

The Effect of Full-Cell Impregnation of Pine Wood (*Pinus sylvestris* L.) on Changes in Electrical Resistance and on the Accuracy of Moisture Content Measurement Using Resistance Meters

Aleksandra Konopka,^{a,*} Jacek Barański,^{a,*} Kazimierz Orłowski,^b and Karol Szymanowski^c

The impact of the full-cell impregnation of pine wood was investigated with respect to changes in electrical resistance and the accuracy of moisture content measurement. This study compared the resistance of impregnated and untreated pine timber harvested from the northern part of Poland (Pomeranian region). The wood was impregnated by the vacuum-pressure method. The preservative (TANALITH E 3475) and coloring (TANATONE 3950) agents were based on copper salts. The results showed a dependence of wood resistance as a function of the moisture content. Impregnated and not treated wood samples were used. This result reflects the greater conductivity of the impregnate solution (based on copper salt) than the water. This phenomenon became more distinctive as moisture content value was above the Fiber Saturation Point (FSP).

Keywords: Wood drying, Full-cell impregnated; Pine wood; Relative moisture content; Resistance of pine wood; Resistance moisture meter

Contact information: a: Gdansk University of Technology, Faculty of Mechanical Engineering, Department of Energy and Industrial Apparatus, G. Narutowicza 11/12 80-233 Gdansk Poland; b: Gdansk University of Technology, Faculty of Mechanical Engineering, Department of Manufacturing Engineering and Automation, G. Narutowicza 11/12 80-233 Gdansk Poland; c: Warsaw University of Life Sciences, Faculty of Wood Technology, Department of Mechanical Processing of Wood, Division of Woodworking Machines and Wood Processing, Nowoursynowska 159, 02-787 Warsaw, Poland;

* Corresponding authors: alkonel@wp.pl; jbaransk@pg.gda.pl

INTRODUCTION

Drying is the process of physically removing volatile substances (usually moisture) to obtain a solid final product (Berthold 1988). Moisture is contained in loose chemical combination (Klement and Huráková 2016). It is present as a liquid solution within the solid and is even trapped in the microstructure of the solid. Therefore it is possible to exert a vapour pressure lower than that of pure water (called bound moisture) (Klement and Huráková 2015). The remaining moisture within wood comprises unbound moisture, which is an excess above the amount of bound moisture.

The main impacts of wood saturation are dependent on the wood species, the share of sapwood and heartwood, and the anatomical direction (Krzysik 1978). Sapwood is located adjacent to the bark and represents the actively conducting portion of the stem. Heartwood, which is found in the interior of logs of sufficient age, surrounded by sapwood, can be regarded as decommissioned sapwood (Rowell 2013). In the case of pine wood saturation, the difference in the uptake of saturant by the sapwood and the

heartwood can vary by up to 110 times (Krajewski and Witomski 2005). In the tracheids of many species of coniferous wood, pits are located on the radial walls of lumens, allowing for tangential flow, while allowing the flow to take place within the lumens along the overlapping fibers. The flow along the vessels of hardwood species may be several times higher than in the tangential direction.

The increasing use of wood in construction, as a renewable and low-embodied energy alternative to reinforced concrete and steel, will play an important role in reducing emissions and solid waste derived from the global construction industry (Ramage *et al.* 2017). Wood in its natural form is a widely used construction material, but in certain environments and applications, issues related to durability, fire resistance, and dimensional stability need to be addressed (Rowell 2007). In general, the treatment of wood through chemical or thermal modifications, coatings, or impregnation offers effective routes to address some of these issues (Hill 2006). In particular, “controlled” impregnation of specific monomers into the cell cavity (lumen), but also possibly into the cell wall (Militz 1993; Schneider 1995; Keplinger *et al.* 2015), followed by polymerization, may enhance the performance of wood in construction by improving its mechanical properties (Rowell and Konkol 1987), better durability (Militz 1993; Lande *et al.* 2004), and fire resistance (Marney and Russell 2008).

For wood treatments that act on the solid mass of wood (*i.e.* on cell walls), such as chemical modification or cell wall impregnation, the obtained extent of impregnation can be directly evaluated by weight percentage gain. However, when impregnation occurs only in the luminal cavity of the cell and the cell wall is left nominally unaltered, the “maximum potential” of impregnation is better quantified relative to the total void fraction, and the obtained extent of impregnation is directly related to the pore-filling ratio (*i.e.* the ratio of luminal cavity filled to the total cavity volume).

The application of chemicals could be realized in many ways. Depending on the exposure of the wood to destructive agents, fungi, or insect infestations, different impregnation efficiencies are required (Vilkovský and Čunderlík 2017). The effectiveness of the treatment depends on the type and amount of the impregnate used and on the magnitude of wood surplus. However, the impregnation method is determined by the amount of impregnate and depth of impregnation.

To determine the depth of the impregnation, there are two kinds of methods: surface and deep. Surface impregnation includes all methods that saturate the outer layer of wood (whiteness) (up to 5 mm of impregnation depth). Deep impregnation includes methods allowing for impregnation of wood tissue up to a depth of more than 5 mm.

Lumen impregnation, unlike most other wood modification methods, is typically assessed by the pore-filing ratio (*i.e.* the fraction of luminal porosity filled) rather than by weight percentage gain. During lumen impregnation, the impregnants act on the voids in the wood rather than on the solid mass (*i.e.* cell walls) (Wu *et al.* 2017). Full cell impregnation implies that the wood is pressure-treated with preservatives in order to impregnate the full wood cell (the cell wall as well as the lumen or interior) with substances that impart resistance to decay, fire, insects and wood-boring marine animals.

Vacuum-pressure impregnation is the most effective way to achieve wood protection. Saturating is performed in cylindrical impregnation tanks using pressure or vacuum. These methods allow for the entire cross-section of sapwood and heartwood to be saturated in just a few hours for a hardwood species. With pressure-vacuum methods, the wood of many species, including pine and oak wood, is well saturated. Spruce and fir wood, however, are unevenly saturated. The unbound water, which fills lumens, prevents



easy penetration of water into wood. Depending on the method used, wood can be full-cell-saturated or “economically” (cell-free saturated). Full-cell saturation consists of filling in the all the free space in the wood with impregnating liquid. Impregnate fills the empty interior of cells and penetrates cell walls. Impregnation of wood “on the full” can be based on the usage of the vacuum-pressure method. The consumption of impregnating liquid in case of impregnation of pine wood can reach 400 L/m³. For this impregnation, the vacuum cannot exceed -0.8 bar, and the liquid pressure in the tank should be approximately 8 bar.

The aim of the present work was to investigate the electrical resistance in pine wood that is impregnated. The applied water-soluble impregnate is an aqueous salt solution that penetrates on a capillary and diffusion basis, so the moisture content of the impregnated wood does not significantly affect on its penetration into the material. The intensity of diffusion is directly proportional to the concentration of the aqueous solution of the impregnation salt and depends on the duration of this phenomenon. The diffusion process continues after removing the wood from the impregnation salt solution until the wood is dried (when its moisture content reaches below the FSP).

Similar research was performed by Brischke and Lampen (2014). However, the results presented in this article differ in that they can be related to the type of impregnate used and its concentration, as well as the method of impregnation. Forsén and Tarvainen (2000) obtained the characteristics of resistance as a function of moisture content (MC) in pine wood. They approximated these characteristics with an exponential function. Some results concerning the influence of metallic salts on the wood electrical conductivity appear in the literature (Flotaker and Tronstad 2000; Brischke and Lampen 2014, Simpson 1994). However, these data are general, describing the phenomena. They do not provide data that could be useful in industrial practice. This manuscript provides both scientific and utilitarian information, due to reference to exact quantification of the impregnation salt, instrumental evaluation of MC, *etc.* The motivation for the authors of this article was to investigate the impact of impregnation on the electrical resistance measurement.

EXPERIMENTAL

The material used in the experiments was pine wood (*Pinus sylvestris* L.). The wood to be used in the impregnation experiments (three boards) was initially dried in industrial conditions until the relative moisture content was near the FSP. Next, they were full-scale impregnated in an autoclave (Fig. 1). The impregnation process lasted 120 min, and the level of retention was 1.0 dm³/(m³·min).

The impregnation method is based on the technique that has been described in detail by Babiński (1992), called full-cell impregnation. The boards were placed in the impregnation solution under atmospheric pressure.

The first impregnation phase lasted 25 min in a vacuum of -0.8 bar. Thereafter, a pressure of 10 bar was maintained for 55 min. After a second impregnation phase when the pressure was reduced to atmospheric, the surplus of impregnation solution was removed from the autoclave. The final impregnation step, during of which the impregnation solution is sucked out of the lumens, was carried out in a vacuum of -0.8 bar and lasted 40 min. The whole impregnation process is presented in Fig. 1. A preservative (TANALITH E3475, Arch Timber Protection, Castleford, UK) and coloring

(TANATONE 3950, Arch Timber Protection, Castleford, UK) agent based on copper salt were used. The concentration of the impregnate was 3.8 %. Another three boards, which were not impregnated, were freshly cut.

It can be said there are also other preservatives, including coal-tar substances such as creosote, oil-based chemicals such as pentachlorophenol (PCP), and aqueous solutions of compounds such as chromated copper arsenate (CCA), ammoniacal copper zinc arsenate (ACZA), and copper azole (CA-B). An example of a CA-B preservative is TANALITH E3475. Creosote, PCP and CCA are used on heavy structural members such as railroad ties, utility poles, marine pilings, and bridge timbers, while ACZA and CA-B are used on common structural timber. The applied impregnate solution contains, among other things, salts, such as copper (III) carbonate and copper hydroxide. In addition it contains alcohol 2-aminoethanol ($\text{NH}_3\text{CH}_2\text{CH}_2\text{OH}$) and organic acids. The longer the chain of organic acid, the weaker it is as an acid and the more slowly it dissociates. As a result of the reaction of 2-aminoethanol with organic acids, salts are formed. Depending on their ionization, the conductivity of the salt impregnated solution changes, which may be the subject of further research.

Water as a component of the impregnate solution is a polar liquid and causes swelling of the cell wall. Cell wall swelling due to the use of water-soluble agents thus enables saturation, which is important for fungi developing inside the cell wall, in the S2 layer, such as the gray fungus.

An important problem that may occur during saturation is the fragmentation of multi-component wood preservatives as a result of the fixation of individual compounds. This in turn results in a non-uniform distribution of the chemical compound's components in wood.

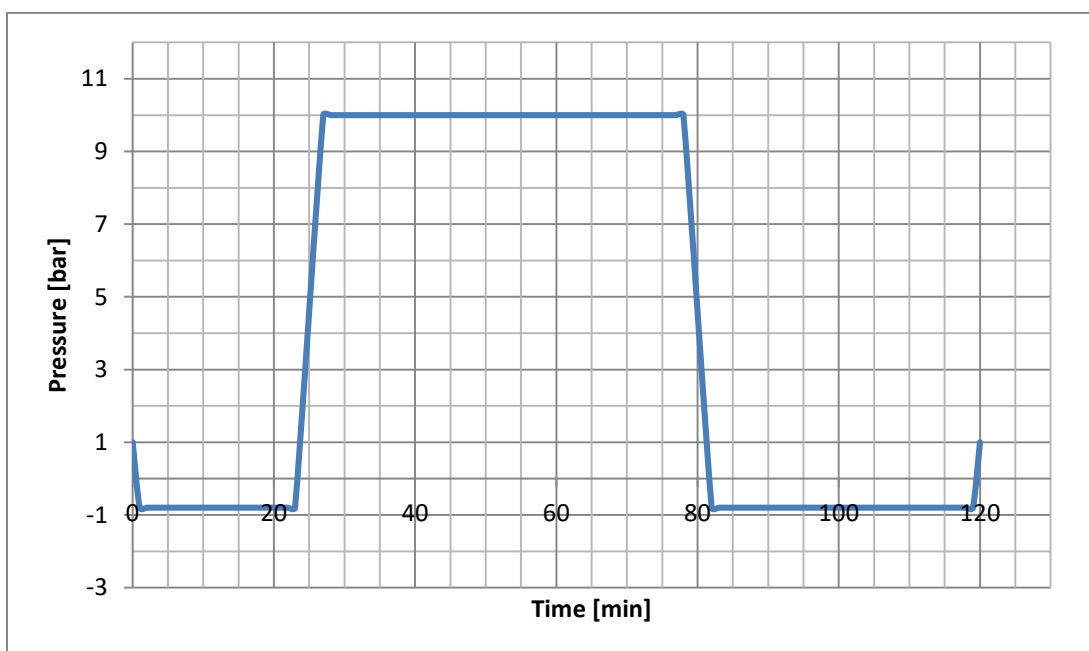


Fig. 1. Consecutive phases of the impregnation process in the autoclave

Before the experiments, the wood was provided as 500 mm length boards (Fig. 2). The growth rings of this wood were tangential (Fig. 3). The wood that was intended for impregnation was cut to pieces (below called as samples) of dimensions 120 mm ×

105 mm × 40 mm (Fig. 4a). The sawnwood (not impregnated before experiments) was also cut into pieces, but the dimensions were 60 mm × 105 mm × 50 mm (Fig. 4b).

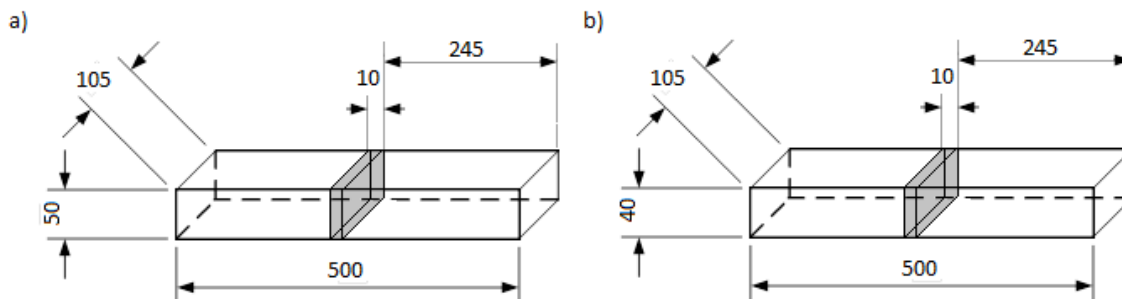


Fig. 2. Dimensions of samples prepared for experiment: a) untreated, sawnwood, b) impregnated wood. The samples taken for determination of the initial relative moisture content of wood (using gravimetric method) are marked with a grey colour.

Samples without heartwood were taken. Mainly longitudinal flow takes place in the sapwood of coniferous wood species. The wood was obtained from Sylva Ltd. Co. sawmill in Wiele, Poland. Values of initial and final moisture content and density of impregnated and untreated pine wood are presented in Table 1. These properties and the concentration of the salt in wood are very important with respect to measured electrical resistance.

Table 1. Values of Initial and Final Moisture Content and Density of Impregnated and Untreated Pine Wood

		impregnated pine wood	untreated pine wood
Average initial moisture content	MC _i [%]	27	55
Average final moisture content	MC _f [%]	6.5	8.6
Average initial density	ρ _i [kg/m ³]	640	520
Average final density	ρ _f [kg/m ³]	530	480

Each pine wood specimen was dried in the open air. The measurements were being performed in 24 h intervals in the laboratory room at 25 °C and an air relative humidity φ of 29.5%. For these parameters, the equilibrium moisture content was $W_r = 6\%$. The drying time was about 30 days for impregnated wood and about 45 days for untreated wood.

The gravimetric method was used to determine relative moisture content of wood. The samples were taken from the middle of the 500 mm boards (Fig. 2). This method is more accurate than the commonly used methods with moisture content sensors based on resistance. The experimental rig contained a scale to measure the weight of the samples. The measurements of the weights were made with a precision of 0.001 g. The drying of samples to an absolute dry state was performed in the laboratory kiln at $103 \pm 2^\circ\text{C}$. Relative moisture content was calculated using Eq. 1,

$$MC_g = \frac{m_w - m_0}{m_w} \cdot 100 [\%] \quad (1)$$

where m_w is the weight of moisture sample (expressed in grams) and m_o is the weight of absolute dry sample (expressed in grams).

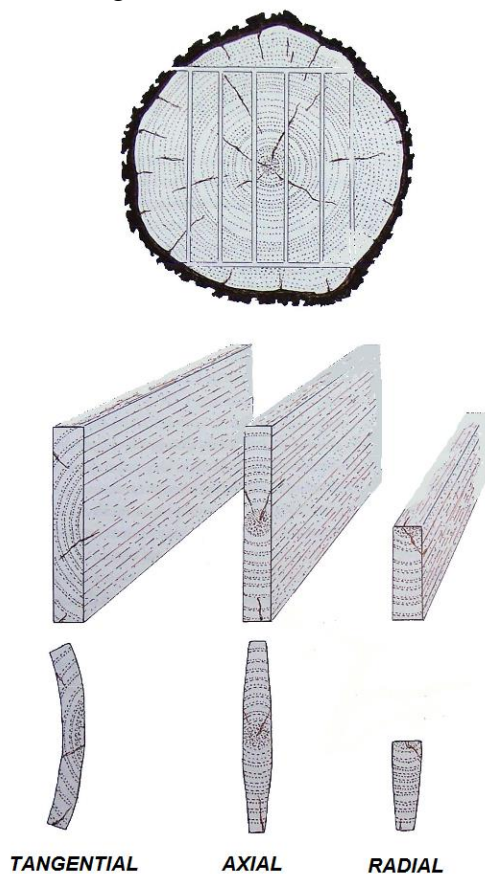


Fig. 3. Types of the orientation of the growth rings within the obtained boards

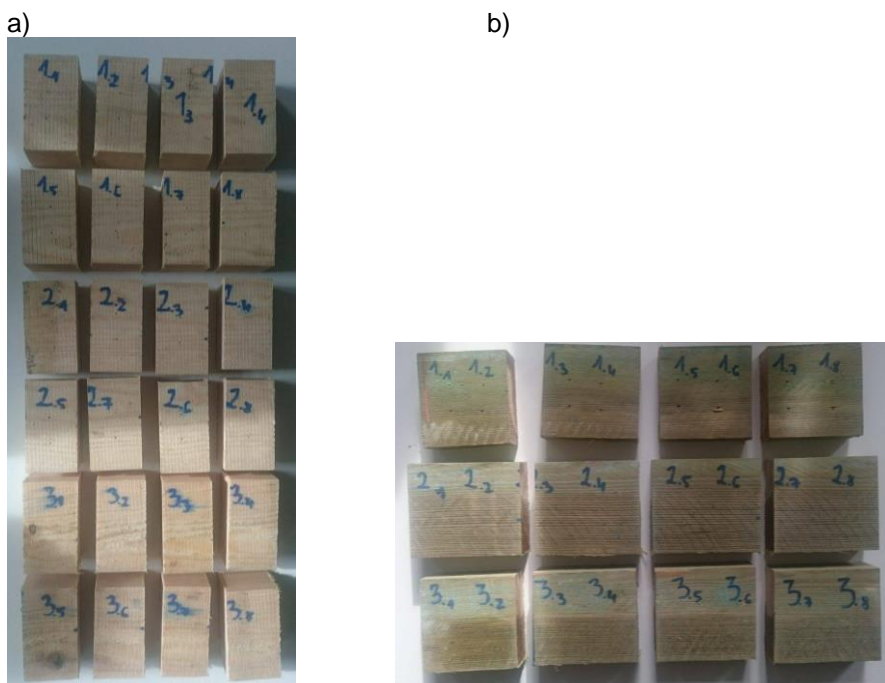


Fig. 4. The view of samples prepared for experiment: a) untreated, sawnwood, b) impregnated wood

Subsequently, the relative moisture content of the wood was measured using an electrical resistance moisture meter Hydromette type TRU 600 (Gann Mess- u. Regeltechnik GmbH, Gerlingen, Germany). The moisture meter was calibrated for a room temperature of 25 °C and for the specified wood species, *i.e.* white pine.

The measurement system shown in Fig. 5 was used to determine the resistance of impregnated pine wood and untreated sawnwood. The measuring system consisted of a multimeter type MUC 2000 (Slandi, Michalowice, Poland; Fig. 6) with an internal resistance of 10 M Ω , a power supply that generated a constant voltage of 9.45 V, and measurement probes within the Hydromette RTU 600 moisture meter (Fig. 6). Measurement probes were placed at the same measuring points in a sapwood zone.

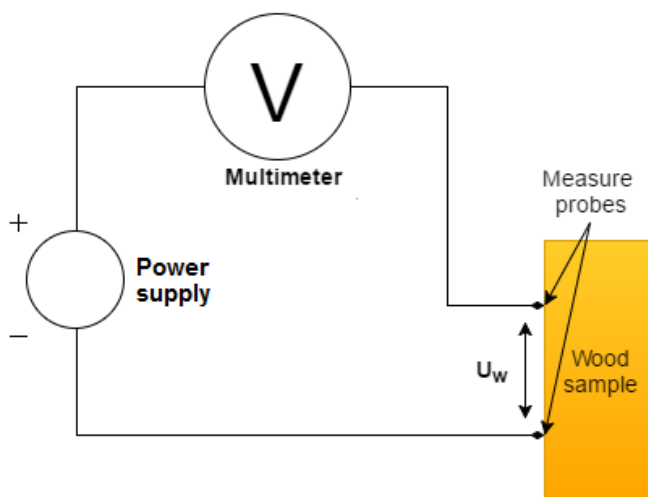


Fig. 5. Schematic diagram of wood resistance measurement system

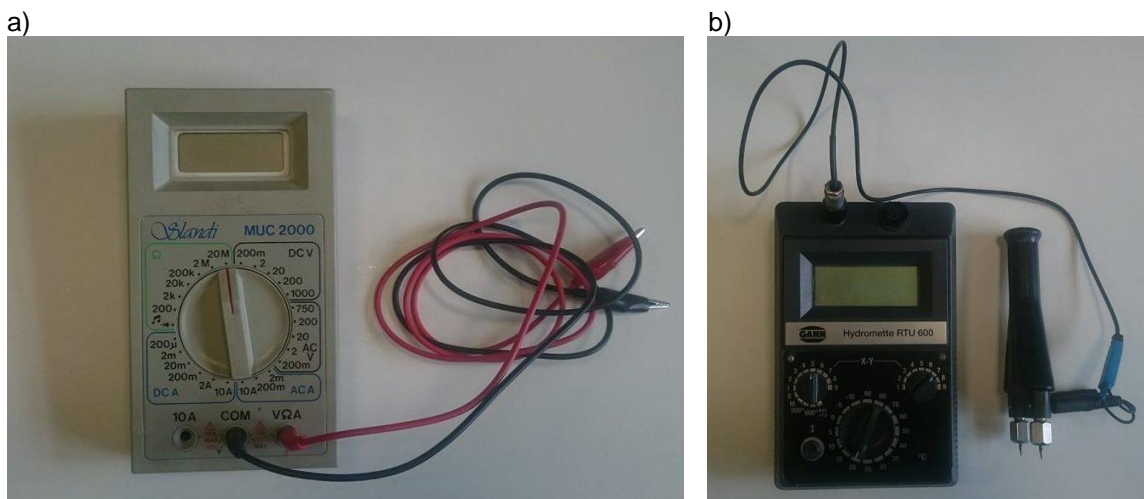


Fig. 6. Photos of measuring devices: a) multimeter MUC 2000, b) resistance moisture meter (hygrometer) Hydromette RTU 600

The resistance of the test samples was determined with the following formulae,

$$I = \frac{U}{R} = \frac{U_s}{R_m + R_w} = \frac{U_m}{R_m} = \frac{U_w}{R_w} \quad (2)$$



$$U_s = U_m + U_w$$

$$R_w = R_m \cdot \left(\frac{U_s}{U_m} - 1 \right)$$

where U_s is the constant voltage generated by power supply (9.45 V), R_m is the internal resistance of the multimeter, (10 M Ω), U_m is the voltage indicated by multimeter, U_w is the voltage of wood samples, and R_w is the resistance of pine wood.

RESULTS AND DISCUSSION

The experiment examined pine resistance as a function of its moisture content; 24 samples of untreated sawnwood and 24 samples of impregnated wood were tested. The resistance curves differed for impregnated and untreated wood. Due to different resistance of the analyzed wood, the device showed different readings. The characteristics of studied wood were approximated with an exponential function (Fig. 7). The results imply that electrical resistance falls more rapidly at first and then more and more gradually with increasing MC. In these regression curves the coefficient of determination R^2 is very high and is equal to 0.8338 for impregnated and 0.9282 for untreated wood. The deviation of the measured resistance values near to the regression curves are considerable because of the large variation in the electrical properties of wood. At higher moisture contents of wood the deviation decreases.

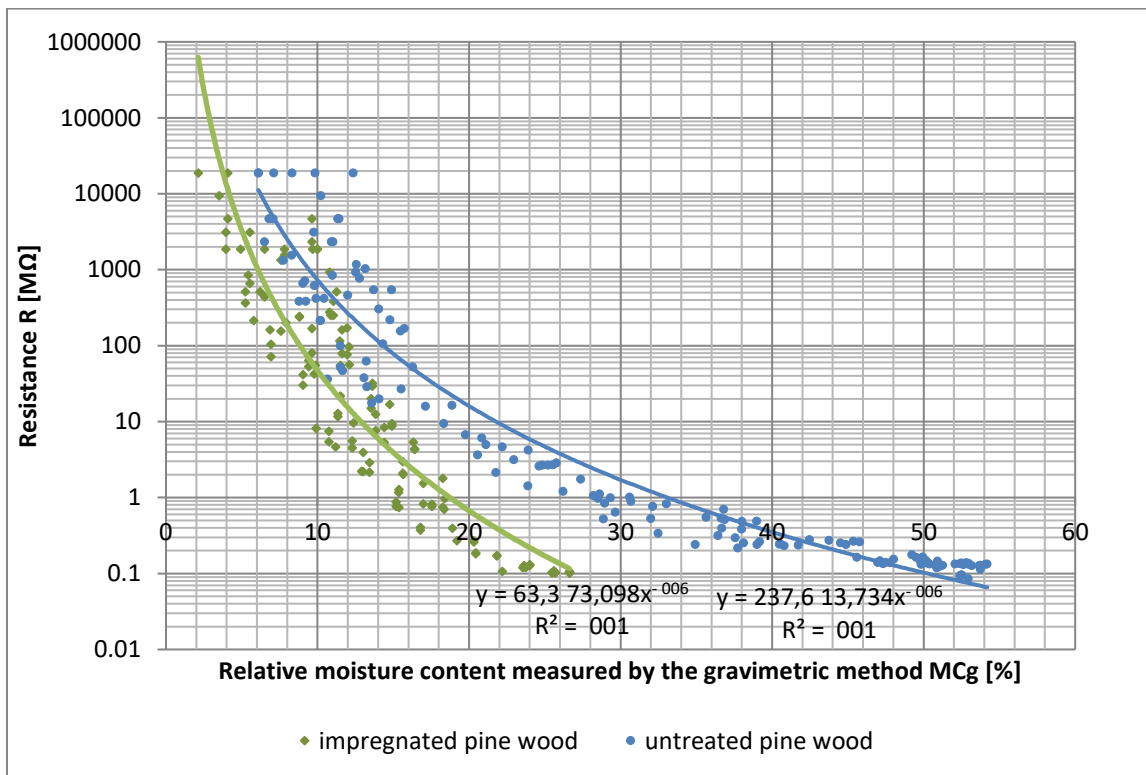


Fig. 7. Resistance characteristics of impregnated and untreated pine wood

Next, the impact of wood impregnation on the error of measuring its relative moisture content was determined using a resistance moisture meter. The real values of relative moisture content were obtained by the gravimetric method. The results are shown in Fig. 8. The untreated wood moisture content measured using the resistance meter was in good agreement with the gravimetric method. This is because there were no chemical additives that would change the resistance of the dried material. However, impregnated wood moisture content using the resistance meter was in good agreement with the gravimetric method only when it was below 20%. In such specimens there was only a small amount of water in the material, such that the chemical additives do not influence on overall wood resistance. Above 20% moisture content, there were very big differences between resistance meter and gravimetric method measurements. This is because the wood contains a mixture of water together with the chemical additives, and this mixture affects wood's electrical resistance.

The results of moisture content measurement of untreated wood with a resistance meter are characterized by a slight deviation from the true values measured by the gravimetric method to the FSP. As the MC increases above the FSP, the measurement error increases, which is consistent with the information in the resistance meter manufacturer's manual. In the case of this measurement for impregnated wood, the deviation increased exponentially above the values of MC equal 15% (measured by gravimetric method). Above this value, the use of an appropriate correction formula is needed.

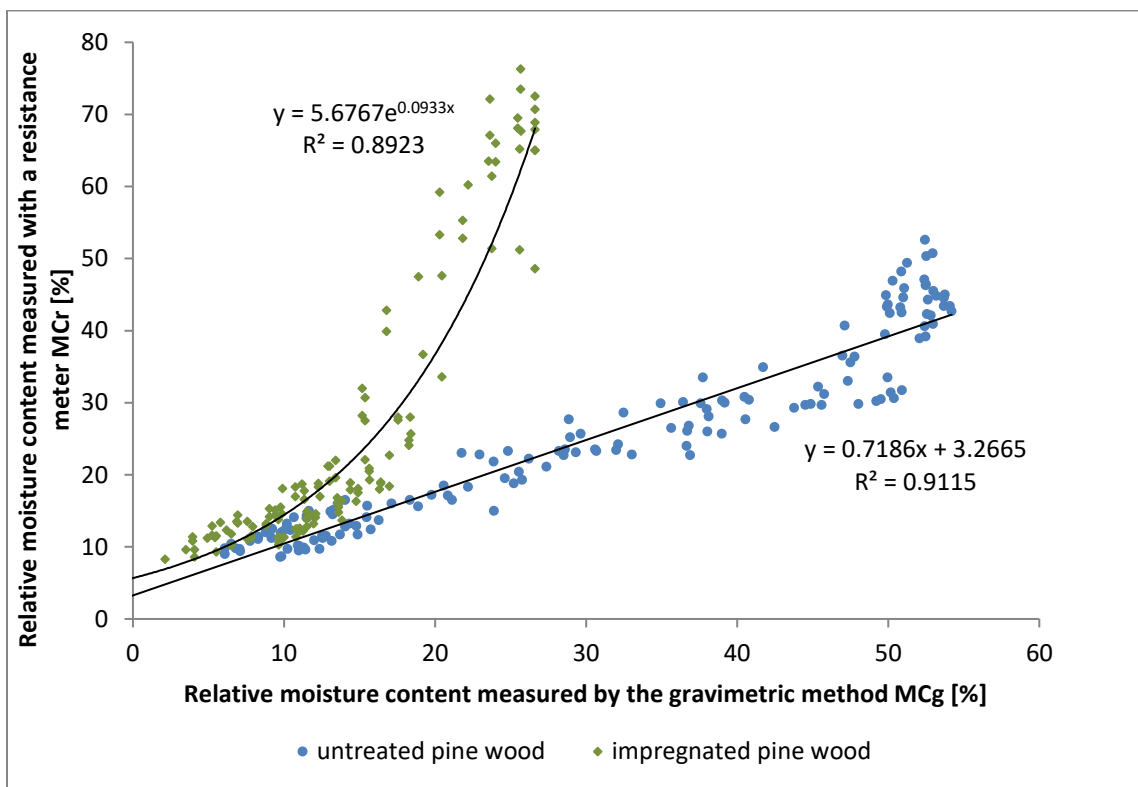


Fig. 8. Measurement error of the relative MC of pine wood as a result of change in the resistance of impregnated wood.

CONCLUSIONS

1. The method of resistance moisture content measurement is not suitable to measure the MC of impregnated pine wood. The application of this method requires correction formulas, which would need to be estimated empirically with respect to the type and amount of impregnant in the wood.
2. The full-cell impregnation of pine wood (*Pinus sylvestris* L.) influenced the resistance values and the accuracy of moisture content measurements. Impregnation of wood with the preservation and colorization agents (that were TANALITH E3475 and TANATONE 3950, respectively) decreased the electrical resistance of the resistance and as a consequence increased the apparent measured moisture content that would be predicted with the moisture meter (Hydromette RTU 600) with the default calibration settings.
3. Measurements of moisture content of impregnated pine wood using a resistance meter significantly differed from relative moisture content measured with gravimetric method. Such a phenomenon was especially noticeable above FSP.
4. The coefficient of determination R^2 for untreated wood was higher than for the impregnated wood based on separate equations used to fit the data. The results corresponding to impregnated wood were better fitted using an exponential rather than a linear function.

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