; Coherent, Santa Clara, CA) was chosen to irradiate the specimens. The laser beam was focused with a 100XL objective (0.9 NA; Olympus, Melville, NY) to a diameter of 1 m generating an intensity 5 mW on the specimen (green line). The confocal hole and the entrance slit of the monochromator were set at 400 and 100 m, respectively. Data were collected by a 25.4-mm open-electrode Peltier-cooled CCD detector (1024 × 256 pixels) (Andor Technologies, Belfast, UK).

RESULTS AND DISCUSSION

Coating Adhesion to Wood Before and After Treatment

Figures 4 – 6 indicate wood-coating adhesion improvement (in percentage) as a function of exposure time for RF inductive plasma treatments at 13.3, 133, and 665 Pa. A value of zero means

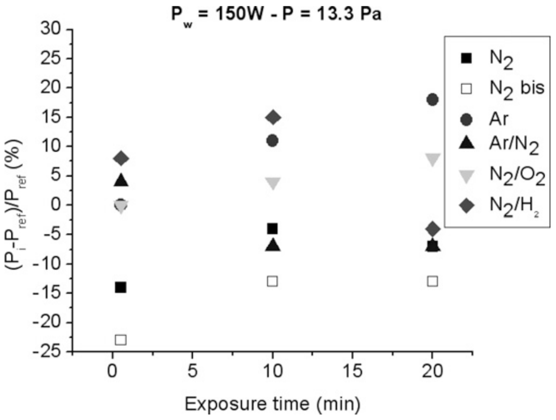

Figure 4. Adhesion improvement as a function of exposure time. Wood was treated by radiofrequency inductive plasma treatment at 13.3 Pa. Adhesion improvement was calculated with respect to a control specimen in each series. Standard deviation was up to 20%. P_w and P are the plasma power and pressure, respectively, in the closed vessel.

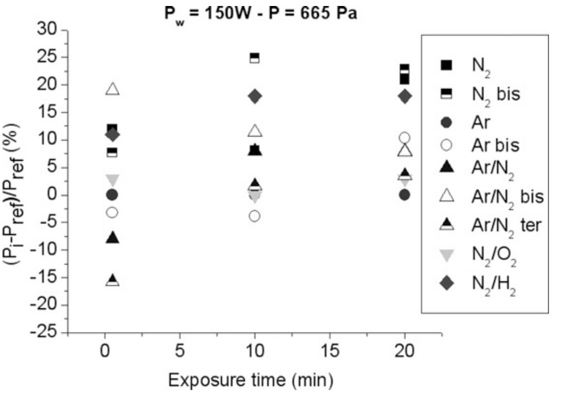

Figure 6. Adhesion improvement as a function of exposure time. Wood was treated by radiofrequency inductive plasma treatment at 665 Pa. Adhesion improvement was calculated with respect to a control specimen in each series. Standard deviation was up to 20%. P_w and P represent the amplificatory power and pressure, respectively, in the closed vessel.

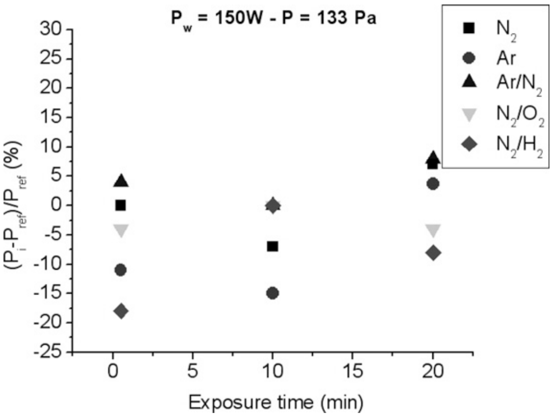

Figure 5. Adhesion improvement as a function of exposure time. Wood was treated by radiofrequency inductive plasma treatment at 133 Pa. Adhesion improvement was calculated with respect to a control specimen in each series. Standard deviation was up to 20%. P_w and P are the plasma power and pressure, respectively, in the closed vessel.

no improvement with respect to an untreated (control) surface. The results show that wood-coating adhesion either increased or decreased depending on gas type, exposure time, gas flow, and vacuum level.

In Fig 4, gas flow was 10 mL/min for a vacuum level of 13.3 Pa. Ar and N_2/H_2 treatments increased adhesion, whereas N_2 and N_2/O_2 treatments reduced it (by as much as 25% after 0.5 min). For Ar treatments, adhesion increased linearly with exposure time, whereas with N_2/H_2 mixtures, the adhesion values could be grouped according to two different time regions: 0.5 – 10 and 10 – 20 min. In the first region, adhesion decreased, whereas it rose for the latter. Wood extractives could have migrated with moisture to the wood surface during the vacuum process, which could have had a negative impact on adhesion (Anon 1999). The extractives could first cancel out any positive effects of the plasma treatment (first region). With time, however, most of the extractives could migrate and be degraded by the treatment (second region).

In Fig 5, pressures were 10 times greater than in the previous case, but gas flow was not modified. In most tests, wood-coating adhesion was severely reduced or slightly improved (less than 10% after 20 min). Different mechanisms may explain these differences. A change in pressure is likely to modify the number density, temperature, and the mean free path (which is pressure-dependent) of active particles. Fewer active particles therefore interact with the wood surface and this decreases the process efficiency with respect to the tests performed at 13.3 Pa.

In Fig 6, gas flow and vacuum level were equal at 0.5 L/min and 665 Pa, respectively. Because the number, density, and energy of impinging particles are directly linked to pressure, the plasma/wood collision rate was likely higher than previously. The results clearly show that adhesion improved significantly in most tests irrespective of exposure time. For example, Ar/N_2 treatment improved adhesion by 20% after only 0.5 min. The best results were obtained with N_2 treatments (about 25% after 10 min). Under these conditions, the ICP treatments were more successful. Several mechanisms could produce the adhesion improvements presented in Fig 6, including removal of extractives, modification of the coating wettability (Anon 1999), smoothing of the surface, and the creation of active sites, new chemical functions, etc.

Figure 7 presents the wood-coating adhesion improvement (in percentage) as a function of plasma exposure time for CCP treatments at 133 Pa. In this test series, the results indicated significant adhesion enhancement (up to 100% for Ar plasmas after 60 min as shown in Fig 7). The reasons for such drastic improvement can probably be attributed to the fact that in the CCP source, the substrate is located inside the plasma, whereas in the ICP source, the substrate is exposed to the postdischarge (expanding plasma; Figs 1 – 3). As a consequence, the substrate in the CCP source is not only exposed to a significant amount of radicals, electrons, and photons, but also to a considerable amount of positive ions. Because positive ions reaching the substrate in CCP can easily have energies > 200 eV (vs a few 10s of eV for ICP) (Lieberman and Lichtenberg 1994), ion bombardment is expected to strongly alter the wood surface properties. From these results, we conclude that ion bombardment plays a significant role in the evolution of coating adhesion after CCP treatment.

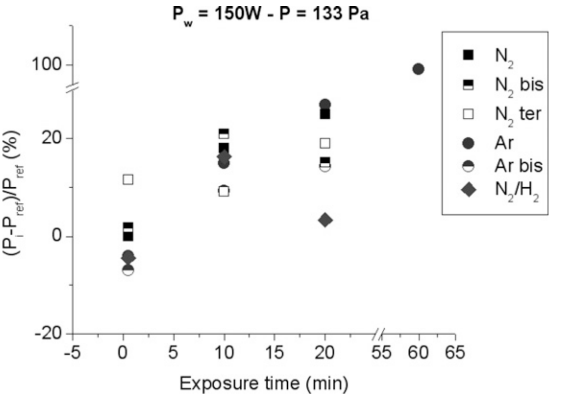

Figure 7. Adhesion improvement as a function of exposure time. Wood was treated by radiofrequency capacitive plasma treatment at 133 Pa. Adhesion improvement was calculated with respect to a control specimen in each series. Standard deviation was up to 20%.

X-ray Photoelectron Spectroscopy Characterization

The nature of wood surface atoms was characterized before and after treatment with results summarized in Table 2. These tests were conducted for only 20-min ICP and CCP treatments at 133 and 665 Pa. Before the CCP treatments used for XPS analysis, Teflon and silica thin-film depositions took place, leaving contaminants in the reactor chamber, which explains why the XPS detected fluor and silica traces. In addition, the capacitive and inductive reactor chambers communicated, which explains the weak contaminant concentrations observed in the N_2 and Ar/N_2 inductive plasma treatments. The results show that nitrogen atoms were grafted onto the wood surface after ICP treatments in N_2 and Ar/N_2 discharges, whereas little grafting was observed after CCP treatments. In the first case, nitrogen atoms created in the plasma source likely reacted with the wood surface to form new chemical functions. In the second case, the substrate was located directly in the plasma, and we expect limited nitrogen grafting because most grafted nitrogen atoms are likely to be sputtered away by the high-energy ion bombardment. Despite the considerable differences in the amount of grafted nitrogen (34% for ICP treatment vs 3.6% for CCP treatment), both treatments produced considerable increases in wood/coating adhesion (Figs 6 and 7). This indicates that both N grafting and ion bombardment can play key roles in

Table 2. *Atomic composition of wood surfaces obtained after different plasma treatments.*

		Inductive plasma (20-min exposure time)			Capacitive plasma (20-min exposure time)	
Chemical component (%)	Untreated Wood	Ar 665 Pa	N_2 665 Pa	Ar/N_2 665 Pa	Ar 133 Pa	N_2 133 Pa
C	75.4	78.8	44.5	54.1	75.4	76.7
O	23.8	21.2	20.3	16.1	19.5	16
N	0.9	0	34.0	29.4	0	3.6
Others[a]	0	0	0.2	0.4	5.1	4.3

[a]Traces of fluor and silica atoms.

coating/wood adhesion. Additional surface analyses will, however, be necessary to better understand the influence of grafted atoms on adhesion (eg amount, kind of new chemical bonds). The presence of primary amines (which facilitate covalent bonds between polyurethane resin and wood) was tested with this mind. Chemical derivations were performed with bromine. Subsequent XPS results showed that no primary amine was formed on the wood surface after ICP treatments. Therefore, the adhesion improvement was not from primary amines reacting during the polyurethane crosslinking process.

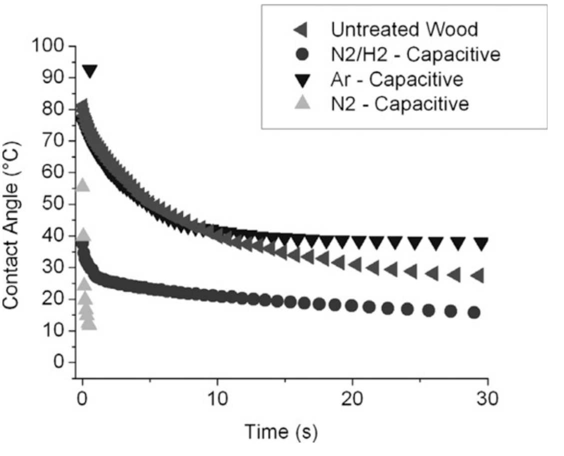

Figure 8. Evolution of mean contact angle as a function of time for different capacitively coupled plasma treatments.

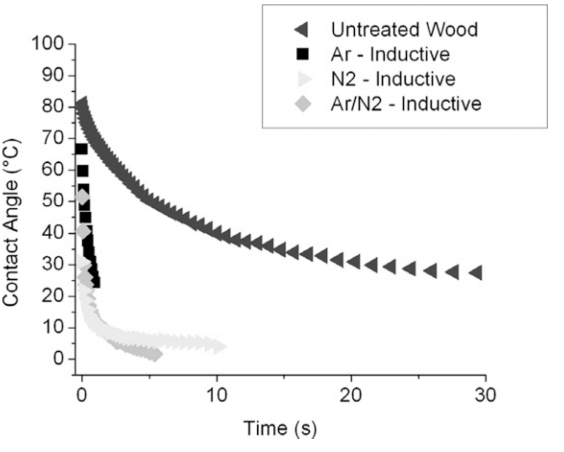

Figure 9. Evolution of mean contact angle as a function of time for different inductively coupled plasma treatments.

Measurement of Contact Angles Before and After Treatment

Wettability can be considered as a form of molecular adhesion between liquid and solid (Huntsberger 1963). Because the sphere of activity of attraction forces is similar to molecular distances, narrow contact between coating and substrate is a necessary condition for good adhesion (Sharpe and Schonhorn 1963; Huntsberger 1967; Collett 1972; Elbez and Bentz 1991). Indeed, the bond between coating and wood was maximized when the maximum wettability had been achieved. The plateau contact angle and gradient were much lower after treatment (except for Ar ICP), which indicates that wettability to water is increased (Figs 8 and 9). Figures 6 – 7 and 8 – 9 can be used to assess the relationship between adhesion and wettability to water, whereas Table 3 lists adhesion enhancement in relation to contact angle plateau values. When wettability to water is high (contact angle plateau is low), adhesion improvement is significant. The influence of plasma power for Ar CCP treatments is shown in Fig 10. The curves followed similar patterns for all treatments. For 150 and 180 W, they were superposed, whereas at 200 W, they were lower, but the curve shape was independent of plasma power. However, contact angles decreased with increasing plasma power, which suggests that plasma treatment efficiency could be enhanced with increasing power.

The best results were obtained in the presence of N_2 for inductive discharges. XPS results (Table 2) show that some of the N atoms were grafted onto the wood surface, forming new chemical functions that may explain in part the improvement in water wettability. As previously explained, the same mechanism cannot occur with argon. In addition, the energy of Ar ions may be relatively low (postdischarge) so that changes to the wood structure are small or nonexistent. That could also contribute to an explanation of the results obtained with Ar ICP treatment in terms of wettability to water and adhesion improvement. Further work is needed, however, to validate these hypotheses.

With wood specimens located within the plasma for capacitive discharges, atom grafting was very likely to be extremely limited on nonexistent, because the ion and/or electron bombardment would prevent or limit grafting. Ion energy was likely to be high enough to modify the wood structure, thus facilitating waterborne coating application. Here again, additional work needs to be performed to clarify the mechanisms involved in the improvements observed.

Finally, these results show relatively good correlation between water wettability and adhesion enhancement. The best adhesion results were obtained with surfaces having the lowest contact angles (best wettability to water). Castell et al (2007) observed similar results with polypropylene substrates covered with different UV-curable powder coatings.

Table 3. *Mean contact angles at plateau level and adhesion measurement before and after radiofrequency plasma treatments.*

Treatment (20 min)	Mean contact angle plateau (°)	Adhesion improvement (%)
Untreated wood	27	0
N_2 – 133 Pa – capacitive	7(*)	27
Ar – 133 Pa – capacitive	13	25
Ar/N_2 – Pa 665 – inductive	2	N/A
Ar – 665 Pa – inductive	38[a]	0
N_2 – 665 Pa – inductive	1	21
N_2/H_2 – 665 Pa – inductive	17	18

[a]Extrapolated values.
N/A = not available.

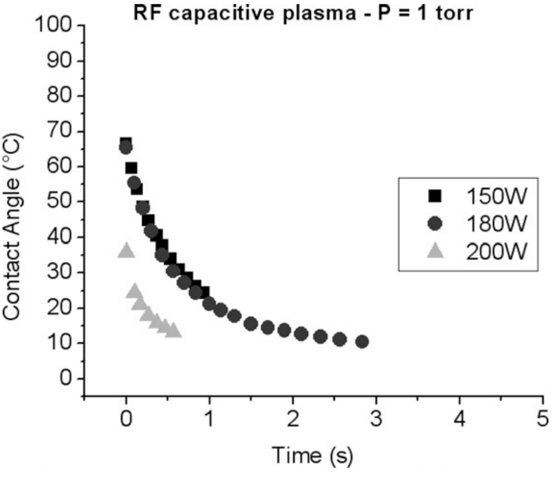

Figure 10. Influence of plasma power on mean contact angle evolution as a function of time.

Coating Penetration Characterization by Confocal Raman Spectroscopy

Coating penetration reinforces mechanical anchoring and extends the specific surface area available for contact between the coating and the wood cell walls (Lewis and Forrestal 1969; Vasishth et al 1974; Liptáková and Kúdela 1994; De Meijer and Militz 1998). Vasishth et al (1974) noted that coating penetration throughout wood vessels contributed to stronger bonds between coating and wood.

The wood–coating interfaces had been previously characterized before and after glow-plasma treatments (Blanchard et al 2009). It was established that plasma treatments tended to smooth surfaces and facilitated coating penetration. Wolkenhauer et al (2009) observed that plasma treatment could give the same results as sanding.

Table 4. *Coating penetration depth as a function of exposure time to radiofrequency plasma.*

Exposure time (min)	150 W	180 W	200 W
0.5	5 μm	15 μm	—
10	10 μm	25 μm	35 μm
20	10 μm	100 μm	80 μm
60	30 μm	—	—

To confirm the previous observations, the rate of coating penetration in wood was estimated for different exposure times and different Ar plasma energy by confocal Raman spectroscopy. The results reported in Table 4 show that coating penetration into wood was deeper as exposure time and plasma power increased. These results tend to confirm that wood coating adhesion would also have a mechanical component as suggested in the literature (Vasishth et al 1974).

CONCLUSIONS

This exploratory study demonstrated that plasma technology could be used to improve or reduce coating adhesion on wood surfaces depending on experimental conditions with three parameters seeming to control plasma efficiency, eg plasma type, exposure time, and pressure.

The type of plasma reactor and pressure were two very important parameters in improving adhesion. These techniques involved a partial vacuum, which greatly affected the plasma constituents (density, temperature, nature). Pressure control appeared to be particularly important. ICP treatments mainly deal with postdischarge phenomena (low-energy ions); hence, wood structure modifications were limited. This technique permitted grafting new atoms at the near surface, which could improve compatibility with the coating. CCP treatments generate high-energy ions that may have modified the wood structure, affecting subsequent wood-coating interactions.

For ICP treatments at low pressures and gas flows, the best improvements were obtained with Ar and N_2/H_2. At high pressures and gas flows, most of the treatments were effective, and adhesion was increased by 25% after N_2 exposure. Under these conditions, the collision rate between plasma constituents and wood was higher and could explain, in part, why the treatment was more efficient than previous ones. Additional work will be needed to confirm this hypothesis. In several tests, it was also observed that adhesion enhancement peaked at 10 min before decreasing. This may be from wood extractives, but no results are available to confirm this.

For CCP treatments, the wood samples were positioned within the plasma and in contact with high-energy plasma constituents. In this case, only pressure controlled the collision rate because the number density increased with pressure. All the tests showed adhesion enhancement; wood-coating adhesion had even doubled in strength for Ar.

The XPS results showed that nitrogen atoms had been grafted onto wood surfaces after ICP treatments, whereas no grafting occurred in CCP treatments.

Adhesion improvement was partially linked to surface energy modifications and explained by enhancement of chemical compatibility. In addition, confocal Raman spectroscopy characterization showed that it could also be related to coating penetration depth. Adhesion enhancement was the result of both chemical and mechanical factors, but this study did not discriminate between these mechanisms.

These exploratory results need confirming through additional testing. Similar experiments could be performed with atmospheric plasma treatments, which would be closer to industrial application. Plasmas should be characterized (energy, composition) to provide a better understanding of the modification mechanisms involved. In addition, magnetron sputtering of inorganic and organic depositions also seem a promising technique for wood, and they deserve a feasibility assessment.

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