The Prolonged Effect of Plasma Treatment upon Dielectric Properties of Wood-Based Composites

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Abstract

A more profitable application of wood-based composites would certainly include the possibility of their use as a dielectric material in the form of electrical insulation. The profitability and the price difference between materials normally used for these insulating purposes and those based on wood would easily justify the interest to study the dielectric properties of such composites. There is another reason why an extension in use of these composites might become important. It originates from the general shortage of high-quality wood. Thus, the related properties of hardboard and medium-density fiberboard were investigated as well as plywood and particleboard. The modifications included treatments by direct current plasma glow discharge, vacuum, conditioning of samples at room temperature for prolonged periods of time, and their combinations.

A relationship was discovered between the applied treatment procedures and the investigated dielectric parameters. The comparison between the treatment effects indicates those most convenient for the purpose of production of electrical insulating and also assists in differentiating which treatment processes lead to improved properties of the investigated wood-based composites.

Different methods for modification of wood, such as acetylation (Rowell 2006), microwave treatment (Torgovnikov and Vinden 2010), ozonation (Korai et al. 2001), and others were developed to improve the properties of wood-based products. Dimensional stabilization, decrease in water sorption (hydrophobicity), or improvements in mechanical properties are the common objectives achieved by the aforementioned modifications. Physical properties of the solid wood, such as dielectric, strongly depend upon the moisture content (Kollmann and Côté 1968).

Different plasma types are widely used for surface modifications, such as activation, inactivation, etching, cleaning, or sterilization. Some authors (Podgorski et al. 2000) suggest plasma treatment for activation of a wood surface by oxygen and nitrogen direct current (DC) plasma will lead to improved bonding with adhesives. Their conclusions were determined based on decreasing the contact angle for the different levels of used power, durations, and electrode distances. No records of the treatment temperature were presented. According to Bente et al. (2004), atmospheric pressure alternating current (AC) dielectric barrier discharge is a powerful tool to increase hydrophobicity of the wood surface. Other studies indicate the ability of plasmas to make wood hydrophobic (Matsui et al. 1992, Denes et al. 1999, Avramidis et al. 2009). They used a nitrogen atmosphere (98%) and measured the drop absorption time, contact angle, and surface tension measurements. The duration of exposure of the samples to plasma was short (1 to 5 s), and their applied frequency was 17 kH. Lommatzsch et al. (2007) used atmospheric pressure plasma jet treatment on a polyethylene surface. They concluded that surface activation correlated with a decreased contact angle and an increased surface tension and lap shear strength. They also concluded that roughness and activity of the surface are strongly correlated.

For a better understanding of plasma–surface interactions, it is very important to emphasize the phenomenon of etching. Plasma–surface interaction is widely used in semiconductor and integrated circuits fabrication when a selected surface area has to be removed (Lieberman and Lichtenberg 2005). For these sophisticated technologies some polymer surfaces are of the great importance, such as polysilicon. There is also evidence of the same phenomenon on the wood surface exposed to glow discharge plasma treatment (Jamali and Evans 2010). Etching occurs when electrons and ions (constituents of plasma) have sufficient

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energy to break chemical bonds of the wood surface structure. Some aromatic components of wood, such as lignin, are more resistant than the polysaccharides cellulose and hemicelluloses (Pederson 1982, Egitto et al. 1990).

Dielectric properties of wood-based composites depend on numerous parameters. These include the original wood species, the moisture content in the incorporated wood material, and the present state of the adhesive. The main purpose of this research was to investigate the possible applications of wood-based composites as electrical insulating materials and the treatments improving such possibilities.

Thus, wood-based composites such as medium-density fiberboard (MDF), fiberboard, particleboard, and plywood were exposed to the effects of vacuum (only) and DC glow discharge plasma. This research was supplemented with the effects of their conditioning for prolonged periods of time and the combination of the quoted treatments.

Extending the use of hardboard and MDF, along with plywood and particleboard, as rather inexpensive wood-based products is justified by modifying their dielectric properties via glow discharge plasma.

Materials and Methods

Sample preparation and treatment

Specimens were prepared from commercially available samples of MDF, hardboard, particleboard, and plywood. The adhesive type used for manufacturing was urea formaldehyde. Their dimensions were as required by the standards for measurements of internal and surface resistances of samples (Figs. 1A and 1B, respectively) as well as the capacitance of a capacitor with specimens representing its dielectric and the corresponding values of tangens of the loss angle (Fig. 2; Todorovic et al. 1997a, 1997b).

Vacuum and DC glow discharge plasma treatments were performed at the ETF-IMT Plasma Technology Centre, Belgrade. The samples were treated in an evacuated chamber (Fig. 3) at a pressure of approximately 0.5 mbar. Following chamber evacuation, nitrogen was introduced, and evacuation continued until this value of pressure was achieved in a nitrogen atmosphere. There was no voltage applied in the vacuum treatment. In the case of the vacuum treatment, the pressure value achieved was held for another 3 minutes. For the plasma treatment, at this pressure value a voltage of 750 V was applied, resulting in the discharge of the same that lasted 3 minutes. A Pirani gauge used for measuring the pressure values indicated that the pressure increased during the glow discharge plasma treatment to about 1 mbar. The particle density of applied plasma was about 10^{10} cm^3, the current was 10 mA, and the temperature achieved during the process was under 200°C. The useful volume of the chamber was Ø230 by 250 mm^3. Electrically, the cylindrical casing was the anode of the discharge, and the inner plate represented a cathode. In order to avoid a short circuit, it was important that no object placed inside the chamber touched the cathode and anode simultaneously.

The vacuum treatment was similar to common vacuum drying but in a controlled (nitrogen) atmosphere. It was legitimate to predict that vacuum alone will prove all dielectric properties of treated specimens because it will decrease the moisture content. On the other hand, the effects of the glow discharge were both heating and ion nitriding of the samples. The discharge is initiated at a voltage value slightly above 650 V. Accelerated ions with sufficient energy penetrate the exposed samples, lose their energy on the way, and are either implanted into the structure of wood-based composites and/or chemically bound, as explained in the Appendix. In both cases, the samples change their properties. The phenomenon of the pressure increase inside the chamber during plasma treatment might be explained by the heating of the cathode and the samples by the discharge, resulting in increased evaporation and the removal of the surface impurities by ion impact (Todorovic et al. 1997b, Lieberman and Lichtenberg 2005). The evaporation caused by introduced plasma is to be understood not just as drying,

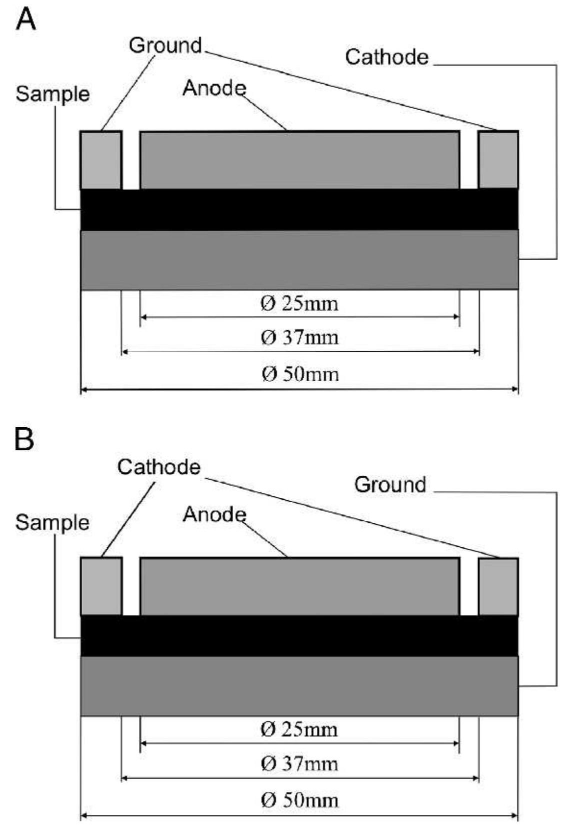

Figure 1.—Sample dimensions and connections for measurements of (A) internal and (B) surface resistances electrodes sample.

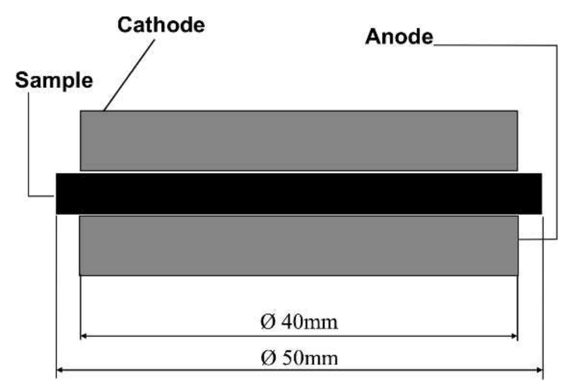

Figure 2.—Geometry of the system for measurement of capacitance and tangens of the loss angle.

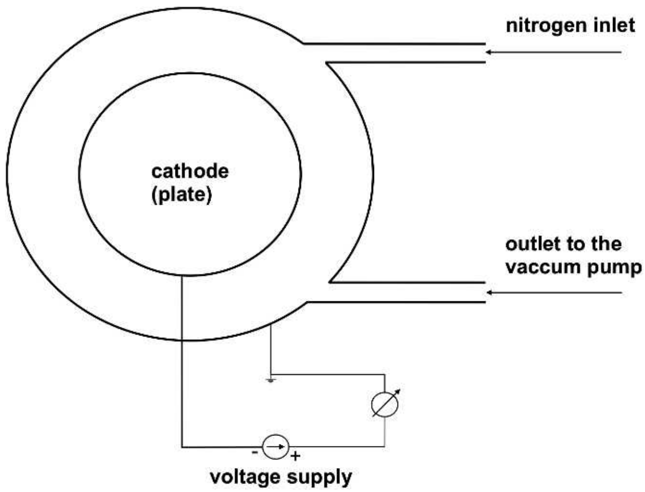

Figure 3.—Schematic representation of the vacuum and glow discharge plasma treatment chamber (pressure and temperature sensors not shown).

but also as losing a part of the surface structure. The process of etching caused by the kinetic energy of plasma particles is responsible for the latter.

All samples were, according to previous explanations, treated by one or more of the following:

- Conditioning
- Vacuum treatment of the conditioned samples
- Glow discharge plasma treatment of the conditioned samples
- Conditioning of treated samples at a temperature of 22°C ± 3°C for 1 year

Determination of the dielectric properties

Capacitances and tangens of the loss angle values were measured by an HP 4261 A RLC meter with a choice of configurations best suited for each particular measurement. The frequency used was 1 kHz (Todorovic et al. 1997a, 1997b).

Other properties that could not have been measured directly were determined by calculations according to the well-known formulae for specific resistivities and relative permittivity.

Relative dielectric permittivity for an insulating material is defined as capacitance C_x of capacitor filled only with corresponding insulating material to capacitance C_0 of the same capacitor with electrodes placed in vacuum:

$$\varepsilon_r = \frac{C_x}{C_0} \tag{1}$$

Further, capacitance C'_x of observed wood-based materials and capacitance C'_0 with parasitic capacitance of contacts C_p can be expressed as

$$C'_x = C_x - C_p$$

$$C'_0 = C_0 - C_p \tag{2}$$

The real capacitance C_0 can be easily calculated from $C_0 = \varepsilon_0 S/h$, $C_0 = 0.0695\mathrm{d}^2/\mathrm{h}$ for circular electrodes, where ε_0 is the dielectric permittivity of the vacuum (physical constant $\varepsilon_0 = 8.85 \cdot 10^{-12}$ C^2/Nm^2), S is the effective surface of protected electrode (cm^2), h is the gap between electrodes (cm), and d is the radius of the circular electrode (cm). Now, parasitic capacitance can be easily calculated from

$$C_p = C'_0 - C_0 \tag{3}$$

Expression for the capacitance with dielectric applied becomes

$$C_x = C'_x - C_p = C'_x - (C'_0 - C_0) \tag{4}$$

and for the relative dielectric permittivity

$$\varepsilon_r = C_x/C_0 = \frac{[C'_x - (C'_0 - C_0)]}{C_0} \tag{5}$$

Internal and surface resistances were measured by an Iskra MOM MA 2075 mega ohmmeter. The applied DC voltage was 1 kV. The scheme of the electrical connections for measurements is shown in Figure 1.

The definition of internal specific resistivity is described as the ratio of the voltage gradient in the current direction through the insulating material and current density.

The internal specific resistivity (TΩm) is given as

$$\rho_i = S/\delta \cdot R_u \tag{6}$$

where R_u is the measured internal electric resistance (TΩ), δ is specimen thickness (cm), and S is effective surface of the protected electrode (cm^2; Fig. 1A). Effective surface can be calculated from

$$S = d_0 \cdot \pi/4$$

where $d_0 = (d_1 + d_2)/2$ for d_1 radius of the protected electrode and d_2 inner radius of the ringed protective electrode.

The surface specific electric resistivity (TΩm) is

$$\rho_s = OR_p/l \tag{7}$$

where R_p is the measured surface electrical resistance (Ω), O is the circumference of the protected electrode (cm), and l is the distance (cm) between electrodes (Fig. 1B).

The circumference (in cm) of the protected electrode is

$$O = d_0 \cdot \pi, \quad l = (d_2 - d_1)/2$$

Owing to the strong influence of the wood moisture content to all dielectric properties, the ovendrying method was performed for control and treated samples after 1-year conditioning. The moisture content u is the weight of water contained in wood, expressed as a ratio to the weight of the ovendry wood (%):

$$u = \frac{W_u - W_0}{W_0} \tag{8}$$

The samples were weighted W_u and then exposed to the temperature of 103°C ± 2°C and maintained at this temperature until a constant weight W_0 was reached.

Results and Discussion

The measurement results of the dielectric properties after different treatments (control = long-term conditioning only, vacuum = vacuum treatment following conditioning, plasma 1y = plasma treatment following 1 year of conditioning, and plasma 2y = additional 1 year of conditioning following previous plasma treatment) are given in Tables 1 and 2 in the following order: Table 1 shows internal resistivity and surface resistivity for plywood, particleboard, hardboard, and MDF, and Table 2 shows tangens of loss angle values and relative permittivity values for plywood, particleboard, hardboard, and MDF.

Moisture content results for control (1 year of conditioning) samples, for vacuum- and plasma-treated samples, and for additionally conditioned plasma-treated samples are presented in Table 3.

It is clear that the effects of vacuum and plasma treatments are quite positive in the sense of improving dielectric properties. Internal resistivity increased as well as surface resistivity, and tangens of loss angle behaved similarly to relative permittivity in the sense of decreasing their values with the negative effect of the latter not being as important as the positive one of the former.

Vacuum treatment increased the value of internal resistivity of all samples by one order of magnitude for plywood and particleboard, by two orders of magnitude in the case of hardboard, and even by three orders of magnitude for MDF. Tangens of loss angle and relative permittivity decreased as a consequence of the vacuum treatment by approximately one order of magnitude and by 25 percent, respectively. At the same time the moisture content for all samples decreased as an expected result of vacuum drying.

Further improvement of investigated properties was achieved using plasma treatment. Increases in internal resistivity continued in the same manner for each type of composite except for hardboard, where a decrease occurred. The surface resistivity acted in an anticipated manner, increasing for all the samples. The tangens of loss angle and relative permittivity kept their former trend as well. Furthermore, plasma treatment slightly lowered the moisture content of all samples. This can be explained as an effect of higher temperature in the chamber and consequently prolonged and intensified drying. The data from Table 3 that relate to plasma 1y samples are not to be considered as exact moisture content values because there were three simultaneous processes during the plasma treatment. The drying and etching caused the loss of the primary weight, while the nitrogen implementation slightly increased the mass of the samples. However, the mass balance was negative, and therefore it can be concluded that additional drying had occurred.

Further conditioning, for one more year, of plasma-treated samples showed a decrease both of internal and surface resistivities, but to a value that is still better than for the untreated samples, reestablishing their moisture content to values of the control samples. The relative permittivity and dielectric losses increased but stayed below the values for control (untreated conditioned) samples, with the exception of the dielectric losses in the case of hardboard. The trends in the treatment effects upon the relative permittivity of plywood and particleboard are very similar to each other.

Each of the dielectric properties studied suffered changes in the same direction for particleboard and plywood (composites structurally closer to original wood). Hardboard differed from all the other cases regarding both surface resistivity and dielectric losses, the former quantity decreasing and the latter not, which was exactly opposite the trends noted for all the other composite samples. The magnitude of the effects upon both types of resistivity was stronger in the case of composites based on wood fibers (MDF and hardboard). In the case of MDF, the effects coincided with those recorded for plywood and particleboard but exceeded the scale of the latter.

Similar changes seem to have been introduced in the cases of vacuum treatments of hardboard and especially particleboard. Thus the vacuum treatment in these two cases seems to have acted as another form of vacuum drying, with all the other cases indicative of more or less persistent

Table 1.—Resistivity of investigated materials for different treatment types.

Composite	Treatment (avg. value)[a]			
	Control	Vacuum	Plasma 1y	Plasma 2y
Internal resistivity, ρ_i (TΩm)				
Plywood	0.0352	0.8326	1.13936	0.092
Particleboard	0.0728	0.47948	1.61896	0.1932
Hardboard	0.0874	6.64	1.37916	0.091
MDF	0.00572	3.15	4.25	0.0533
Surface resistivity, ρ_s (TΩm)				
Plywood	4.56	177.2	345.44	11.2
Particleboard	35.6	212.96	431.16	61.6
Hardboard	3.87	195.08	388.3	9.53
MDF	0.562	584	682	7.75

[a] Control = conditioning only; vacuum = vacuum treatment after conditioning; plasma 1y = plasma treatment after 1 year of conditioning; plasma 2y = an additional year of conditioning after plasma treatment.

Table 2.—Tangens of a loss angle and relative permittivity of investigated materials for different treatment types.

Composite	Treatment (avg. value)[a]			
	Control	Vacuum	Plasma 1y	Plasma 2y
Tangens of a loss angle, tan δ				
Plywood	0.102	0.0264	0.0182	0.0704
Particleboard	0.0814	0.031	0	0.0612
Hardboard	0.0284	0.0287	0.0091	0.0352
MDF	0.0404	0	0	0.0374
Relative permittivity, ε_r				
Plywood	4.02	3.13	2.85	3.77
Particleboard	4.17	3.43	3.26	4.03
Hardboard	3.74	2.9	2.8	3.26
MDF	4.06	3.06	3.13	3.57

[a] Control = conditioning only; vacuum = vacuum treatment after conditioning; plasma 1y = plasma treatment after 1 year of conditioning; plasma 2y = an additional year of conditioning after plasma treatment.

Table 3.—Moisture content (%) of investigated materials for different treatment types.

Composite	Treatment (avg. value)[a]			
	Control	Vacuum	Plasma 1y	Plasma 2y
Plywood	8.271	6.225	6.2014	8.209
Particleboard	12.0373	9.1722	9.003	9.8327
Hardboard	7.3321	6.35	6.299	7.2987
MDF	8.5818	7.5519	7.497	8.558

[a] Control = conditioning only; vacuum = vacuum treatment after conditioning; plasma 1y = plasma treatment after 1 year of conditioning; plasma 2y = an additional year of conditioning after plasma treatment.

structural changes in the material properties of the examined composites.

The effects of vacuum treatment decreased the moisture content of the samples, strongly influencing all dielectric properties. This is explained by the exponential correlation between moisture content and dielectric properties (Stamm 1964) causing a large increase in resistivity for a rather small decrease of moisture in the wood.

However, with further conditioning for one more year, the moisture content returned to the previous level, leaving values of the observed dielectric properties of the plasma-treated samples above values of the control. Thus, the effect of plasma treatment is not to be attributed only to the primary loss in moisture content, but also to the interaction of the nitrogen and sample surface.

Conclusions

Generally, it can be concluded that during the implantation of nitrogen ions by glow discharge plasma treatment, physical and chemical changes of the wood-based composite structure are significant and, thus, influence its dielectric properties. Vacuum treatment did not (if not applied separately) act just like common drying but introduced glow discharge stabilized modifications to the surfaces.

According to the results shown here, it is plausible to conclude that

1. Vacuum treatment influenced all dielectric properties of all sample groups, improving both surface and internal resistivity and decreasing tangens of the loss angle (except in the case of hardboard) and relative permittivity values that can be explained by the fact that vacuum treatment lowered moisture content acting like a simple drying.
2. Further improvement of resistivities and dielectric losses was achieved by the glow discharge plasma treatment, accompanied by a less significant decrease of permittivity except in the case of MDF, where it even increased above the value for vacuum treatment.
3. Comparing the effects of glow discharge plasma treatment with those of vacuum treatment, the following should be noted.
   a. Overall improvement due to the vacuum treatment was smaller than that due to the plasma treatment (which inherently included also the vacuum treatment).
   b. The effect of vacuum only (with respect to control samples) on the dielectric properties was more efficient than that of the plasma treatment itself with respect to vacuum.
4. Glow discharge plasma treatment acted as a slightly stabilizing factor for the achieved values of dielectric properties. This resulted from nitriding (or any other interaction of N_2^+ ions with composite surface) with the surface layer, which not only impeded extensive moisture penetration, but also disabled alternative (proton mechanism) current conduction (Zelinka et al. 2008).
5. Structural changes seem to have occurred with our plasma treatment in all the cases, and their effects were reduced by further conditioning and also in the cases of vacuum-treated hardboard and especially particleboard as a result of increased moisture content.
6. The trend appeared with values of all the quantities settling down closer to original (control) values.
7. The values of dielectric losses and relative permittivity remained lower than in the control case, except dielectric losses with hardboard.
8. The resistivity values stayed considerably higher than the control case, pointing to the other resistivity lowering influence apart from the drying.

More detailed research into the structural effects should be performed for the purpose of more precise interpretation, especially for the aging phenomena and in particular monitoring surface chemistry.

It seems reasonable to assume that the prolonged effects of glow discharge plasma treatment are

1. favorable regarding most dielectric properties of the composites studied, apparently improving the hydrophobicity of wood-based composites, and
2. stronger with a two-phase treatment (DC nitrogen plasma and vacuum) in comparison with the vacuum treatment alone, especially after additional conditioning, leading to the conclusion that the loss of moisture is not the only reason for improving dielectric properties.

Appendix

Glow discharge plasma in nitrogen atmosphere includes many different processes that are very common for molecular gasses. Its dynamics are characterized with the power lost electron–ion pair created going to excitation, ionization, electron-neutral elastic scattering energy losses, and kinetic energy of the electrons and ions striking the wall (Lieberman and Lichtenberg 2005). Commonly, DC glow discharge parameters are

- Pressure, 10^{-2} to 10^{-5} bar
- Voltage, 100 to 2,000 V
- Current, 0.1 to 100 mA
- Current density, 10^3 to 10^5 A/cm^2
- Temperature, 300 to 1,000 K
- Density, 10^6 to 10^{13} cm^{-3}

For applied low-voltage electric fields in the nitrogen atmosphere, the effect of primary ionization is small compared with the associative collision ionization of metastable nitrogen molecules. The processes of associative ionization due to collisions between these states have the form

$$\begin{aligned} N_2(A) + N_2(a) &\rightarrow N_4^+ + e \\ N_2(a) + N_2(a) &\rightarrow N_4^+ + e \end{aligned} \tag{9}$$

where A and a are lowest triplet and singlet metastable states of nitrogen molecules (Goloubovskii et al. 2002).

Some other glow discharge particle processes may also occur, such as

$$N_2(A) + N(^4S) \rightarrow N_2(X) + N(^4S) \rightarrow N_2(X) + N(^4P);$$

$$N_2(A) + N_2(X) \rightarrow 2N_2(X) \tag{10}$$

where A, S, P, and X correspond to excited states of nitrogen molecules (Gordiets et al. 1995).

Nitrogen anions with elevated kinetic energies collide with a cathode (and the samples placed on it) causing

- Heating of the cathode
- Electron emission

- Deposition of nitrogen ions on, and establishing new chemical bonds with, material surface constituents of the sample
- Etching or erosion of the surface to some degree

The method of nitrogen ion deposition is commonly used for different tool coating, resulting in a higher abrasion resistance. When applied to metals, nitrogen ion deposition causes some typical chemical reactions, such as

$$TiCl_4 + N_2 + 4H_2 \rightarrow 2TiN + 8HCl \quad (11)$$

forming titanium-nitride coating.

In the case of wood-based boards the expected chemical reaction is between OH^- groups and N^+ ions of surrounded plasma:

$$-OH^- + N^+ \rightarrow -ONH, \quad (12)$$

resulting in a neutral –ONH group.

Further, the application of radio-frequency low-pressure nitrogen plasma (Deslandes et al. 1998) on pure cellulose paper (simulating one of the major wood constituents) generates nitrogen-containing functionalities even for a very brief plasma exposure. According to the time-of-flight secondary ion mass spectrometry, the peak of CNH_4^+ group raised significantly with increased plasma exposure (up to 60 s) as well as $C_3NH_6^+$ and $C_3NH_8^+$. The other alterations were ascribed to the surface erosion rather than chemical and structural degradation. X-ray photoelectron spectroscopy analysis also indicates increased presence of nitrogen, suggesting $-C_xH_yON$, $-C_xH_yO_2N$, and C_xH_yN functionalities.

As for lignin, its chemical structure is much like that of polycarbonate. Nitrogen plasma treatment produces the following functional groups: C–N, C=N, and O–C≡N (Dahl et al. 1999). The lignin is water-repellent polymer, and therefore its modification supposedly does not have much influence on properties examined in this research.

The effect of cathode heating has to be in previously determined margins, not exceeding 200°C, although the process is oxygen free and consequently deprived of thermal destruction.

High temperatures can cause hornification at 80°C to 120°C, but when much longer exposure is performed (Kato and Cameron 1999). Conversion of wood components and significant occurrence of gaseous degradation products take place at temperatures above 200°C (Fengel and Wegener 1989). Thermogravimetric (TG) and differential TG studies of beechwood flour illustrated that wood decomposition began at ~200°C (Dinesh et al. 2006). There are very few data about thermal degradation of wood-based composites, but according to some (Kercher and Nagle 2002), pyrolysis of MDF occurs at temperatures from 200°C to 400°C.

Since the wood structure is fibril and similar to other thread-like structures, it is logical to presume that the wood is an ionic conductor (Zelinka et al. 2008). By reducing the amount of free ions into the structure or onto the surface of the board, it can be presumed that the conductance of observed samples will decrease.

The resistivity is given by

$$\log \rho = \alpha + \beta\varepsilon'(\omega) \quad (13)$$

where α and β are fit parameters related to the mobility and activation energy, respectively (Brown 1962). Implementing the percolation theory (Clerc et al. 1990, Stauffer and Aharony 1992, Nan 1993) the conductivity σ (and resistivity as reciprocal value of conductivity) of the composite can be described as

$$\sigma = \begin{cases} 0 & p < p_c \\ \sigma_0 (p - p_c)^t & p \geq p_c \end{cases} \quad (14)$$

where p is the percolation threshold or continuous path of the conducting phase (water) through the material (moisture content W_c for wood), σ_0 is conductivity of conducting phase, and t is the critical exponent related to the fractal dimension of the conducting path.

However, there is a certain abnormality when it comes to the wood (Zelinka et al. 2008). There is nonzero electrical conductivity below the percolation threshold, and it is believed that this is result of a different mechanism than ionic conducting where the charge carriers are protons (Christie et al. 2004).

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