Wood Surface Modification by Atmospheric-Pressure Plasma and Effect on Waterborne Coating Adhesion

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In this paper the effect of an atmospheric-pressure plasma treatment on the surface properties of sugar maple (*Acer saccharum* March.) and black spruce (*Picea mariana* (Mill.)) is analyzed by contact angle measurements and water-based coating pull-off tests. The plasma gases used are Ar, N_2, CO_2, and air. It is found that the wettability with water and the coating adhesion of maple and spruce can be highly influenced by the nature of the plasma gas and the plasma treatment time. For example, in the case of sugar maple, coating adhesion increases by 66% after 1.5 s of exposure to argon plasma. Repetition of contact angle measurements one and two weeks after the plasma treatment further revealed that the plasma-induced modification is not permanent. Improvement of the wettability and adhesion were also obtained with the simpler and cheaper air plasmas, a very promising result for the development of advanced plasma reactors at atmospheric pressure specially designed for the wood industry.

Keywords: Atmospheric-pressure plasmas; Sugar maple; Black spruce; Coating adhesion; Water contact angle; Pull-off test

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INTRODUCTION

Plasma treatment has been used to modify the surface properties of many polymers, including those with complex chemistry and structure such as textiles or wood. The treatment of wood by plasma is a relatively new technology, and data reported from scientific papers are still rare. Depending on the plasma reactor's design, the plasma gases used, and plasma treatment parameters, specific properties can be improved, such as wood wettability, water repellence, and coating adhesion. In terms of final properties of the treated wood species, the plasma gas is probably one of the most important parameters to be considered as it determines the nature of the active species (radicals, metastables, ions, and photons) interacting with the wood substrates. Inorganic plasma gases such as nitrogen, oxygen, carbon dioxide, air, argon, and others are generally used to alter surface properties such as wettability (Blanchard *et al.* 2009; Podgorski *et al.* 2002; Evans *et al.* 2007; Rehn *et al.* 2003; Denes *et al.* 2005), surface energy, and coating adhesion (Blanchard *et al.* 2009; Evans *et al.* 2007; Rehn *et al.* 2003). Plasmas generated in such inorganic gases can alter the near-surface composition of the wood surface by ions or atoms implementation, can produce etching of low molecular weight species, or can also modify the chemical structure following ultraviolet irradiation. On the other hand, organic gases such as ethane, hexamethyldisiloxane (HMDSO), polymethyldisiloxane (PMDSO), and fluorine compounds are often used together with inorganic carrier gases for the growth of hydrophobic functional coatings on wood surfaces by Plasma-Enhanced Chemical Vapour Deposition (PECVD) (Podgorski *et al.* 2001; Zanini *et al.* 2008; Kim *et al.* 2009; Manolache *et al.* 2008; Levasseur *et al.* 2012).

At this time several wood species treated by plasmas have been investigated, from common northern species such as maple (Blanchard *et al.* 2009; Busnel *et al.* 2010), pine (Podgorski *et al.* 2001), spruce, poplar, or chestnut (Zanini *et al.* 2008) to more exotic species like coconut palm or eucalyptus (Blantocas *et al.* 2007). Besides wood species and the nature of the plasma gas, another variable to be considered is wood conditioning and finishing. Prior to plasma treatment, wood samples can be specifically conditioned and aged, sanded to a specific degree, or not sanded at all. It is important to account for sample finishing, since sanding could lead to the removal of wood extractives from the surface. The influence of wood samples' preparation (sanded *vs*. not sanded) was evaluated by Wolkenhauer *et al.* (2009).

Many other plasma parameters and their role on wood modification dynamics have been investigated, including injected power and excitation frequency (Podgorski *et al.* 2000, 2001), operating pressure and gas flow rate (Blanchard *et al.* 2009; Podgorski *et al.* 2000, 2001), plasma exposure time (Podgorski *et al.* 2001), and cut orientation with respect to wood grain direction (Evans *et al.* 2007). Given the highly complex composition of wood, which is composed of cellulose, hemicelluloses, and lignin, a number of mechanisms can arise following plasma exposure.

The effect of a specific plasma treatment is often characterized by contact angle analysis (Blanchard *et al.* 2009; Podgorski *et al.* 2000, 2001; Zanini *et al.* 2008; Evans *et al.* 2007; Rehn *et al.* 2003; Podgorski *et al.* 2001b; Busnel *et al.* 2010; Asandulesa *et al.* 2010) and adhesion tests (Blanchard *et al.* 2009; Evans *et al.* 2007; Rehn *et al.* 2003; Nussbaum 1999; Kim *et al.* 2009; Busnel *et al.* 2010). Other analyses include water droplet absorption tests (Blantocas *et al.* 2007; Manolache *et al.* 2008; Wolkenhauer *et al.* 2007), permeability tests (Chen *et al.* 1990), X-ray photoelectron spectroscopy (XPS) (Avramidis *et al.* 2009; Busnel *et al.* 2010), and atomic force microscopy (Mahlberg *et al.* 1999). For example, using XPS, Klarhöfer *et al.* (2010) showed that Dielectric Barrier Discharges (DBD) at atmospheric pressure operated in oxygen-oxidized lignin while those operated in argon reduced both lignin and cellulose.

Plasma reactors could roughly be classified as either vacuum (low-pressure) reactors or atmospheric-pressure reactors. The differences between these types of plasmas were explained by a number of authors (Denes *et al*. 2005). Both types of plasma reactors have been used to alter the near-surface properties of wood. Low-pressure systems operate in close enclosures in which the pressure (order of a few mTorr) has to be created and maintained prior to and during treatment. This usually involves expensive installations, time consuming processes, and high operating costs. Besides, since the space of the enclosure is limited, only relatively small quantities of wood batches can be treated at once. After that, the reactor has to be turned off and then opened in order to replace the samples, operations which dramatically affect the productivity. On the other hand, atmospheric-pressure plasma reactors operate in open environments, where samples can be inserted or removed anytime into the plasma. From a practical point of view, such reactors present the advantage that they are highly productive and relatively inexpensive to operate. Thus, they could be inserted in a continuous batch production line at a very low cost, allowing the development of a new generation of wood products. The authors of this study consider that, even if vacuum plasma reactors are very useful for the treatment of some very high-added value products, such as medical prosthesis or microchips, atmospheric-pressure plasma reactors are the most suitable approach to treat low-added value materials such as wood and still create competitive products.

Among reports on plasma treatment of wood surfaces, reviews of those realized under atmospheric pressure conditions are particularly scarce. Rehn *et al.* (2003) used an atmospheric pressure DBD to improve fracture strength and uptake time of water droplets on wood surfaces. The plasma gas was atmospheric air, and the wood species investigated were robinia, spruce, beech, teak, and oak. Another research involving a DBD operated in air was conducted by Wolkenhauer *et al.* (2007). Samples consisted of wood plastic composites, particle boards, and fibre boards. The authors found that the water contact angle on these surfaces decreases proportionally with plasma exposure time on all type of samples. Also, delamination tests have proved that adhesion of coatings increased after plasma treatment. Busnel *et al.* (2010) examined the effect of DBD treatments on sugar maple and black spruce. The plasma gases used were Ar, O_2, N_2, CO_2, and some of their mixtures. Depending on the gas composition, contact angle and coating adhesion for both species were significantly modified. Based on the set of data reported, a correlation was proposed between the plasma-induced improvement in the hydrophobic character of wood samples and the corresponding improvement in wood-coating adhesion. The hydrophobic behaviour of wood has also been investigated by Asanduleasa *et al*. (2010). Studied wood species were oak and beech, the plasma gas was He, and the treatments were also performed with a DBD tool. The wettability of wood improved following the treatment, as shown by water contact angle analysis. Avramidis *et al.* (2009) also investigated how atmospheric-pressure plasma alters the hydrophilic characteristics of beech, oak, spruce, and pine.

Low-pressure or close to vacuum plasma treatments include the works of Podgorski *et al.* (2001), Evans *et al.* (2007), and Chen *et al.* (1990). Podgorski *et al.* (2001) investigated the influence of fir plasma exposure time, plasma power, sample to plasma distance, and aging time after treatment. Another study which monitored the effect of plasma energy and aging time after plasma treatment of several eucalyptus species was conducted by Evans *et al.* (2007). Chen *et al.* (1990) investigated the effect of plasma-induced permeability increase on white fir and Douglas fir.

According to the review presented above, it is obvious that plasma treatment at either low or atmospheric pressure is an effective way to alter wood wettability and coating adhesion. The effect of plasma exposure on wood depends on many parameters such as time of exposure, plasma gas, elapsed time after treatment, wood species, and others. While the influence of some of these parameters was evaluated for low-pressure plasma, there are little or no data available on this subject under atmospheric pressure conditions. It is the aim of the present study to contribute to the understanding of wood plasma treatment at atmospheric pressure by investigating the effect of several plasma factors on maple and spruce wood. This work is somehow complementary to the work of

Busnel *et al.* (2010), because the same wood species and plasmas are used, but additional parameters such as plasma exposure time and elapsed time after treatment are investigated. Also, wood surface preparation is different (wood samples were not sanded before plasma treatment).

MATERIALS AND METHODS

Black spruce and sugar maple wood blocks (3.5" x 3.5" x ½") were cut tangentially-longitudinally with respect to the wood grain direction. Four hundred specimens were made for each species. After cutting, all the wood samples were randomized and conditioned at 20 °C and 50% RH for two weeks before proceeding to the various plasma treatments. The plasma apparatus is shown in Fig. 1. It consists of a plane-to-plane dielectric barrier discharge opening to ambient air (Plasmionique, Quebec, Canada). In this system, the discharge is sustained between two metallic electrodes (285 cm^2) covered with two 1.5 mm thick quartz discharge plates.

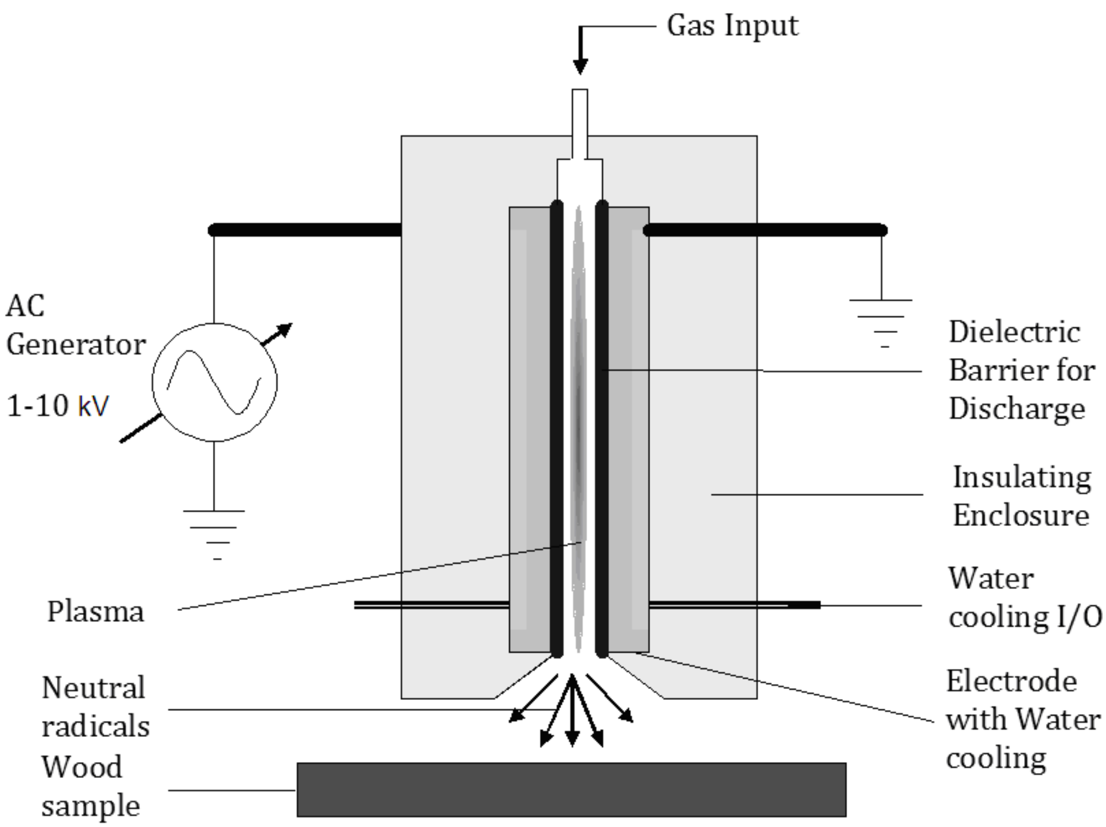


Fig. 1. Schematics of the flowing DBD apparatus used in this work - wood samples are moved on a conveyer along the plasma jet

The discharge gap was set to 1.4 mm. The system also includes a gas inlet line located near the end of the electrodes and composed of several independent channels allowing the creation of plasmas with various gases (here N_2, O_2, CO_2, and Ar). The electrical circuit used to power the plasma consists of several elements. The first one is a function generator which provides the reference signal. This signal is then applied to the input of a linear power amplifier connected in series with the primary of a step-up transformer. The discharge cell is connected to the transformer's secondary in series with a 50 Ω resistor to measure the potential drop and thus the current using Ohm's law. For all gases investigated, the electrical stimulation was sinusoidal with a frequency of 9 kHz. The peak-to-peak voltage and the resulting power absorbed or dissipated in the discharge for each gas investigated are given in Table 1. After subtraction of the capacitive current from the measured current (to obtain the discharge current) and of the voltage drop across the quartz dielectric plates from the applied voltage (to obtain the voltage applied to the gas), the power absorbed or dissipated in the plasma was obtained using the approach described by Naudé *et al.* (2005).

One of the specificities of this reactor with respect to other atmospheric-pressure DBD units is that high gas flows (50 L/min) are blown between the two electrodes where the plasma is sustained such that wood samples are exposed to the flowing afterglow or plasma jet. Depending on the wood inherent roughness, the plasma-to-surface distance was in the 3-5 mm range. No modification of wood surfaces was observed for small gas flows (below about 1 L/min), which confirms that the observed change in wettability and adhesion reported below results from exposure to a flowing afterglow or plasma jet and not to a coplanar surface discharge. In order to control the plasma exposure time, wood samples are placed on a treadmill on which the speed can be adjusted. In this work, a constant speed of 1.5 cm/s was used, which leads to an estimate treatment time per pass of 0.1 s. For most experiments, multi-pass treatments were performed, allowing detailed investigations of the plasma exposure time (between 0.1 and 1.5 s) on the wood surface modification dynamics. For all experimental conditions investigated, 10 to 15 repetitions were performed to account for the random variation of properties in wood samples.

Table 1. Electrical Characteristics of the Discharge Used for Wood Treatments

Nature of the gas	Applied peak-to-peak voltage (kV)	RMS discharge current (mA)	Power absorbed or dissipated in the discharge (W)
Ar	3.5	8.4	4.0
N_2	10	4.4	7.1
CO_2	10	13	32
Dry air	10	18	37

Following each plasma treatment, two types of analysis were performed: contact angle measurements and coating pull-off tests. The wettability with water of plasma-modified wood samples was analyzed the same day as the plasma treatment. The goniometer used was First Ten Angstroms 200 Dynamic Contact Angle Analyser. Contact angle values were recorded immediately after droplet deposition on the wood sample surface. Since the water droplets spread asymmetrically with respect to the wood grain angle, the wood samples were placed such that the axis of the camera was always perpendicular to the wood grain direction, as shown in Fig. 2. Mean contact angles were then determined by averaging over several points on a sample, and this was repeated for 10 wood samples.

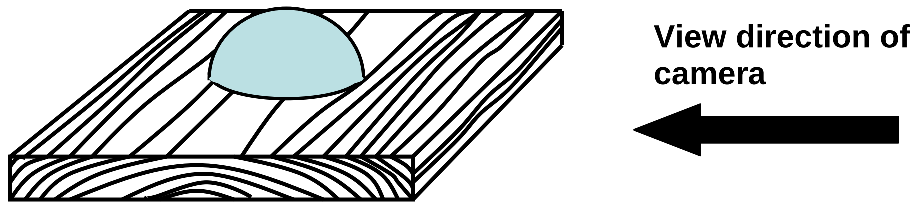


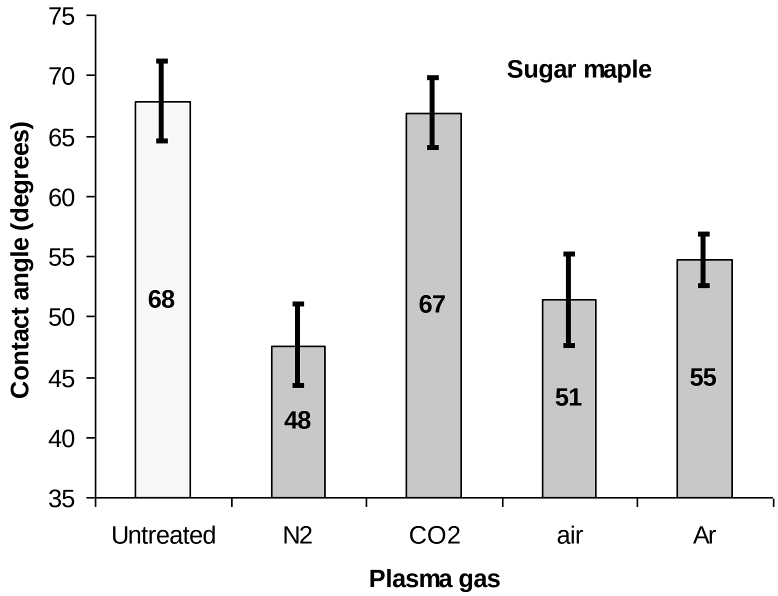
Fig. 2. View direction of camera with respect to the wood grain

To examine the long-term stability of the plasma-modified wood samples following natural aging, contact angle measurements on uncoated samples were repeated after one and two weeks. During this natural aging time, the samples were kept in the conditioning chamber at 20 °C and 50% RH.

For the pull-off tests, 15 repetitions were performed for each set of plasma conditions. Immediately after treatment, wood samples were covered with a waterborne UV-curable polyurethane/polyacrylate coating, the same that was used by Busnel *et al.* (2009). The coating was applied with a square applicator, allowing a thickness of 0.1 mm. The coating was then allowed to dry for 10 min at 60 °C. Then, a second layer was applied in the same way. After that, all the specimens covered with resin underwent UV curing (Sunkist mercury lamp/UVA = 53 J/m^2). Once the resin cured, the pull-off test was performed accordingly with ASTM D4541-02 standards. In this framework, metal dollies were glued on the coating with an epoxy resin. After 24 h or more, the coating around the dollies was sawn to separate it from the rest of the resin film, and the dollies were then pulled off with a hand machine made by Positest. The maximum load, which occurs at the time of the delamination of the UV coating from the wood surface, was recorded. When important wood failure had been noticed, the measurement was eliminated from the statistics.

RESULTS AND DISCUSSION

In Fig. 3, the hydroscopic behaviour of sugar maple and black spruce wood samples following their exposure to various plasma gases are compared for a total exposure time of 1.5 s. One can see that nitrogen, argon, and air-based plasma jets dramatically decreased the mean contact angle of water for sugar maple. On the other hand, carbon dioxide did not significantly alter the wettability of maple (Fig. 3a). For black spruce, none of the four plasma gases investigated in this work produced a significant modification of the mean contact angle of water, even for maximum treatment durations (Fig. 3b). As discussed by Avramidis *et al.* (2012), part of the increase in the wood surface energy resulting in lower water contact angles following plasma exposure can be attributed to the removal of wood hydrophobic extractives. Given the highly porous nature of black spruce, one can readily expect a more rapid migration of wood extractives contained in the cell lumen towards the topmost surface. For such softwood species, even if some removal occurs in the plasma treatment, outdiffusion of extractives is so rapid that the wood rapidly returns to its pristine state and thus to the water contact angle before plasma exposure. Despite such rapid migration and the lack of modification of the contact angle, improvement of the adhesion may still be observed. Indeed, in contrast to the simple removal of wood extractives following sanding, the wood surface can also undergo significant chemical, structural, or morphological changes in the presence of an active species in the plasma jet, and these modifications are all likely to play a significant role on the evolution of the wood-coating adhesion properties.



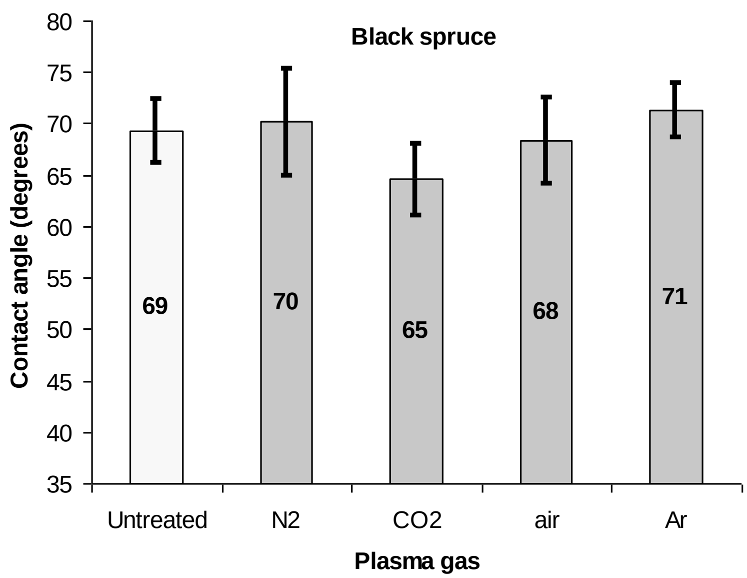


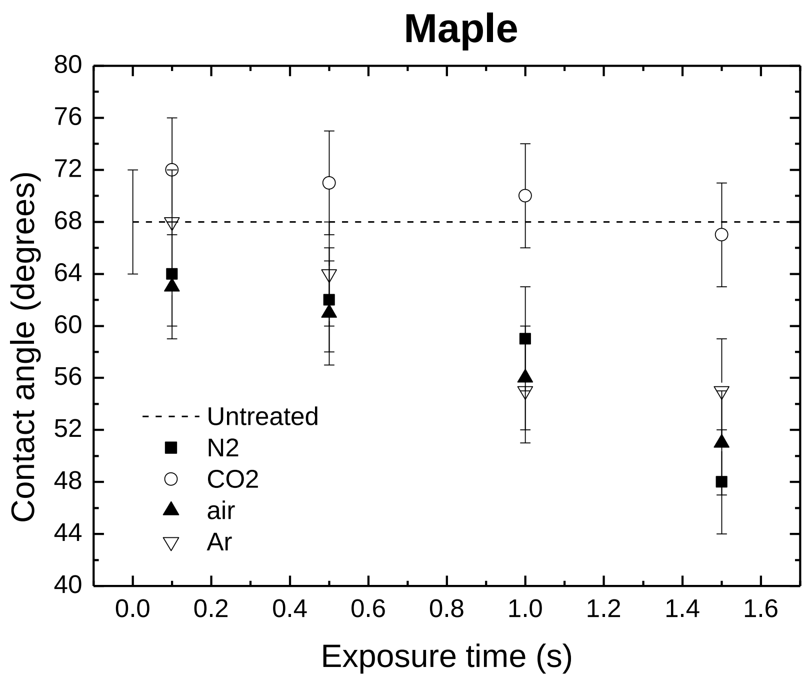
Fig. 3. Influence of plasma gas on wettability with water of sugar maple (top) and black spruce (bottom) for a plasma exposure time of 1.5 s

It is notable that air-based plasmas can decrease the contact angle of water of sugar maple from 68° to 51° (25%) after only 1.5 s of treatment, and this is very encouraging from a practical point of view. Indeed, such results represent a strong step towards the development of atmospheric-pressure plasma reactors running with simple and cheap feed gases, which makes them more easily transferable to the wood industry. Such a reactor would operate using the ambient filtered air instead of the more expensive compressed gases. Since the formation of plasma jets such as those used in the present study consumes very large amount of gases (flow rates > 50 L/min), air-based plasmas would be very economical and thus more suitable for wood producers.

The influence of plasma exposure time on mean contact angle is represented in Fig. 4. For sugar maple, experimental results demonstrate that the water mean contact angle loss is proportional to plasma exposure time, and this relation is approximately linear. The exception is carbon dioxide, which fails to significantly modify the wettability of wood irrespective of the exposure time (Fig. 4a). In the case of black spruce, it was impossible to establish a relation between exposure time and mean contact angle. In Fig. 5b, one can see that the values of contact angles related to exposure times are randomly distributed above or under the control value (dotted line), so there is no effect on spruce wettability or at least nothing that could be detected by contact angle analysis.

The stability of each plasma treatment for both wood species is shown in Fig. 5. As seen by contact angle analysis, the effect of plasma active species is not permanent, no matter the plasma gas used. The repetition of contact angle measurements after one and two weeks following plasma treatment demonstrates that mean contact angle values increase progressively during the period elapsed after treatment. In some cases, for low plasma exposure times, the initial contact angle value of untreated specimens is reached after only one week. This is the case for argon and air treatments on sugar maple. For longer exposures, such as 1 and 1.5 s, the initial plasma effects are strong enough to last more than two weeks on sugar maple, but the trend is ascendant for all exposure times (Fig. 5 a to d). It is likely that the mean contact angle values of high exposure durations would eventually reach the initial values after a period longer than two weeks. For black spruce the effect of elapsed time after plasma treatment is less obvious, probably because there was not any improvement in the first place (Fig. 5 e to f), but one can still see a tendency of increasing contact angle values for nitrogen (all exposure times) and argon (all but 1.5 s exposure time).

This instability in the water contact angle may be due to the nature of the chemical changes in the wood surface. When wood is cut, contact angles with water vary with time due to oxidation and migration of extractives to the surface (Nussbaum 1999). So, the same is bound to happen after plasma treatment. Sakata *et al*. (1993), following corona treatment of the wood surface, suggested the changes in the surface energy of wood following treatment were due to the oxidation of hydrophobic extractives and the wood substance itself, as cellulose and lignin were not affected. Since this oxidised layer is generally quite thin, unreacted extractives in the unaffected wood may diffuse back into the surface and eventually bring the surface energy back to its original values after a few weeks. Avramidis *et al*. (2012) also interpreted changes in the surface energy of the plasma-treated wood in terms of oxidation of extractives.



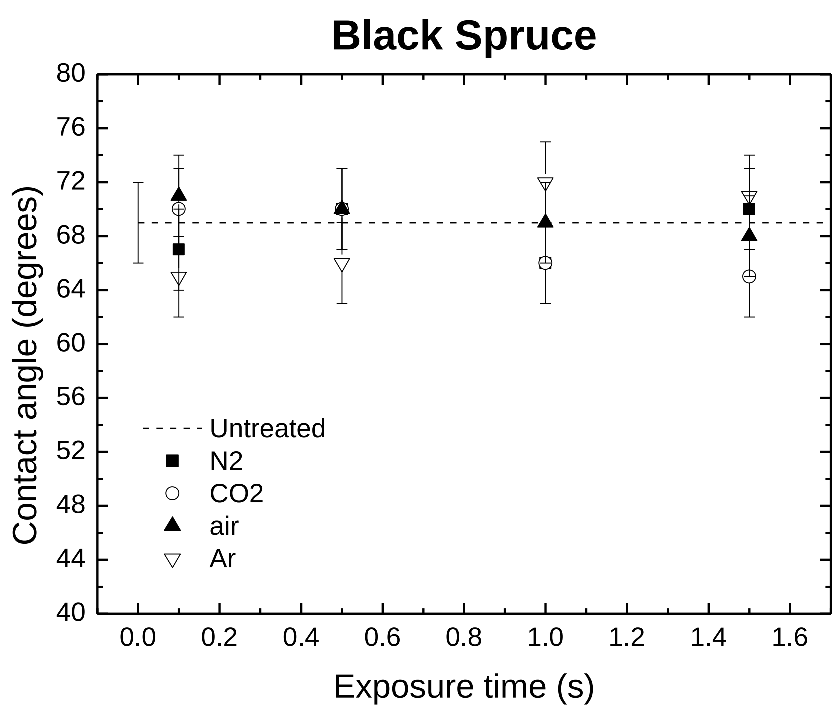


Fig. 4. Mean contact angle of water as a function of plasma exposure time for sugar maple (top) and black spruce (bottom). Dotted lines represent values for control samples.

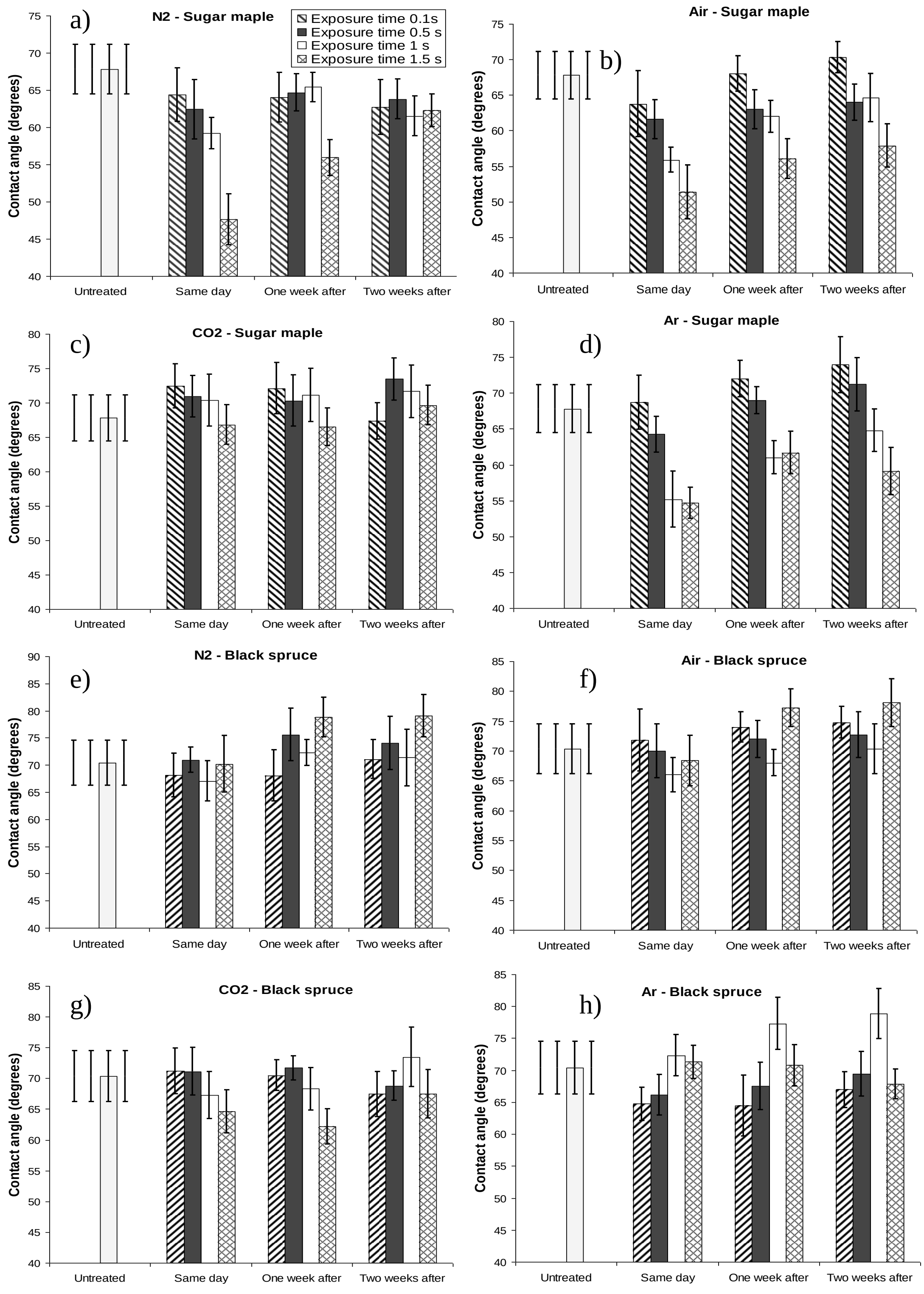


Fig. 5. Stability of plasma treatment of sugar maple (a to d) and black spruce (e to h)

Regarding the pull-off tests, statistical analysis revealed that plasma treatment significantly increased coating adhesion on black spruce for all plasma gases but carbon dioxide (Fig. 6). This is true for almost all exposure times for black spruce, the only exception being the air plasma treatment of 0.5 s. Carbon dioxide failed to improve the coating adhesion on spruce, no matter the exposure range. For other gases, the improvement was dramatic in some cases, especially for high exposure times (66% increase in coating adhesion after 1.5 s of spruce exposure to argon plasma). The reason why plasma improves coating adhesion of a waterborne resin but fails to improve wettability of black spruce is still unclear. A possible explanation could be that, since pull-off tests were performed immediately after plasma treatment and contact angle analysis a few hours later, plasma effect on spruce would lead to unstable surface species (such as free radicals) which deactivate during this time. Creation of free radical species was deemed at the origin of enhancement of adhesion in plasma-treated polyethylene by several authors (Bahners and Gutman 2011; Svorcik *et al*. 2006; Tahara *et al.* 2003).

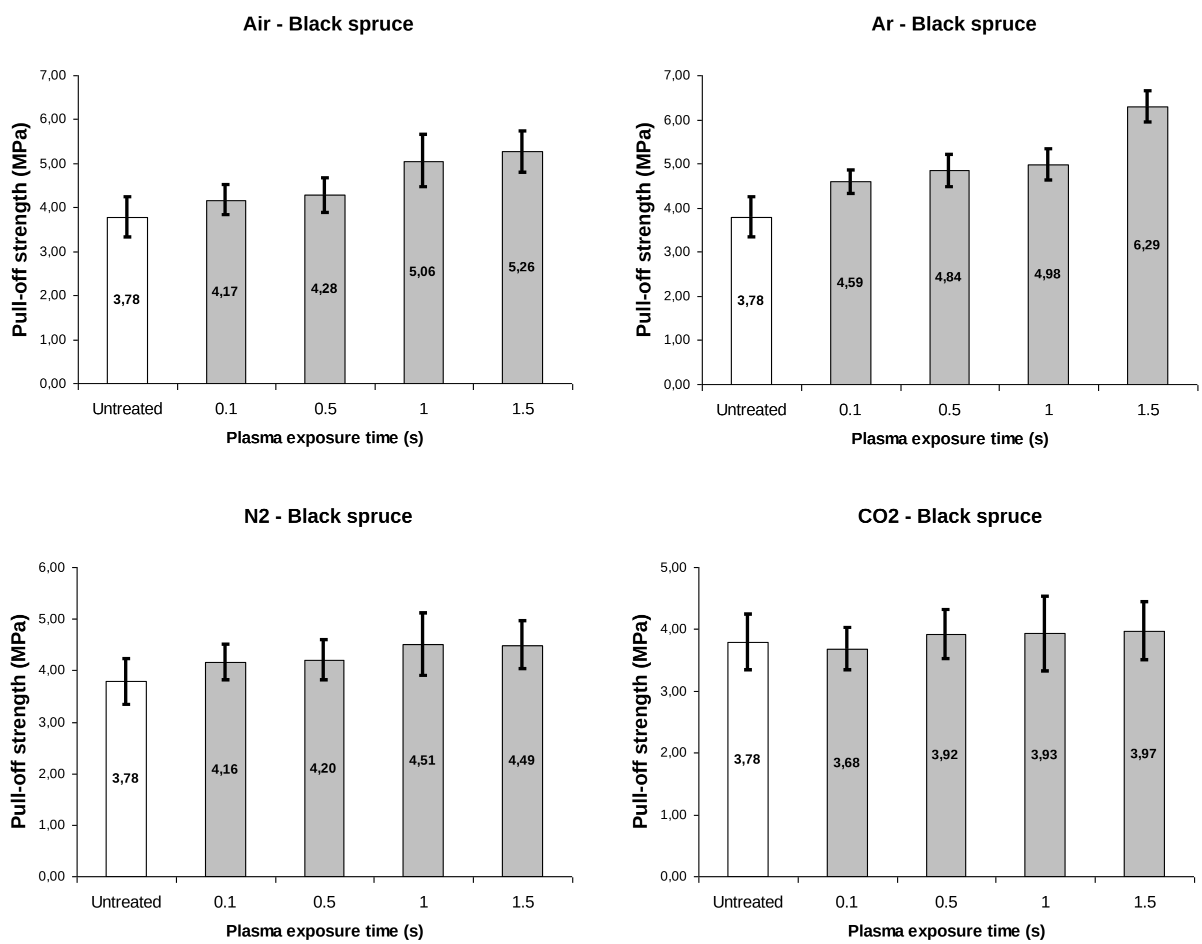


Fig. 6. Black spruce pull-off strength as a function of plasma exposure time

As well, pull-off tests of sugar maple revealed that coating adhesion can be altered by most of the atmospheric-pressure plasmas examined, the only exception being the carbon dioxide plasma, which did not produce any significant change for all exposure times investigated (Fig. 7). Carbon dioxide plasma usually increases the polymer surface acidity (Aouinti *et al.* 2003), which could be the reason why both wettability and coating adhesion were not improved by this plasma gas. In addition, despite the higher power absorbed or dissipated in the CO_2 discharge with respect to other gases (see Table 1), the overall plasma emission turned out to be much lower (not shown), which is an indicator of a much lower number density of charged particles. This obviously reduces the number of active species being formed in the discharge and their availability for wood surface modification. On the other hand, nitrogen and argon plasmas significantly increased the adhesion only for 1 and 1.5 s of plasma treatment. Changes induced by air plasma are statistically significant starting with the lowest exposure time of 0.1 s. The most important modification of coating adhesion is obtained for 1.5 s of treatment by argon plasma (63% increase) and air plasma (51% increase). As detailed above, the performance of the flowing afterglow of the atmospheric-pressure dielectric barrier discharges operated with dry air on the coating adhesion of both wood species is very promising, since such treatments could by very cost-effective for the wood industry.

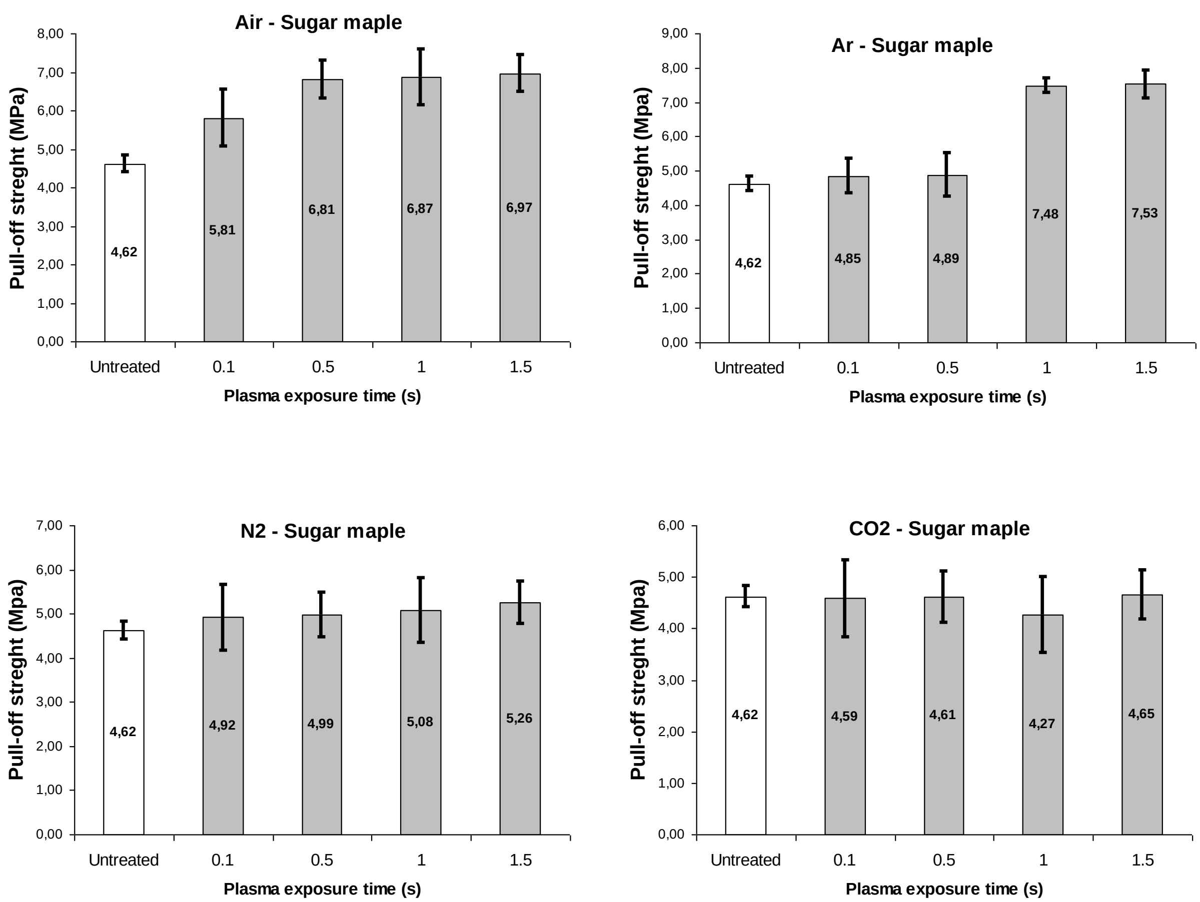


Fig. 7. Sugar maple pull-off strength as a function of plasma exposure time

Further analysis of the results presented in Figs. 3 to 7 indicates that in contrast with common beliefs, improved wettability with water does not necessarily imply improved wood-coating adhesion. This is because, in addition to the surface energy (dispersive adhesion), a number of other phenomena drives the adhesion dynamics, including surface roughness (mechanical adhesion), formation of chemical bonds, (chemical adhesion), presence of electrical discharges (electrostatic adhesion), and solubility of the coating (diffusive adhesion). Given the diversity of active species (ions, radicals, electrons, metastables, and photons) present in the flowing afterglow of Ar, N_2, CO_2, and dry air discharges at atmospheric pressure, such treatments can alter all of these contributions through either surface texturing, polymer cross-linking, surface charges, surface oxidation, surface etching, and bond breaking.

CONCLUSIONS

Atmospheric-pressure plasma treatment of sugar maple (*Acer saccharum* March.) and black spruce (*Picea mariana* (Mill.)) resulted in increased coating adhesion strength of both wood species. This is true for all the plasma gases selected for this study with the exception of carbon dioxide. The precise reason why carbon dioxide failed to increase coating adhesion remains unclear, although the low discharge currents seem to point towards a low number density of active species over the range of experimental conditions investigated. The wettability of maple has also been increased by plasma treatment, but this is not the case for spruce, no matter the time of the plasma treatment, which is possibly due to the creation of unstable reactive species on the wood surface. The study of influence of plasma exposure time on maple wettability revealed that the loss of contact angle is directly proportional to the time of treatment. The repetition of contact angle measurements demonstrated that the effect of atmospheric-pressure plasma treatment on wood wettability is not permanent: after treatment, the gain in wettability begins to fall and in some cases reaches the control value within two weeks. Consequently, one should take advantage of plasma-surface modification as soon as possible after treatment. From a practical point of view, the effect of air plasma on wood is highly important, since it possibly leads the way to the development of industrial air plasma reactors at atmospheric pressure for the wood industry.

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