

DEVELOPMENT OF RESISTANCE-TYPE CONTROL METHODS FOR WOOD DRYING

ELEKTRILISTE TAKISTUS-TÜÜPI KONTROLLIMEETODITE ARENDAMINE PUIDUKUIVATUSES

VALDEK TAMME

A Thesis for applying for the degree of Doctor of Philosophy in Forestry

Väitekiri filosoofiadoktori kraadi taotlemiseks metsanduse erialal

Tartu 2016

Eesti Maaülikooli doktoritööd

Doctoral Theses of the Estonian University of Life Sciences



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According to verdict No 2 of June 1, 2016, the Doctoral Committee of the Forestry of the Estonian University of Life Sciences has accepted the thesis for the defence of the degree of Doctor of Philosophy in Forestry.

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Defence of the thesis:

Estonian University of Life Sciences, room 2A1, Kreutzwaldi 5, Tartu on August 25, 2016, at 13:00.

The English in the current thesis was revised by Tiia Kaare and the Estonian by Ülle Sihver.

© Valdek Tamme, 2016 ISSN 2382-7076 ISBN 978-9949-569-38-0 (trükis) ISBN 978-9949-569-39-7 (pdf)

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LIST OF ORIGINAL PUBLICATIONS

The thesis is based on the following papers, references to which in the text are given by Roman numerals. The papers are reproduced with the kind permission of the publishers.

- Tamme, V., Muiste, P., Mitt, R., Tamme, H. 2011.
 Determination of Effective Diffusion Coefficient and Mechanical Stress od Pine Wood during Convective Drying. *Baltic Forestry*, 17: 110–117.
- **Tamme, V**., Muiste, P., Padari, A., Tamme, H. 2014. Modelling of Resistance-Type Wood Moisture Meters for Three Deciduous Tree Species (Black Alder, Birch, Aspen) in Moisture Contents Above Fibre Saturation Point. *Baltic Forestry*, 20 (1): 157–166.
- III Tamme, V., Muiste, P., Kask, R., Padari, A., Tamme, H. 2012. Experimental study of electrode effects of resistance type electrodes for monitoring wood drying process above fibre saturation point. *Forestry Studies*, 56: 42–55, v10132-012-0004-6.
- IV Tamme, V., Muiste, P., Tamme, H. 2013. Experimental study of resistance type wood moisture sensors for monitoring wood drying process above fibre saturation point. *Forestry Studies*, 59: 28–44.

Paper	Ι	II	III	IV
Original idea	VT	VT	VT	VT
Study design	VT	VT	VT	VT
			ΗT	ΗT
Data	VT	VT	VT	VT
collection	RM		RK	
Data analysis	VT	VT	VT	VT
	AP	AP	AP	
Preparation of	VT	VT	VT	VT
manuscript	ΗT	ΗT	ΗT	ΗT
-	\mathbf{PM}	PM	\mathbf{PM}	\mathbf{PM}

The contributions of the authors to the papers were as follows:

RK – Regino Kask, PM- Peeter Muiste, RM – Risto Mitt, AP – Allar Padari, HT - Hannes Tamme, **VT – Valdek Tamme**

ABBREVIATIONS

alternating current
constant phase element
direct current
three-dimensional
one-dimensional
electrical impedance spectroscopy
electrical single impulse response (method)
fibre saturation point
James's hypothesis
moisture content
magnetic resonance
magnetic resonance imaging
noise (signal)
near-infrared spectroscopy
polarization-depolarization (method)
reference electrode
relative humidity (of air)
(useful) signal
research question
standard error (of regression model)
Vermaas's hypothesis

1. INTRODUCTION

In Estonia chamber-type convection dryers are widely used for drying sawn timber with heated air. Convection is a transfer phenomenon where the transfer of heat (and also water vapour) occurs upon a turbulent mixing of gas (in this case drying air). Convection dryers can be warm air and hot air dryers depending on the applied temperature. In the case of warm air dryers an operating temperature range of 50– 80°C is used, the regular range being 50–60°C. In the case of hot air dryers the drying temperature is raised above 100°C. At high temperatures the speed of moisture release from wood rises and drying time shortens due to the increased water vapour diffusion coefficient. A disadvantage of hot air dryers is the resulting relatively low quality of timber. This thesis focuses on warm air dryers and the related drying control methods.

In Estonia the main drying control method used in most popular convection dryer types (e.g. Mühlböck, Secal, Nardi, Hekotek, Vetorex, etc.) is the control automatics, which is based on the principle of electrical resistance measurement. For monitoring electrical resistance of wood 4–10 measuring channels in these dryer types are used. In general insulated pin sensors that are hit into wood with a hammer or insulated screw sensors that are screwed into wood are used as resistance-type sensors.

The electrical resistance method is widely used for monitoring wood drying because this method is economical and reliable (Tronstad et al., 2001; Onysko et al., 2008). However, above the fibre saturation point (FSP), in case of which the moisture content (MC) is 30%, the measurement is complicated as the measuring accuracy is insufficient and starts decreasing with increasing wood MC (Edwards, 1974; Vermaas, 2002). The decrease in measuring accuracy can also be described quantitatively, by the 2S-value (Rozema, 2010). Insufficient measuring accuracy above the FSP also reduces the accuracy of effective diffusion coefficient monitoring in the process of wood drying (Tamme et al., 2010, 2011). For precise determination of MC it is recommended that moisture meter measurements should be combined with the oven dry method for the measuring range above the FSP (Brookhuis, 2009).

Besides the method based on the measurement of electrical resistance other methods have also been used in laboratory research on wood drying.

- (a) Microwave method (James et al., 1985). By its working principle it is a regular method based on measuring electrical capacitance, but it uses extraordinarily high frequencies (4.5– 6.0 GHz). The use of ultra-high frequencies allows significant reduction of the dimensions of the measuring capacitor. The microwave method allows determining both the average MC and the MC gradient in wood.
- (b) X-ray scanning computing tomographic method. (Danvind, 2005; Cai, 2008). The working principle of this method is the absorption of X-rays in a medium. This method is mainly used for monitoring MC gradient of wood.
- (c) Nuclear magnetic resonance (MR) and magnetic resonance imaging (MRI) method. It is used in laboratory research for monitoring average moisture content as well as the MC gradient of wood (Lamason et al., 2013). The working principle of this method is the spin resonance interaction of strong external high-frequency magnetic field and water molecules in an environment containing water (wood contains over 30% water when its MC is above the FSP, but e.g. the human body contains ca. 70% water, etc.).
- (d) Neutron activation method. The working principle of this method is similar to the X-ray method, but instead of X-rays that are absorbed in wood it uses neutron beams consisting of elementary particles without electric charge that are directed to the wood being researched (Mannes et al., 2009). In order to create a low intensity neutron beam a radioactive isotope californium-252 (Cf-252) is used. In case a more powerful neutron beam is required for researching wood, a nuclear reactor has to be used as the neutron source. This method has been used for monitoring the MC gradient in wood (Mannes et al., 2009).
- (e) Near-infrared spectroscopy (NIRS) method. This method represents a modification of a known infrared thermography method (in practice also known as thermal camera method). Spectroanalysis of the absorbed component of the long-wave (wavelength range 700–2500 nm) infrared light reflected from

the wood surface is carried out and conclusions are drawn about the properties of the absorbed surface. The NIRS method is used to determine the moisture content on the surface of the discs sawn from wood across the grain and used between the wavelengths of 939.5 and 1796.6 nm (Hans et al., 2013).

Regarding the quality of drying, it is also important to measure in real time the triaxial stress that occurs in wood to obtain information on the risk of possible drying cracks and other defects. The research on triaxial stress in wood is still in early stages (Zelinka et al., 2007). Qualitative information on the existence of triaxial stress is provided by the acoustic emission method (Kawamoto and Williams, 2002). Unfortunately, this method does not provide three-dimensional tensile and compressive stresses with quantitative values about the distribution of stresses in wood at different depths starting from the surface.

The traditional approach to the problems with stresses in wood is as follows. First, deformations that occur on the wood surface in the drying process are measured either optically (Rémond et al., 2007) or with contact-type deformation sensors (Lazarescu et al., 2010). Then the deformation measurements are calculated using stresses from Hooke's law by means of strain-stress relations.

Compared to the previously described simple methods the most advanced numerical calculation algorithms for drying stress are used by wood drying simulation programs (Torksim, Transpore etc.). For this reason, the wood drying simulation program may be considered as one of the wood drying stress control methods.

Sometimes (usually upon deployment of a new drying schedule in production) the control methods in the standard equipment of an industrial timber dryer are insufficient. The control methods described and developed in the current thesis are at least temporarily usable in an industrial timber dryer as well. This would be a potential practical future benefit gained from this doctoral thesis in addition to new knowledge on resistance-type control methods.

2. REVIEW OF LITERATURE

2.1. Electrical resistance method for determining the MC of wood

The electrical resistance method is known since 1927 when Stamm (Stamm, 1927) presented his formula for a simple linear relation between the common logarithm of electrical conductivity and the relative MC of wood (regarding dry weight). The electrical resistance method is temperature sensitive. The respective electrical resistance temperature compensation formulas were derived from the Arrhenius law by Norberg (1999). Large-scale comparison of this method as well as calibratability research was carried out within the IMCOPCO programme (Forsen and Tarvainen, 2000). It was found that when the MC is below the FSP (below 30% MC to 4% MC) the electrical resistance method is calibratable on the basis of the moisture standard ISO/IEC 98-3:2008, and calibrations of moisture meters from different manufacturers can be compared to one another. It is recommended that in the changeover area, where the MC is 20-30%, a somewhat different formula than Stamm's formula should be used for the calibration of the moisture meter (Straube et al., 2002).

2.2. Vermaas's hypothesis

Vermaas's hypothesis (Vermaas, 2002) includes a negative assessment for the possibility of practical use of resistance-type wood moisture meters when the MC is above the FSP (30–150% MC), since there is no reliable calibration function for these moisture meters in that area (Bergman, 2010; Ressel, 2006; Brookhuis, 2009). By using statistical modelling it was shown in (Tamme et al., 2012, 2014) that Vermaas's hypothesis has two specific distinguishable exceptions where the calibration function clearly exists. Therefore, the resistance method can also be used in practice under specific limited conditions and only within these exceptions in an area above the FSP (MC 30–150%).

2.3. Wenner method

In geophysics a measuring method with four electrodes (Wenner, 1916) is used for determining soil resistivity. Differently from the

measuring method with two regular electrodes the Wenner method allows eliminating the contact resistance effect between soil and electrodes, which in some cases may be remarkable (Schuetze, 2003). The Wenner method has been adjusted to measuring the electrical contact resistance of semiconductors (Valdes, 1954) and between the roots of growing trees and topsoil (Urban et al., 2011). In (Tamme et al., 2012) a Wenner-type measuring system with four electrodes was used for the separation of the measuring electrode /wood contact resistance and wood electrical resistance in pine softwood above the FSP with the MC range 30–140%.

2.4. Electrode effects at wood MCs above the FSP

Measurement of wood electrical resistance with MCs above the FSP involves a few phenomena that were first pointed out by Skaar (1964). Skaar highlighted the polarization effect, which makes the measured electrical resistance dependent on time and measurement causes formation of deposits on electrodes during long-term measurements. The general polarization theory of dielectrics is described in general physics and theoretical physics textbooks (e.g. Debye, 1945; Savelyev, 1975). Rather comprehensive experimental research on electrode effects can be found in (Tamme et al., 2012, 2013) where the relative measuring errors caused by them as well as the various effects of moisture sensitivity are studied. The research also tries to provide mathematical descriptions of various effects.

2.5. Electrical single impulse response method (ESIR method or PD method)

The method for studying the polarization/capacitive effects in wood (Tamme et al., 2013) was developed based on methods of determining the corrosion current in reinforced concrete (Petersen, 2003) and soil chargeability (Van Voorhis et al., 1973; Summer, 1976). This method allows studying processes with long and extremely long relaxation time in wood during the polarization phase and the following depolarization phase in the time domain. Considering the order of the measuring processes, the method can be called, in short, the polarization–depolarization method (PD method, PDM).

The permitted limits of voltage and current strength used in this method have been set according to the optimal values found in (Romann et al., 2014).

2.6. James's hypothesis (JH)

In 1975 W.L. James proposed a hypothesis concerning a multitude of relaxation times in wood (James, 1975). The validity of the hypothesis in the frequency domain was proved by Zelinka et al. (2007). In the frequency domain the relaxation time in the experiment is not a directly measurable variable but it is presented via complex theories by Cole and Cole (1941), Debye (1945) and Zelinka et al. (2007). In (Tamme et al., 2013) James's hypothesis is referenced and its validity is proven in the time domain, although proving the hypothesis was not the direct objective of that article.

2.7. Mahematical modelling of wood drying

Mathematical models of wood drying are based on the Luikov-type differential equations system that describes heat and mass transfer in wood (Luikov, 1966). Luikov-type equations are usually solved numerically (Younsi et al., 2006). In wood drying simulation programs solving a Luikov-type equation is also added to the numerical calculation of the stress in wood (Salin, 1990; Rémond et al., 2007). The simulation program TORKSIM ver. 3.1 developed by Salin (2007) was used in this thesis.

2.8. Monitoring of wood drying

Monitoring options in industrial timber dryers are quite limited. In addition to monitoring parameters important with regard to the completion of the drying schedule (such as relative humidity, temperature and velocity of the drying air), the average MC of wood in the dryer is also monitored in real time, usually with the electrical resistance method. Monitoring results in a drying curve shows the time dependence of the average MC of wood. A significantly more reliable experimental drying curve can be obtained in an industrial dryer with special reference specimen and using an oven-drying method (Tronstad et al., 2001). The Master's (Poljakov, 2013) and Bachelor's theses (Mändoja, 2015) supervised by the author of this thesis involved the use of the oven-drying method described by Tronstad et al. (2001) for validating the simulation program TORKSIM in industrial conditions to achieve an experimental drying curve, but for checking the time interval of the occurrence of the simulated maximum drying stresses a case hardening test was used as well. A good match was found between the simulated and experimentally determined drying results, which confirmed the reliability of the wood drying stresses calculation algorithm of the TORKSIM program.

2.9. Research needs

The current study attempts to develop wood drying control methods mainlyby making use of the new possibilities offered by the traditional resistance method. To discover new possibilities of the resistance method at MCs above the FSP requires extensive original, experimental and theoretical research, first and foremost explaining the nature of James's and Vermaas's hypotheses based on macro- and microphysical parameters of wood and the dielectric polarization theory.

3. AIMS OF THE STUDY

The aim of this thesis is to describe and mathematically analyse the electrode effects involved in the wood MC measurement when the MC is above the FSP.

The main hypotheses of the study were:

- Vermaas's hypothesis on the calibratability of resistance-type wood moisture meters at MCs above the FSP is generally still valid, but there are a few possible exceptions.
- James's hypothesis on the multitude of relaxation times in wood is also valid in the time domain (e.g. in the case of measurements performed with the PD method).

The specific aims of the study were:

- To find possibilities of compensating for well-known weaknesses of the electrical resistance method and for increasing the measuring accuracy of this method at wood MCs above the FSP.
- To find a satisfactory electrical equivalent circuit for the developed PD method.
- To find ways for determining an experimental local diffusion coefficient in the wood drying process using the electrical resistance method based on Fick's first law.

4. MATERIALS AND METHODS

4.1. Methodology for studying electrode effects (III)

In studying electrode effects on a raw data file (set of unprocessed measurement data with numeric or analogous filters) of electrical resistance measurement (figure 4 in III), a pine sapwood specimen with the dimensions of 100 mm x 60 mm x 60 mm (length x width x thickness) was used. It was dried in the climatic test chamber Feutron (Feutron, 2013) at room temperature (20°C, relative humidity 96% RH) in stationary air for 90 hours and thereafter at 32°C and 96% RH at the air velocity 0.4 m s⁻¹ for another 244 hours. During the drying process of a total of 334 hours the wood MC was reduced from 146% to 36.4%.

For slow processes like the resistance of electrode-wood contact and corrosion, the resistance meter Scanntronik Material Moisture Gigamodule with Scanntronik Thermofox data logger was used (Scanntronik, 2013). For measuring the voltage of the slow process of residual polarization, the profi-tester Meterman 38 XR was used (The Test Equipment Depot, 2013). In measuring slow processes, the minimum interval between two measurements was 1 hour.

For measuring the fast process of polarization resistance, the resistance meter AlphaLab Inc. was used within the measurement range of 0-20 M Ω and measuring accuracy of $\pm 2\%$ (Trifield, 2013). For saving measurement data in the data logger Ahlborn (Type ALMEMO 2590-9) (Ahlborn, 2013), an analogue output (with a range of 0–1 V), which had a linear connection with the resistance measuring range of 0-20 $M\Omega$, was attached to the resistance meter AlphaLab. Readings of the analogue output and Alphalab display were related by the following formula: 10 (analogue output reading in volts (V) = display reading in megaohms (M Ω). The voltage of the analogue output could be measured at a speed of ten measurements per second or one measurement per second by using the Ahlborn DC voltmeter ZA9000FS3. For measuring the voltage of another fast process, depolarization, the Meterman 38 XR profi-tester with an input resistance of 10 M Ω was used, which enabled the measurement speed of one measurement per second. For the measurement during the

experiments insulated pin electrodes were used (ram-in electrode M18, 60 mm, manufactured by Gann).

In the measurement of electrical resistance, polarization resistance, depolarization process and residual polarization, electrodes e1 and e2 were used at a distance of L = 30 mm (measuring direction was across the grain) (see figure 2 in (III)). In examining electrode/wood resistance R_c and electrode corrosion, electrodes e1 and e2 as well as an extra pair of electrodes e3 and e4 were used at a distance of L/2 = 15 mm. The distance between the main measuring electrodes (e1, e2) and extra electrodes (e3, e4) was D = 40 mm. Distance D was experimentally selected in order to avoid interaction between neighbouring electrodes, i.e. transmission of electrical potential by wet (143% MC) wood. The difference between transmitted potentials (electrical voltage) was measured in dead neighbouring electrodes, and was found to be below 1/30 of the measured voltage when D = 40 mm.

The specimen was weighed with a scale manufactured by KERN (Model KERN EW B 620-M, resolution 0.01 g) (Kern and Sohn GMBH, 2013) at 24-hour intervals on average; the oven-dry weight of the specimen was determined with the same scale.

Gann HT 85 T (Gann, 2013), NDT James Moisture Master (NdtJames Inc., 2013) and Brookhuis FMD-6 (Brookhuis, 2013) resistance-type wood moisture meters were used in the experiments.

Unfortunately, the electrical resistance measurement raw file volume and internal data processing algorithm in the resistance meter Scanntronic Gigamodule and in the resistance type wood moisture meters used in the experiments remained hidden from the user.

4.2. Methodology for studying James's hypothesis (H1 in (IV))

James's hypothesis was referred to in paper IV: 'Polarization and depolarization processes in wood have a complex physical background. James (1975) has named three classes of mechanisms to explain the dielectric properties of wood: (a) mechanisms with short relaxation times including electronic, atomic, and fast molecular polarizations; (b) mechanisms with intermediate relaxation times including slow molecular, fixed dipole, and fast interfacial polarizations; and (c) mechanisms with long relaxation times including slow dipole and interfacial polarizations. James's analysis was based on different polarization times of molecules.'

Electrical resistance is determined for an equivalent circuit directly with a resistance meter, as described in (Tamme et al., 2013). If the applied working voltage is known, the strength of the current flowing through wood can be calculated using Ohm's law. Another option is to measure the voltage applied to measuring electrodes and the strength of the current flowing through wood, and then calculate wood electrical resistance using Ohm's law. The sum of (different) electrical capacitances in the equivalent circuit is calculated using measurement results and empirical equations (6) and (7).

To examine wood polarization and depolarization processes on a black alder specimen (20°C, 96% RH) an experimental setup was used, the circuit diagram of which is given in Figure 1.



Figure 1. An experimental circuit diagram of the polarization–depolarization method.

An Ahlborn 2.6 V DC voltmeter (type ZA9000FS3) with an input resistance of 10 M Ω was used in the experimental setup. As electrometers E_1 and E_2 the Keithley Model 6517B electrometer was used according to the positions of switches S_1 , S_2 and S_3 (Keithley, 2013).

The experimental assessment of the JH and also the study of electrode effects in the previous section were based on the optimum values of current intensities and voltages in the measuring cell, which were used in (Romann et al., 2014), in order to avoid the unwanted effect of electrolysis on the measuring results. The maximum allowed measuring voltage of ionic liquids is 4.1 V at a smooth carbon electrode and 3.5 V at a porous carbon electrode. The maximum current density used in the experiment is ~1–20 μ A cm⁻² at a smooth measuring electrode. The maximum voltage of aqueous solutions is usually 1.2 V. In the case of low-conductivity materials (such as wood which has the resistance of approximately 10 k Ω up to 100 k Ω in the MC range above the FSP) the Ohmic voltage drop occurs and applied voltages may be up to three times higher (up to 3.6 V). In the experimental assessment of the JH the impulse voltage of 2.6 V was applied and in studying electrode effects the voltage values of 1–3.5 V were applied.

The status of the input relay switches S_1 , S_2 and S_3 disconnected from the optically isolated circuits was controlled with the Raspberry microcomputer. The functions of the microcomputer included timing of the measurements, data storage and file management. Since the circuit diagram enables studying the processes of wood polarization and depolarization sequentially as a kind of response to a single voltage impulse, this type of measurement described in paper IV was called the electrical single impulse response method (ESIR method), or in short, the polarization–depolarization method (PD method).

4.3. Methodology for studying Vermaas's hypothesis (H2 in (IV)). (II and IV)

Vermaas's hypothesis states that there is no calibration function (for the purposes of the ISO/IEC 98-3:2008) for resistance-type wood moisture meters in wood MCs above the FSP. If there is no calibration function, there can be no accurate one-to-one correspondence between the measured electrical resistance and the wood MC; therefore, the method proves useless in practice in the range above the FSP.

In examining Vermaas's hypothesis, an attempt was made to answer two research questions (RQ): First, is it possible to recalibrate resistance-type wood moisture meters with the manufacturer's calibration to function better and more accurately in the range above the FSP (above 30% MC) (RQ1)? Second, what are the parameters of the obtained calibration model if an electrical resistance meter is calibrated into a wood moisture meter (RQ2)?

(RQ1): The research was made using specimens of black alder (Alnus glutinosa), silver birch (Betula pendula) and European aspen (Populus tremula), each with the dimensions of 100 mm x 60 mm x 60 mm (height x width x thickness). The specimens were dried in the Feutron climatic chamber (feutron, 2013) under equal conditions (32°C, 98% RH, air velocity 0.4 m s⁻¹) until the desired MC was achieved. All wood MC measurements were carried out at a room temperature of 20°C. Resistance-type wood moisture meters Gann HT 85 T (gann, 2013), and NDT James Moisture Master FMD-6 (brookhuis, 2013) (ndtjames, 2013) by three different manufacturers were used. The measurement resolution of all moisture meters used in the experiments was $\pm 0.1\%$ MC. The chosen measuring depth of measuring electrodes was 1/3 of the thickness of the specimen (that is, in the case of a 60 mm specimen, the measuring depth was 20 mm from the surface of the specimen). The number of specimens per each tree species was n = 60.

(RQ2): The testing method involves producing a statistical prediction model for a specific tree species and specimen dimensions, given drying schedule and range of the average MC of the specimen. The reliability of the model is assessed with tests on the normality of regression residuals and visually by using the probability paper. Model testing may be supported by the individual calibration method for pin electrodes, as seen in researches by Tamme et al. (2012) and Lazarescu et al. (2010). Details of model repeatability conditions (i.e. validation) have been analysed by Tamme et al. (2013) in (III). In the experiments a black alder specimen with the dimensions of 500 mm x 120 mm x 35 mm (length x width x thickness) conditioned in the Feutron climatic chamber (Feutron, 2013) at a room temperature of 20° C and relative humidity of 96% RH for 72 hours and thereafter at an average temperature of 50° C for another 96 hours according to the drying schedule (table 1 in (IV)) was used. The ends (120 mm x 35 mm) and sides (500 mm x 35 mm) of the specimen were coated with nitrocellulose lacquer to avoid water vaporization. By adopting this measure, one-dimensional (1D) moisture distribution was ensured in the specimen.

The specimen had been sawn from a black alder log in a way that the length of the specimen was oriented along the grain, the width across the grain in the tangential direction and the thickness across the grain in the radial direction. The sawn specimen was located approximately 8 cm from the pith of the log.

Electrical resistance measurements of wood were conducted with one specimen in 80 different pin electrode insertion spots and at an equal measuring depth of 12 mm from the surface of the specimen (that is, 1/3 of the thickness of the specimen). Mathematically, it may be demonstrated that in the case of a parabolic moisture profile, the local MC numerically equal to the average MC is evident approximately at a depth of 1/3 from the shield of the wood specimen (Kretchetov, 1972). Due to the short duration of measurements (not more than one minute in the spot of the application of the electrodes), the effect of electrode corrosion was neglected.

The relative MC of the specimen according to dry mass was determined in compliance with the standard ISO 3130:1975. Based on dry weight and electrical resistance measurement data, a statistical regression model was later produced for the specimen dried according to a specific drying schedule (table 1 in (IV)). Teflon insulated pin electrodes (ram-in electrodes M18) with a length of 40 mm manufactured by Gann (Gann, 2013) were used as measuring electrodes in the experiments. A resistance meter Scanntronik Material Moisture Gigamodule (Scanntronik, 2013) was used to measure the electrical resistance of wood.

4.4. Theoretical backround to wood drying and methodology for determining significant parameters

4.4.1. Coupled, uncoupled and diffusion-based simplified models

The coupled model to calculate the combined heat and moisture transport through a porous medium was developed by Luikov (1966), and specific to wood by Siau (1984).

The governing equation for heat transfer through wood board is

$$\rho c_p \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(\lambda \frac{\partial T}{\partial x} \right) + \frac{\partial u}{\partial t} \rho_w G_m H_m, \qquad (1)$$

where x is distance along the flow direction (m), t is time (s), ρ is wood density (kg m⁻³) as a function of the moisture content u, c_p is specific heat capacity of wood (J kg⁻¹ K⁻¹) as a function of temperature and moisture content u (kg kg⁻¹), T is temperature (K), λ is wood thermal conductivity (W m⁻¹ K⁻¹) expressed as a function of temperature and moisture content u, ρ_w is water density (kg m⁻³), G_m is wood specific gravity (kg kg⁻¹) and H_m is latent heat of moisture in wood (J kg⁻¹).

The specific gravity of wood G_m is the ratio of the density (mass of a unit volume, i.e. oven-dry mass of wood) of a substance to the density (mass of the same unit volume, i.e. mass of water) of a reference substance. The moisture content n in the wood material is expressed as the weight of the water present in the wood divided by the weight of the oven-dry wood substance.

The governing equation for the unsteady state isothermal moisture transfer through a wood board is given as Fick's second law (Crank, 1956) in one dimension:

$$\frac{\partial u}{\partial t} = \frac{\partial}{\partial x} \left(D_t(T, u) \frac{\partial u}{\partial x} \right), \tag{2}$$

where D_i is the transverse wood moisture diffusion coefficient (m² s⁻¹).

It is possible to simplify a coupled model and turn it into an uncoupled model on the assumption that there is no heat generation inside the wood. This assumption can approximately be implemented on certain conditions of an experiment, i.e. in case the velocity of heat transfer process through the wood sample is over ten times higher than the velocity of mass transfer, i.e. the diffusion process. Another simplifying assumption would be the use of empirical formula for thermo-physical properties of wood and other non-linear transfer coefficients (Younsi et al., 2006).

In exceptional cases the isothermal diffusion equation (i.e. Fick's second law) can be used to describe the process of drying wood in a narrow temperature range 50–60°C with reasonable accuracy, ref. Eq. (2). The prerequisite for using the extremely simplified diffusion-based model is presence of as many reference points for comparison as possible to observe the dynamics of moisture content and temperature. Such drying schedule resulting in a parabolic moisture content distribution in the cross-section of the material perpendicularly to the surface was named quasi steady-state by Luikov (1966). In this case Fick's second law, Eq. (2), can be presented in the following form:

$$D_t \frac{\partial^2 u}{\partial x^2} = const , \qquad (2a)$$

The solution of its differential equation is a square root function.

Upon experimental determination of the diffusion coefficient it is very important to fulfil the assumptions of isothermal diffusion. Isothermal properties were checked by constant observation of differences in the temperature on the surface of the sample and at different distances from the surface of the sample both inside the sample (Figure 2) and in the drying air.

The local diffusion coefficient can be experimentally determined according to Fick's first law (Fick, 1855; Salin, 1990):

$$F = -D\frac{\partial u}{\partial x} , \qquad (3)$$

where *F* is mass flux (kg m⁻² s⁻¹), *D* is diffusion coefficient (m² s⁻¹), *u* is mass concentration (kg m⁻³) and *x* is coordinate (m).

The mathematical model that is the basis for the wood drying simulation program TORKSIM ver. 3.1 has not been disclosed in detail. However, according to Salin (1990), it can be assumed to be a perfect isotropic Luikov-type coupled model.

4.4.2. Stress calculation model

In order to ensure a good quality of wood in the process of drying it is necessary to calculate on the basis of previously calculated moisture profile the strain and stress evolved in wood. The mathematical model in the one-dimensional isotropic case can be presented after Salin (1990). The primal equation for stress calculation is

$$\sigma = aE\left(\frac{\int_{0}^{1/2} E\rho_b dx}{\int_{0}^{1/2} Edx} - \rho_b\right), \qquad (4)$$

where σ is tensile stress (Pa), ρ_{b} is the content of bound water (kg m⁻³), E is modulus of elasticity (Pa), l is board thickness (m) and x is coordinate from the surface of the board (m).

The governing equation for creep calculation is as follows:

$$\frac{\partial \varepsilon}{\partial t} = \frac{1}{E} \frac{\partial \sigma}{\partial t} + \frac{\partial \varepsilon_v}{\partial t} + (a + m\sigma) \frac{\partial \rho_b}{\partial t}, \qquad (5)$$

where ε is total strain, σ is tensile stress (Pa), E is modulus of elasticity (Pa), ε_{ν} is viscoelastic strain, a is unrestrained shrinkage coefficient (m³ kg⁻¹), m is mechano-sorptive creep coefficient (m³ kg⁻¹ Pa), ρ_{b} is content of bound water (kg m⁻³) and t is time (s).

The modulus of elasticity E is not a constant, but depends on both the moisture content and temperature.

4.4.3. Material

In this thesis the data of computer simulations of two industrial experiments and one laboratory experiment of convective drying of pinewood using the 1D program TORKSIM ver. 3.1 (Trätek, 2006) are presented. Also, the approximate inverse determination of efficient diffusion coefficients is demonstrated using the parabola method well known from simulated moisture profiles (Kretchetov, 1972). The diffusion coefficient in this context is defined as the effective diffusion

coefficient of the total diffusion flux of the liquid and vapour phases and free water and bound water.

4.4.4. Experiment

In the industrial experiments samples of pinewood (length 6 m, dimensions of the cross-section 150 mm x 22 mm and 150 mm x 50 mm, sawing pattern 4EX-log), located in the middle of the pile, were dried in an industrial kiln. For the laboratory drying experiment the samples of pinewood were 600 mm in length and had cross-sections of 200 mm x 56 mm. In all three experiments the average content of heartwood was $40\% \pm 10\%$. In the laboratory experiment the ends and sides of the wood samples were covered with neutral silicone (a product of Bostik) to ensure the validity of the assumptions of the one-dimensional mathematical model in the experiment.

In the laboratory drying test the diffusion coefficients were determined experimentally by means of the electrical resistance method using Fick's first law (Fick, 1855). In addition, differences in the temperature of the material subjected to drying and the initiation of the first drying crack as a result of drying stress were examined. For this purpose, a forced drying schedule three times shorter than the regular schedule used in industry was used.

In the industrial chamber-type convective dryer a reliable mild drying schedule that had been successfully tested in practice was used. Such a schedule ensures a high final quality of wood. The drying time of the 22 mm sample was 90 hours and of the 50 mm samples 336 hours. The initial moisture contents were $55 \pm 10\%$ and $36 \pm 10\%$, respectively.

The location of the different sensors on the surface and near the surface of the sample in a single specimen used in the laboratory experiment is shown in Figure 2.



Figure 2. Location of sensors in the pine wood specimen in the laboratory experiment.

(TC – thermocouples: TC0 in the air, TC1 under the surface, TC9 9 mm from the surface, TC28 28 mm from the surface; MS4.5, MS.9 and MS28 – moisture content of sensors 4.5 mm, 9 mm and 28 mm from the surface, respectively; DS – displacement sensor; TA – thermo-anemometer; SC – silicone coating).

Differences between the temperatures in the sample, on its surface and in the ambient air were registered by a thermocouple AHLBORN (type FTA 3901, resolution 0.1 K) and the data were saved using a ninechannel data logger AHLBORN ALMEMO 2890-9. The locations of five thermocouples were 10 mm from the surface of the sample in the air, on the surface of the sample and at depths of 4.5 mm, 9 mm and 28 mm in the sample. The MC of wood was measured from the same depths using AHLBORN timber moisture sensors (type FHA 636M) and the data were saved by the data logger. Also, at least twice in 24 hours the MC of wood was measured manually using a moisture measuring device GANN HYDROMETTE HT 85T from the depths of 4.5 mm and 9 mm from sapwood (board thickness 56 mm) and 28 mm from heartwood. The velocity of air was registered by an AHLBORN thermo-anemometer of type FVA645 TH2 at 10 mm from the surface of the sample. The strain in the surface layer of the sample and the time of the initiation of the first drying crack were registered by the data logger and a displacement sensor of type FWA 025T with the resolution of 0.001 mm.

The accuracy of the timber moisture sensors used in the experiment was $\pm 2\%$ (the accuracy depends on MC). For GANN HYDROMETTE HT85T, used for manual measurement of the MC of the wood, the accuracy data are not presented in the manual. However, the instrument operates on the principle of electric resistance of wood and thus, the accuracy could be considered to be the same as that of AHLBORN sensors, i.e. $\pm 2\%$. In the experiment the moisture flux and gradient were measured with the same sensor and because the diffusion coefficient was calculated as the quotient of these two indications the accuracy of the determination of the diffusion coefficient is ca $\pm 4\%$. According to Gann (2011), the accuracy of resistance-type moisture sensors decreases above the FSP compared to the accuracy below the FSP.

4.5. Statistical analyses

The testing method involves producing a statistical prediction model for a specific tree species and specimen dimensions, given the drying schedule and the average moisture content range of the specimen. Statistical calculations were performed in Excel (Microsoft Corp., USA). The regression diagnostics of the models was assessed with tests on the normality of regression residuals (Kolmogorov–Smirnov and Shapirio–Wilk tests) and visually by using the probability paper.

The simple regression analyses were done in the statistical software environment of R (2011).

5. RESULTS

5.1. Study of electrode effects (III)

Figures 5 and 7 in (III), which depict the time dependence of the total electrical resistance (figure 5) and the time dependence of the resistance of the wood-electrode contact (figure 7) seem to have a relatively similar shape. Based on this similarity, a hypothesis may be proposed that the resistance method owes its moisture sensitivity mainly to the resistance of the electrode/wood contact.

The figures given in tables 1 and 2 of (III) allow estimating the ed average moisture sensitivity of different measuring processes may be determined. An about 50% increase in wood MC (from 97.2% up to 146%) brought about a corresponding increase in the depolarization II

initial voltage relation, $\frac{U_1}{U_0}$ 100%, a four-fold increase in the relative

increment of polarization resistance $\frac{R-R_0}{R_0}$ 100%, Fig. 3) and an

eight-fold increase in the measured electrical resistance R_0 . Thus, the measurement of electrical resistance seems to involve the highest moisture sensitivity, followed by the measurement of the relative increment of polarization resistance, and lastly, the measurement of depolarization initial voltage relation. In the practical use of the methods for measuring wood MC, the moisture sensitivity of a method is not the only selection criterion. It is also essential to minimize the variance of the measurement data recorded. A more detailed research of moisture sensitivity and variance would require more measurement data.



Figure 3. Example of moisture sensitivity of the polarization process: Relative increment of polarization resistance in pine sapwood at three different moisture contents: MC1 = 146%; MC2 = 97.2% and MC3 = 36.4%

Figure 11 in (III) shows that residual polarization voltage between measuring electrodes is relatively stable (average 28.5 mV), but suggests a downward trend as wood MC decreases. Residual polarization voltage may lead to a systematic measurement error when measuring wood resistance; for example, at an input measuring voltage of 3 V of the resistance meter, it depends on the measuring voltage polarity up to $\pm 1\%$, and at a measuring voltage of 1 V up to $\pm 3\%$ because the resistance scale has a linear connection with the input voltage.

5.2. Assessing James's hypothesis (H1) (IV)

5.2.1. Results of testing pine sapwood

Measurement data from (Tamme et al., 2012) (see figure 4 in (III)) were used to determine the polarization current in the polarization (or wood charging) process, the time dependence of which is given in figure 2 in (III). The polarization current of the pine sapwood specimen was determined at an average moisture content of 146% and temperature of 20°C.

The polarization process of the specimen was examined in more detail for 20 seconds starting from the beginning of the process (that is, the initial phase of the polarization process). The observation period of 20 seconds was selected because the resistance meter Scanntronik (Scanntronik, 2013) uses the measuring cycle of the same duration. Since the theoretical equation (10) in (IV) fails to approach a 20-second polarization process with sufficient accuracy, the empirical equation

$$I = I_0 \exp(\frac{-t}{\tau_0 + 0.17t}) , \qquad (6)$$

where $\tau_0 = 5.15$ s and $I_0 = 65.81 \,\mu\text{A}$ were used, was found to be a more accurate approximation of the process. Graphs showing the experimentally determined polarization current, the polarization current calculated with the theoretical equation (10) in (IV) and the polarization current calculated with the empirical equation (12) are given in figure 3 in (IV) in the same scale. The dependence of the time factor $\tau = \text{RC}$ (see equations 10 and 12) of the polarization process on time is given in figure 4 in (IV). The figure shows that the dependence of the time factor on the polarization time, from the starting moment, is practically linear (R-squared 0.99). Taking into account that polarization resistance had been experimentally determined by direct measurement, the time factor definition equation enables to determine also the dependence of the summarized electrical capacitance *C* of 146% MC pine sapwood and of a double layer of electrodes on time, which is shown in figure 4 in (IV).

Physical considerations make it obvious that neither polarization resistance R, polarization capacitance C nor the time factor τ can increase limitlessly; these quantities must approach asymptotically a certain limit value or plateau.

Pine sapwood depolarization process graphs at 20°C are presented in (Tamme et al., 2012) (see figure 9 in (III)). A more detailed analysis of the depolarization process at 146% MC (i.e. graph corresponding to MC1 in figure 9 in (III)) allowed constructing the following empirical equation:

$$U = U_0 \exp(\frac{-t}{\tau_0 + 0.6t}) , \qquad (7)$$

where $\tau_0 = 4.685$ s and $U_0 = 0.411$ V for the 20 seconds of the initial phase of this process, which approaches the time dependence of depolarization voltage better than the theoretical equation (9a) in (IV). The experimentally determined depolarization voltage U, voltage calculated with the theoretical equation (9a) and depolarization voltages calculated with the empirical equation (7) for 146% MC pine sapwood are given in the same scale in figure 5 in (IV). Figure 6 in (IV) shows the dependence of the time factor of the same depolarization process (the one in figure 5) on time, which proved practically linear in the initial phase of the depolarization process. Physical considerations make it obvious that also the increase in the time factor of the depolarization process needs to slow down, and the time factor has to asymptotically approach a certain limit value or plateau. As the depolarization current was not measured in (Tamme et al., 2012), finding the summarized electrical capacitance C of wood and electrodes based on the found time factor unfortunately deemed impossible in the depolarization process.

5.2.2. Results of testing black alder

To examine wood polarization (electrical charging) and depolarization (electrical discharging) processes and improve the measuring methodology a few experiments were carried out involving another tree species (black alder *Alnus glutinosa*) at 90% MC and temperature of 20°C. The general shape of the polarization–depolarization measuring cycle in the voltage graph for black alder is given in figure 7 in (IV). The transition from the polarization phase to the depolarization phase shown in figure 7 involves a voltage drop or the Ohmic drop, which can be described with equation (9) in (IV). Figure 4 shows the dependence of polarization current on time, which was measured directly with the electrometer E_1 . Figure 9 in (IV) shows the dependence of polarization resistance on time for black alder (90% MC and 20°C).



Figure 4. Dependence of the polarization current on time, measured with the electrometer E_1 for black alder (90% moisture content and 20°C).

During the measurement switches S_1 and S_2 were closed and S_3 was in the off position (see Figure 1). Figure 5 shows the two consecutive measurements of depolarization current and depolarization voltage, which are depicted in a single scale. During these measurements, switch S_1 was open and switches S_2 and S_3 were in the closed position.


Figure 5. Two consecutive measurements of the depolarization current and depolarization voltage for black alder (90% moisture content and 20°C), depicted in a single scale.

Based on the depolarization voltage and depolarization current measurement data, figure 11 in (IV) shows the dependence of the electrical resistance of black alder calculated according to Ohm's law, on time in the depolarization process. Figure 11 in (IV) shows that the electrical resistance of the black alder specimen with 90% MC increases in the initial phase of the depolarization process but later approaches a certain value. In the final phase of the process, current and voltage measurement errors also become substantial because since choosing a more sensitive measuring area for the electrometer is not possible without interrupting the process.

It may be concluded from the empirical equations 6 and 7 that there are, specifically, 20 different relaxation times for the polarization and depolarization process. However, James's hypothesis only states three relaxation times. In order to explain this formal contradiction, the depolarization voltage schedule of black alder was analysed in more detail in Figure 5, excluding the depolarization current schedule shown in the same figure for the sake of simplicity. If we were to succeed in approximating the depolarization process shown in Figure 5 in a way that the combined approximation function included a linear combination of the exponential function with three different relaxation times, James's hypothesis would be plausible. However, if it proves necessary to use the empirical equations 6 and 7 in approximation, there will be more than three different relaxation times. So, it may be presumed that the question of the number of relaxation times depends greatly on the approximation method of experimental data and the quality of the obtained approximation function. In a mathematical formulation, James's hypothesis poses the question of whether three exponent base functions and three constants are enough in the desired linear combination or should there be more of them.

5.3. Assessing Vermaas's hypothesis (H2 in ((IV)) (II, IV)

(RQ1) Calibration models for wood resistance-type moisture meters pre-calibrated by the manufacturer:

The results of testing different moisture meters along with descriptive statistics parameters are given in tables 1, 2 and 3 in (II).

Calibration models with the main parameters and model diagnostics with the results of the Kolmogorov–Smirnov and Shapiro–Wilk tests are given in Tables 1, 2 and 3.

	Meter		
Parameter	Gann HT 85T	NDT James	FMD-6
No. of	134	133	134
observations			
R-squared (R ²)	0.963	0.847	0.765
Standard error	0.04836	0.09783	0.1213
of regmodel			
<i>p</i> -value of	<i>p</i> < 0.001	<i>p</i> < 0.001	<i>p</i> < 0.001
regmodel			
$K-S^*$ test of	<i>p</i> = 0.5412	<i>p</i> = 0.4156	<i>p</i> = 0.1224
residuals, <i>p</i> -value			
Shapiro–Wilk	<i>p</i> = 0.2274	<i>p</i> = 0.0177	<i>p</i> = 3.0e-5
test of residuals,			
<i>p</i> -value			

Table 1. Modelling of moisture meters on black alder specimens. The independentvariable in the regression model is moisture meter reading (in moisture content %)

	Meter		
Parameter	Gann HT 85T	NDT James	FMD-6
No. of	134	134	134
observations			
R-squared (R ²)	0.968	0.920	0.942
Standard error of	0. 0385	0.0604	0.0514
regmodel			
K-S* test of	<i>p</i> = 0.1069	<i>p</i> = 0.7638	<i>p</i> = 0.1995
residuals,			
<i>p</i> -value			

Table 2. Modelling of moisture meters on silver birch specimens. The independent variable in the regression model is moisture meter reading (in moisture content %)

*K-S test -Kolmogorov-Smirnov test

Table 3. Modelling of moisture meters on aspen specimens. The independent variable in the regression model is moisture meter reading (in moisture content %)

	Meter		
Parameter	Gann HT 85T	NDT James	FMD-6
No. of	140	140	140
observations			
R-squared (R ²)	0.982	0.946	0.936
Standard error of	0.0361	0.0619	0.0676
regmodel			
K-S* test of	<i>p</i> = 0.4579	<i>p</i> = 0.1469	<i>p</i> = 0.09119
residuals,			
<i>p</i> -value			

*K-S test -Kolmogorov-Smirnov test

An example of the calibration model (i.e. linear regression model) for black alder wood is given in Figure 6.



Figure 6. Regression line of testing wood moisture meter Gann HT 85T on black alder specimens with - their 95% confidence and tolerance bands (LOG(MC actual %) – actual moisture content in log₁₀; Gann – meter reading (moisture content %).

(RQ2): Calibration models for calibrating a specific electrical resistance meter (type: Scanntronik Gigamodule) into a wood moisture meter are presented in Table 4.

Parameter	Model		
	Individually calibrated,	Moisture content	
	model 1)	predicting regression	
	(Figure 8)	model 2 (in situ	
		calibrated)	
		(Figure 7)	
Observations	10	60	
R-squared	0.99	0.71	
Standard error	0.26	0.84	
of regmodel			
Kolmogorov-	_	<i>p</i> -value = 0.6558	
Smirnov test			
of residuals of			
regmodel			
Shapiro–Wilk	_	<i>p</i> -value = 0.8622	
test of			
residuals of			
regmodel			

 Table 4. Regression parameters for model 1 (individually calibrated pine sapwood)

 and model 2 (in situ calibrated black alder)

Excluding the 20 measurements carried out below the FSP (26% MC) and shown in figure 12 in (IV) resulted in a simpler linear calibration model for the 60 measurements performed above the FSP in a logarithmic scale: *x*-axis 10LOG(R) and *y*-axis 10LOG(MC) (Figure 7), the main parameters of which are the following: $R^2 = 0.71$, SE of the calibration model is 0.84, *p*-value of the calibration model is smaller than 0.01.

When comparing the main parameters of the calibration model in the range above the FSP shown in Figure 7 and the individual calibration model given in Figure 8, a significantly better quality of the individual calibration model is apparent than in the case of the parameters of the in situ or the so-called collective calibration model. In the case of the individual calibration model, the R-squared value of 0.99 and the value of the residual standard error SE = 0.26 are achieved with only 10 measurements, whereas in the case of the in situ calibration model, the R-squared value of 0.71 and the value of the standard error SE = 0.84are obtained with 60 measurements. It may be concluded from the comparison that for the purposes of the standard ISO/IEC 98-3:2008 both models have the measuring function, i.e. the calibration function, but in the first case (the so-called in situ calibration model) we are dealing with type A evaluation of measurement uncertainty. In the second case (i.e. individual calibration), type B evaluation of measurement uncertainty, that is, a classical reference-based calibration procedure is possible.

A pine sapwood specimen passing various average MC levels during slow drying was selected as a reference model for moisture content in paper (III). The average MC of the specimen was determined with a highly reliable dry-weighing method according to the standard ISO 3130:1975. Both cases can be viewed as exceptions of Vermaas's hypothesis due to the existence of calibration functions.

A simple geometric interpretation of the VH circumstances was made when the calibration model obtained with 60 measurements above the FSP was presented with the signal-to-noise ratio (Figure 7). Figure 7 clearly shows that the noise signal (N1) constitutes approximately 2/3 of the useful signal (S). The tolerance interval of the model was interpreted as the noise signal N1 and the absolute increment of the electrical resistance logarithm within the entire area of applicability of the model was interpreted as the useful signal S.



Figure 7. Simplified regression line for the black alder specimen obtained with 60 measurements at 50°C according to the regression curve given in figure 12 of paper (IV) by excluding 20 measurements below the fibre saturation point. S – useful signal; N1 – noise signal via the tolerance interval; N2 – noise signal via the SE of the calibration model.

For comparison, the individual calibration model of the measuring electrodes can also be presented as the signal-to-noise ratio (figure 12 in (III) and Figure 8).



Figure 8. Example of the individual calibration function between the average moisture content logarithm and wood electrical resistance logarithm (figure 12 in (III)) when the average moisture content of the pine sapwood specimen decreased from 141% to 36.4%. S – useful signal; N1 – noise signal via the tolerance interval.

The noise signal N1 constitutes approximately 1/10 of the useful signal S as may be visually determined in Figure 8 for the individual calibration model of the measuring electrodes. Therefore, the signal-to-noise ratio is considerably better for a high-quality calibration model (Figure 8) than for a low-quality calibration model (Figure 7). So, the final conclusion could be the following: Increasing the number of measurements does not significantly improve the signal-to-noise ratio of the low-quality calibration model, but it may considerably improve (that is, in the case of good convergence of the model) the SE of the model.

In the case of a large number of measurements, the standard error SE of the calibration model may be interpreted as the noise signal N2 instead of the noise signal via tolerance interval (N1). Therefore, the noise signal N2 obtained on the basis of the standard error of the model compiled for the 60 measurements (Figure 7) constitutes approximately 1/10 of the useful signal S. So, as the number of measurements increases, the signal-to-noise ratio can be significantly improved because the increasing number of measurements does not influence the useful signal but it does decrease the noise signal N2.

5.4. Equivalent circuit of the PD method (III, IV)

Electrical substitute circuit diagrams are used for modelling complex real electrical processes. Based on the information gathered in papers (III) and (IV), an equivalent circuit (Figure 9) may be proposed for the PD method for the first 20 seconds (polarization phase):



Figure 9. Equivalent circuit of the polarization-depolarization method.

Wood resistance R_{wood} is constant, but wood electrical capacitance $C_{\text{wood}}(t)$ and electrode/wood contact resistance $R_{\text{contact}}(t)$ are variable quantities the first of which $(C_{\text{wood}}(t))$ varies according to the linear function and the second $(R_{\text{contact}}(t))$ according to the logarithmic function.

This equivalent circuit is essentially a modified Debye circuit (Debye, 1945) in which all elements are no longer constant compared to the original circuit.

5.5. Determination of relative chargeability of wood with the PD method (IV)

It is possible to determine the relative chargeability of black alder wood (Figures 4 and 5) by comparing the electrical energy that has passed through the wood and the electrical energy that has been retained in the wood. The electrical energy W that passed through wood during the last second of the polarization process $W_{pol} = I_{20,pol}U_{20,pol}t_{20}$ and the electrical energy measured during the first second of the depolarization process $W_{dpol} = I_{1,dpol}U_{1dpol}t_1$ were compared. Considering that the time period for calculating both energies is 1 second, the corresponding electric power ratio was found:

 $\frac{W_{dpol}}{W_{pol}} = \frac{P_{dpol}}{P_{pol}}$, which also represents wood relative chargeability. By substituting corresponding numerical values of current intensities and voltages from Figures 4 and 5, the approximate wood relative chargeability found was 6%. Thus, only 6% of the energy passing through wood during the last second of charging is retained in wood. Since according to paper (III), both the polarization process and the depolarization process are moisture-sensitive, it may be presumed that wood relative chargeability depends on wood average moisture content.

The relative chargeability of wood probably depends also on the selected measuring interval. The reason behind this is the high rate of discharging of the double layer of the wood/measuring electrode in the initial phase of the depolarization process. In other words, wood loses significantly more retained electrical charge within 1 second than within 0.01 seconds.

5.6. Determining the significant parameters of wood drying (I)

The graphs of log files of the results of industrial drying experiments and relevant simulations with the program TORKSIM ver. 3.1 are shown in Figure 10.



Figure 10. Graphs of the log file and simulation of the results of the industrial drying experiment of pine wood (measurements based on electrical resistance, simulated with TORKSIM ver. 3.1)

For 50 mm material (Figure 10B) the formula of linear regression was: MC(m) = 0.886608 + 0.853483MC(s),

Multiple *R*-squared: $R^2 = 0.9919$,

Comparison of the MC(m) and MC(s) gave the standard deviation of 2.6%.

Maximum differences in the temperature on the surface of the sample and inside the sample did not exceed ± 0.7 °C (figure 4 in (I)). Also TORKSIM ver. 3.1 shows differences in the temperature on the surface of the sample and in the drying air near the surface of the sample. There was a good match with the differences in temperature measured in the course of the experiment (figure 4 in (I)).

The processing of the simulation results of industrial experiments with the program TORKSIM ver. 3.1 (22 mm pine 91 hours and 50 mm pine 336 hours) showed a strong correlation with the quasi-stationary drying regime, the values of R^2 varied within the range $R^2 = 0.991-0.9999$ for the 22 mm board and $R^2 = 0.9939-0.9998$ for the 50 mm board (see figure 5 in (I)).

The correlation with the quasi-stationary drying regime was weaker in the case of the forced drying regime in the laboratory experiment of drying a 56 mm pine board for 121 hours. The processing of the simulation results demonstrated variation of R^2 in the range of $R^2 = 0.9821-0.9885$. Measured and simulated local MC at depths of 4.5 mm, 9 mm and 28 m from the surface of the board were compared in the laboratory experiment using the forced drying regime. The results are shown in figure 6 in (I).

According to the simulation results, the moisture content can also be determined by the first time derivate. These data enabled the approximation of the inverse determination of the effective diffusion coefficient by means of the parabola method (Kretchetov, 1972), well known from Fick's second law. Also, it was possible to define the dependence of inversely determined effective diffusion coefficients on the average MC of wood shown in figures 7 and 8 in (I).

Figure 11 shows the dependence of the diffusion coefficient of the approximate inverse determination of TORKSIM simulation and the

local diffusion coefficient determined directly by the laboratory experiment from the mean wood MC.



Figure 11. The dependence of the effective diffusion coefficient on the inverse determination (I.D.) of TORKSIM simulation (I.D. 50 mm and I.D. 22 mm) and the local diffusion coefficient determined directly by the laboratory experiment from the mean moisture content of the pine wood.

The first crack in the 56 mm thick pinewood sample emerged 82 hours after the initiation of drying. The computer simulation of the laboratory experiment indicated maximum relative tensile stresses of 0.33 units at 80–90 hours, indicating the risk of the initiation of drying cracks. Distributions of tensile and compressive stress at a defined moment of time (75, 82 and 121 hours from the start of the drying process) were found in a simulation in the laboratory experiment. The result is shown in figure 10 in (I).

6. DISCUSSION

6.1. Electrode effects (III, IV)

There are a number of ways to look at electrode effects. On the one hand, it may be assumed that all electrode effects are detrimental because they cause various measuring errors when measuring wood electrical resistance above the FSP. If the purpose is to measure only wood electrical resistance, eliminating all electrode effects would probably be the right solution. On the other hand, however, if electrode effects are used with the aim of solving a specific research problem, it would be expedient to describe electrode effects mathematically as accurately as possible instead of neglecting them. An example of this might be the application of the ESIR (or PD) method in the experimental test of James's hypothesis. Eliminating electrode effects is actually quite simple. In the time domain, measuring should be done quicker (within the measuring interval of 0.01–0.3 seconds), while also limiting the measuring voltage (below 2.6 V) and the general measuring time (max 1 second). These measures guarantee that polarization processes do not fully develop in the double layer of the electrodes, or if they do, at least their development direction can be monitored. Further extraction of the polarization process should be done mathematically. When measuring in the frequency domain, the measuring voltage should be even lower (50 mV and below), and the DC voltage component should be left out, because it could cause additional polarization. A certain DC component (ca 210 mV) may be added in the measuring system with the Ag/AgCl reference electrode (RE), which may also cause additional polarization.

In order to eliminate the residual polarization effect, the measuring interval between two consecutive measurements from the same electrodes should be long enough (1 hour and longer). To avoid corrosion of the measuring electrodes, non-corroding electrode materials, such as graphite, glass-carbon or bismuth, should be used. For avoiding the occurrence of electrolysis effects (e.g. deposits on electrodes and exhaust gases) optimum current densities should be used in the experiments (Romann et al., 2014).

The most important electrode effects include wood polarization and depolarization. Sometimes the wood polarization process is also called polarization/capacitance effect in the literature (Chaumat et al., 1999). Concerning the numerical value of electrical capacitance, in the case of 146% MC in pine sapwood, the electrical capacitance measured with the PD method at the start-up moment was 0.5 mF. This may be considered a relatively high capacitance, approaching the lower limit of typical capacitances of supercapacitors and pseudocapacitors (Zuleta, 2005). Therefore, this raises the question of a certain analogy with wet wood and a supercapacitor or pseudocapacitor. Zelinka et al. (2007) showed that in the frequency domain, wood can be described well with the Debye equivalent circuit containing a constant phase element (CPE). The CPE and pseudocapacitor transfer equations are given as examples (AUTOLAB, manual; http://www.ecochemie.nl). The electrical capacitance lower than that of a supercapacitor and conformity between the CPE model and experimental data support the hypothesis of the analogy between wet wood and a pseudocapacitor. Such approximate analogies sometimes help specify the details of an equivalent circuit.

6.2. Testing James's hypothesis (JH) and the PD method (IV)

The number of relaxation times probably also depends on the approximation methodology of experimental curves found with the PD method. Approximation of the experimental curves found with the PD method with non-linear approximation functions needs further research.

In connection with the equivalent circuit obtained with the PD method, it should be mentioned that knowing the equivalent circuit is not entirely necessary for calculating the figures from the experimental curves, since these are found according to approximation functions and not the equivalent circuit. This might be compared to the electrical impedance spectroscopy (EIS) method (Zelinka et al., 2007) in which electrical parameters of interest, such as the real and imaginary part of wood electrical resistance and the CPE etc., are calculated based on an equivalent circuit. Thus, knowing the equivalent circuit is obligatory in the case of the EIS method.

These papers applied the PD method for experimental testing of a specific theoretical problem, namely JH. This raises a justified question: Could the PD method be somehow applied also in practice in the future? Keeping in mind the further application of the method in practice, the experimental device for the PD method has been engineered technically in a way that enables autonomous battery-powered operation during a reasonable time period (for example when measuring growing trees) and it would be available at a competitive cost price.

The main problem concerning the practical application of the PD method does not lie in the technical details of the method, but in how the measured electrical parameters can be correlated with the physical and mechanical wood parameters of practical interest, such as wood average MC, the static and dynamic modulus of wood elasticity, wood stress and deformation, wood dry mass and density. There is probably no other way to find the listed correlations of practical interest than to apply statistical modelling. Modelling, in turn, requires a large number of similar repeated measurements. However, a lot of the work is often lost in such modelling projects if it appears that no reasonable correlation occurred in the experiments. Therefore, one has to be prepared for not getting the expected results from all models.

6.3. Testing Vermaas's hypothesis (VH). (II) and (IV)

Surprisingly, the two exceptions of Vermaas's hypothesis have been long used in practice, although intuitively, without complex statistical modelling or any other form of scientific reasoning. For instance, manufacturers of wood dryers have designed control engineering in a way that it has 10 wood electrical resistance measuring channels. This has probably been done becasue intuitively it has been understood that the standard error is smaller in the case of ten average measurements compared to a single measurement. So, engineers did, in fact, covertly presume the existence of some sort of calibration model, although the VH denied it. These statements have clearly described the principles of producing calibration models related to the first exception of the VH, so there is no need to covertly presume anything. In wood drying practice, the calibration model associated with the first exception of the VH enables the wood dryer operator to plan the desired measuring accuracy with the number of measurements. Sometimes additional measurements are necessary for determining wood average MC with greater accuracy at the beginning of drying. Paper (II) pointed out the possibilities of applying the first exception of the VH in non-destructive ultrasound measurements of wood (see Wang et al., 2004; Carter et al., 2005).

Another practical example makes use of individual calibration of resistance-type pin electrodes for monitoring wood MC dynamics above and below the FSP (Lazarescu et al., 2010) simultaneously in wood surface and core. Therefore, the researchers intuitively presumed the existence of the corresponding individual calibration function (or, in our terms, the existence of the second exception of the VH), although the VH denied it. Therefore, in the given example, the researchers must have covertly presumed also an excellent time stability of the individually calibrated pin electrodes, because otherwise reliable monitoring of wood MC dynamics would not be possible. The author of these statements has, in fact, tested the time stability of the individually calibrated corrosion-free carbon electrodes. but unfortunately, the results could not be presented in this thesis due to lack of space. The mentioned stability test lasted for more than 5 days.

In relation to pointing out the two exceptions of the VH in these statements, the justified question of whether there are more exceptions is raised. The author of the statements has conducted a few experiments to determine the existence of a third exception of the VH and received some interesting results, which, unfortunately, could not be presented in this thesis due to lack of space.

6.4. Determing the significant parameters of wood drying (I)

The accuracy of experimental determination of the diffusion coefficient in a given coordinate point was largely dependent on the resolution capacity and time stability of the measuring device. As the gradient itself is a function of the coordinate, the accuracy was also dependent on the size of the measured sample. When comparing the accuracy of the experimental determination of the diffusion coefficient with the method of computed tomography ($\pm 3.6\%$) (Danwind, 2005) and resistance method ($\pm 4\%$), it appeared that the difference in accuracy was not significant. The achieved accuracy can be considered satisfactory for diffusion-based control of wood drying.

Comparison of the measuring data of the laboratory drying experiment with computer simulation showed a relatively good match between the measured and the simulated moisture content in the layer near the surface of the sample. However, the MC in the middle part of the sample measured at the end of the experiment was considerably higher $(39\pm10\%)$ than in case of simulation (18%). Probably the MC in the middle of the sample was measured with lower accuracy, as the accuracy of resistance-type moisture sensors decreases above the FSP.

The results of the study indicate that there is a good correlation between the MC of wood measured during the industrial experiment and the results of computer simulation. It was proved that by the method of electrical resistance the effective diffusion coefficient dependent on the average MC can be used as an alternative to the oven-dry method.

The experience gained in the course of the study showed that the electric devices for measuring MC can be used for experimental determination of the local diffusion coefficient according to Fick's first law.

7. CONCLUSIONS

The papers on which the present thesis is based study the practical application possibilities of the electrical resistance method for determining wood MC above the FSP. Vermaas's hypothesis (VH) clearly gives a negative evaluation of the practical application possibilities of the electrical resistance method for wood MCs above the FSP (that is, above 30% MC), stating that the method lacks the calibration function in the given MC range. Without the calibration function, however, the measurement method can obviously not be applied in practice. In order to address the problem, the circumstances of the VH were statistically modelled in two typical cases. In the first case research question, RQ1, the resistance meters had been precalibrated as wood moisture meters. In this case, recalibration could presumably ensure a more reliable calibration function and naturally also show whether the calibration function even exists. In the second case (RQ2), the procedure of the calibration of a specific electrical resistance meter into a wood moisture meter was modelled and the main parameters of the obtained calibration model were studied. Modelling results were interpreted according to the principles of the standard ISO/IEC 98-3:2008.

1) It was found that the standard error of the calibration models developed for the case of both RQ1 and RQ2 decreases as the number of measurements increases. This phenomenon of the standard error of the calibration model decreasing as the total number of measurements increases was called the first exception of the VH in these papers. The Discussion chapter presented possibilities of applying the first exception of the VH in practice, for example in wood drying. Paper (II) pointed out the possibilities of applying the first exception of the VH in non-destructive ultrasound measurements of wood MC.

2) It was found that a calibration function with a sufficiently good quality existed in the individual calibration of the measuring electrodes in the MC range above the FSP, which enables the calibration function also with the method of type B evaluation of measurement uncertainty according to the standard ISO/IEC 98-3:2008, that is, the traditional calibration procedure based on the reference moisture. This situation was called the second exception of the VH in these papers. As shown

in this thesis, the second exception of the VH plays an important role in monitoring wood drying.

3) In order to study the electrical processes with a long and extra-long relaxation time in the wood moisture content range above the FSP, the polarization-depolarization method (PD method) was developed. The PD method along with the empirical equations found enabled us to experimentally test James's hypothesis on the multiplicity of relaxation times in wood. An electrical equivalent circuit of the PD method was also found for the wood polarization phase. The method was applied to determine the relative chargeability of black alder wood in case of the average MC of 90%.

4) In relation to wood drying the experimental diffusion coefficient determined according to Fick's first law at various depths from the wood specimen shield proved to be an extremely informative parameter for describing the drying process. The experimental diffusion coefficient (also called the local diffusion coefficient in some literature sources) enables quantitative separation of ranges of slower moisture diffusion and faster moisture diffusion existing in a specific drying plan. This, in turn, seems to allow preferring ranges of faster diffusion when optimizing the drying plan and, if possible, ignoring ranges of slower diffusion.

Fick's second law was applied to find the effective diffusion coefficient that describes the drying process in general. This coefficient is rather a quantity that describes the drying process on average, but still allows determining whether a specific drying plan is soft or rigid.

A few fairly conceptual issues of wood science pointed out in these papers, such as Vermaas's hypothesis (VH) and James's hypothesis (JH), need further in-depth research for developing the theoretical aspects as well as improving the experimental methodology:

- The existence (or non-existence) of <u>the third exception of the</u> <u>VH</u> would need reliable experimental verification.
- The further theoretical analysis of the JH would presume derivation of differential equations describing the processes of wood polarization and depolarization. In relation to the practical applicability of the JH and the PD methods, it would be necessary to enhance the methodology of obtaining approximation functions for the experimental curves.

Concerning a more detailed experimental study of the JH, the development of a new method combining the Wenner method and the PD method should be considered.

The methodology for determining the local diffusion coefficient according to Fick's first law described in these papers will be applied in the future to study the industrial drying of deciduous tree species such as birch, aspen and alder, which are important deciduous trees in Estonia. Information regarding the ranges of maximum values of the experimental local diffusion constant near the wood surface, namely approximately at a depth of 2.5 mm from the surface, would be of the greatest interest.

These papers also presented two new hypotheses: first, the hypothesis about the existence of the third exception of the VH; second, the hypothesis on the existence of a statistical correlation between the parameters of raw data approximation functions with the PD method and a parameter that macroscopically describes wood (such as MC, density, dry density, deformation, mechanical stress, modulus of elasticity etc.).

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SUMMARY IN ESTONIAN

ELEKTRILISTE TAKISTUS-TÜÜPI KONTROLLIMEETODITE ARENDAMINE PUIDUKUIVATUSES

Sissejuhatus

Puidukuivatuses on levinud puidu niiskusesisalduse mõõtmine elektrilisel takistusmeetodil, sest see meetod on odav ja enamasti töökindel. Puuduseks on asjaolu, et puidukiu küllastuspunktist alates (niiskusesisaldus \geq 30%) hakkab mõõtetäpsus kahanema korrelatsioonis puidu niiskusesisalduse suurenemisega.

peamine eesmärk oli puidukuivatuse Uurimuse täiustada kontrollimeetodeid, kasutades selleks traditsioonilise takistusmeetodi Takistusmeetodi arendamine juhul, võimalusi. kui puidukiu niiskusesisaldus ületab küllastuspunkti, eeldab eksperimentaalset ja teoreetilist uurimistööd. mis lähtub Vermaasi ning Iamesi hüpoteesidest, puidu makro- ja mikrofüüsikalistest parameetritest ning dielektrikute elektrilise polarisatsiooni teooriast.

Uurimuse peamised hüpoteesid olid järgmised.

- Kui puidu niiskusesisaldus ületab puidukiu küllastuspunkti, kehtib üldiselt Vermaasi hüpotees puidu takistus-tüüpi niiskusmõõtjate kalibreeritavuse kohta, kuid on võimalikud ka mõned erandid.
- Jamesi hüpotees relaksatsiooniaegade paljususe kohta puidus kehtib ka ajaskaalal, näiteks elektrilise üksikimpulsi mõõtmisel reaktsioonimeetodi ehk polarisatsiooni-depolarisatsiooni (PD) meetodiga.

Püstitatud hüpoteeside eesmärk oli matemaatiliselt kirjeldada ja analüüsida elektroodefekte, mis kaasnevad puidu niiskusesisalduse mõõtmisel elektrilise takistusmeetodiga, kui puidukiu niiskusesisaldus ületab küllastuspunkti.

Väitekirja eesmärgid olid järgmised.

- Leida võimalusi elektrilise takistusmeetodi üldtuntud nõrkuste kompenseerimiseks ja meetodi kalibreeritavuse parandamiseks niiskusesisaldusel üle puidukiu küllastuspunkti (II, III ja IV artikkel).
- Leida PD-meetodi jaoks rahuldav elektriline aseskeem (III ja IV artikkel).
- Leida puidukuivatusprotsessis võimalusi eksperimentaalse lokaalse difusioonikoefitsiendi määramiseks elektrilisel takistusmeetodil Ficki esimese seaduse alusel (I artikkel).

Materjal ja metoodika

Puidukuivatuse oluliste parameetrite määramisel kasutatud materjal ja metoodika on esitatud I artiklis. Kaht 6 m pikkust männipuidust katsekeha ristlõikepindalaga 150 x 22 mm ja 150 x 50 mm kuivatati tööstuslikus kuivatis ning üht samast puidust katsekeha mõõtmetega 600 x 200 x 56 mm (pikkus x laius x paksus) kuivatati labori kuivatuskambris. Kõigis kolmes katses oli katsetükkide lülipuidusisaldus 40%. Laborikatses olid katsekeha otsad ja küljed kaetud neutraalse silikooniga, et tagada ühemõõtmelise matemaatilise mudeli eeldusi.

Vermaasi hüpoteesi uurimisel kasutatud materjal ja metoodika on esitatud II, III ja IV artiklis. Vermaasi hüpoteesi modelleerimiseks puidu eelkalibreeritud niiskusmõõtjatega kasutati sanglepast, arukasest ja harilikust haavast katsekehasid mõõtmetega 100 x 60 x 60 mm (pikkus x laius x paksus). Katsetes kasutati juhtivate tootjate takistustüüpi eelkalibreeritud niiskusmõõtjaid Gann HT 85 T, FMD-6 ja NDT James. Kokku tehti ligikaudu 1300 üksikmõõtmist, mille alusel koostati Vermaasi hüpoteesi modelleerimiseks üheksa kalibreerimismudelit.

Elektroodefektide uurimisel kasutatud materjal ja metoodika on esitatud III artiklis. Elektroodide ja puidu vastasmõju uurimisel kuivatati männi maltspuidust katsekeha mõõtmetega 100 x 60 x 60 mm (pikkus x laius x paksus) kuivatuskambris temperatuuril 32 °C suhtelise õhuniiskuse 96% juures 244 tundi. Katsekeha niiskusesisaldus kuivkaalu suhtes vähenes kuivatamise käigus vahemikus 146% kuni 36,4%. Polarisatsiooni takistuse mõõtmiseks kasutati AlphaLab Inc. takistusmõõtjat mõõtepiirkonnas 0–20 M Ω üldise mõõtetäpsusega ± 2%. Jamesi hüpoteesi uurimisel kasutatud materjal ja metoodika on esitatud IV artiklis. Katsetes kasutati ühemõõtmelise niiskusprofiiliga sanglepapuidust katsekeha mõõtmetega 500 x 120 x 35 mm (pikkus x laius x paksus). Polarisatsiooni- ja depolarisatsiooniprotsessi katseandmeid lähendavate empiiriliste valemite koostamisel kasutati III artiklis täiendavalt töödeldud katseandmeid. Pinget mõõdeti Ahlborni 2,6 V alalispinge voltmeetriga, mille sisendtakistus oli 10 M Ω . Voolu mõõtmiseks kasutati Keithley elektromeetrit (mudel 6517B).

Tulemused

Puidu elektritakistuse mõõtmise käigus uuriti tekkida võivaid mõõtmisvigu, arvestades elektroodefekti, kui niiskusesisaldus ületab puidukiu küllastuspunkti. Leiti, et puidu niiskusesisalduse vahemikus 30–120% võib polarisatsioonitakistuse suhteline juurdekasv ulatuda kuni 120%-ni.

Uuriti ka erinevate elektroodefektide suhtelist niiskustundlikkust. Niiskustundlikkus on kõige suurem elektritakistuse mõõtmise korral, sellele järgneb polarisatsioonitakistuse suhtelise juurdekasvu mõõtmine ja sellele omakorda depolarisatsiooni algpingete suhte mõõtmine. Puidu niiskusesisalduse suurenemisel *ca* 50 % võrra (97,2–146%) suurenes depolarisatsioonitakistuse suhteline juurdekasv neli korda ja mõõdetud elektritakistus vähenes kaheksa korda.

Uuriti ka puidu jääkpolarisatsiooni nähtust. Leiti, et puidu jääkpolarisatsiooni pinge on püsiv (ligikaudu 25 mV) ega sõltu oluliselt puidu niiskusesisaldusest.

Nelja elektroodiga Wenner-tüüpi elektroodide süsteemiga leiti puidu ja elektroodi kokkupuutel võimalus takistuse ja puidu elektritakistuse eraldamiseks. Leiti, et puidu elektritakistus laias niiskusesisalduse vahemikus üle puidukiu küllastuspunkti on suhteliselt püsiv. Nimetatud asjaolu kasutati PD-meetodi elektrilise aseskeemi koostamiseks. Nelja elektroodiga mõõtesüsteemi abil töötati välja ka kvalitatiivne korrosiooniindikaatori meetod, mis võimaldab hinnata mittetasakaalulise korrosiooni esinemist mõõteelektroodidel.

Jamesi hüpoteesi uurimiseks töötati välja PD-meetod, mille abil mõõdeti kõigepealt polarisatsiooni pinget ja voolu ning seejärel vahetult pärast mõõteelektroodide pingeallikast lahtiühendamist ka depolarisatsiooni pinget. Polarisatsiooni ja depolarisatsiooni lähendusfunktsioonidele konkreetsema füüsikalise sisu andmiseks koostati protsesside esimese 20 sekundi jaoks vastavad empiirilised valemid, mis sisaldasid relaksatsiooniaega. Relaksatsiooniaja parameetri abil oli omakorda võimalik arvutada puidu elektrimahtuvuse ajalist käiku, kusjuures elektritakistuse väärtus saadi mõõtmistulemustest. Nimetatud empiiriliste valemite abil õnnestus tõestada relaksatsiooniaegade paljusus puidus, mis on Jamesi hüpoteesi sisu. Lahtiseks jäi siiski küsimus, kas relaksatsiooniaegu on kolm, nagu väidab hüpotees, või võib neid olla ka rohkem.

PD-meetodil saadud esialgsetest andmetest määrati energeetilisel meetodil musta lepa puidu (niiskusesisaldus 90%) suhteline laadumisvõime. Leiti, et polarisatsiooniprotsessi lõpus on suhteline laadumisvõime ligikaudu 6%, seega salvestub puidus ainult 6% elektrienergiast.

Puidu katseliselt leitud elektrimahtuvus ligikaudu 0,5 mF osutus üllatuslikult suureks, arvestades mõõteelektroodide väikest pindala. Sellest järeldati, et puidu mikro- ja nanopoorne struktuur (karkass) koos puidumahlaga (elektrolüüt) oli elektrimahtuvuse moodustumisel määrav. Võib oletada, et puidul on teatav analoogia pseudo- ja superkondensaatoriga.

Kasutades kogutud teadmisi elektroodefektide ja Jamesi hüpoteesi kohta, esitati PD-meetodi elektriline aseskeem.

Puidu niiskusmõõtjate modelleerimise tulemusi tõlgendati standardi ISO/IEC 98-3:2008 põhimõtete kohaselt. Leiti. et kalibreerimisfunktsioon sobib nii puiduniiskuse määramiseks kasutatava eelkalibreeritud niiskusmõõtia kui ka elektrilise takistusmõõtja puhul, kuid üksikmõõtmisel jääb see üsna ebamääraseks. Puidu niiskussisalduse üksikmõõtmise korral (niiskussisaldus $\geq 30\%$) on puidu niiskussisalduse eeldatav absoluutne mõõtmisviga vahemikus \pm (2,5–17,5%). Samas selgus, et mõõtmiste arvu suurenedes väheneb mudeli jääkviga, mis omakorda tähendab, et kalibreerimismudeli koonduvus on hea. Leiti, et 136 mõõtmise korral on võimalik, et ühe mudeli standardviga on $\pm 1.2\%$ niiskusesisaldust. Kalibreerimismudeli standardvea vähenemist mõõtmiste koguarvu kasvades nimetatakse väitekirjas Vermaasi hüpoteesi esimeseks erandiks. Hüpoteesi teine takistus-tüüpi mõõteelektroodide erand ilmnes individuaalse kalibreerimise protseduuri modelleerimisel. Leiti, et puiduniiskuse >30%) mõõteelektroodide (niiskusesisaldus individuaalsel kalibreerimisel olemas piisavalt kvaliteediga on hea kalibreerimisfunktsioon, mis võimaldab standardi ISO/IEC 98-3:2008 järgi ka B-tüüpi mõõtemääramatuste määramise meetodile vastavat ehk traditsioonilist niiskuse etaloni põhist kalibreerimist. Seda olukorda nimetatakse väitekirjas Vermaasi hüpoteesi teiseks erandiks.

Puidu kuivatamisega seoses leiti, et Ficki esimese seaduse alusel määratud eksperimentaalne lokaalne difusioonikoefitsient puidust katsekehas erinevatel sügavustel on kuivamisprotsessi iseloomustamiseks väga informatiivne parameeter. Eksperimentaalne lokaalne difusioonikoefitsient võimaldab kvantitatiivselt eraldada kuivatusplaanis esinevaid aeglasema ja kiirema difusiooniga piirkondi. See omakorda võimaldab kuivatusplaani optimeerimisel eelistada kiirema difusiooniga vahemikke ja ignoreerida võimaluse korral aeglasema difusiooniga piirkondi.

Ficki teise seaduse alusel leiti ka kuivatusprotsessi iseloomustav efektiivne difusioonikoefitsient. Efektiivne difusioonikoefitsient on pigem kuivatusprotsessi keskmiselt iseloomustav suurus, kuid võimaldab siiski otsustada, kas kuivatusplaan on puitmaterjali teatud ristlõike suhtes aeglane või kiire.

Väitekirjas püstitati ka kaks uut hüpoteesi: hüpotees Vermaasi hüpoteesi kolmanda erandi olemasolu kohta ning hüpotees PDmeetodil saadud katseandmete lähendusfunktsioonide parameetrite ja vähemalt mõne puitu makroskoopiliselt iseloomustava parameetri (niiskussisaldus, tihedus, kuiv tihedus, deformatsioon, pinge, elastsusmoodul jm) omavahelise statistilise seose olemasolu kohta.

ACKNOWLEDGEMENTS

I would like to thank my supervisors Professor Peeter Muiste and Jaak Pikk PhD, whose valuable advice and dedication have been of great help in planning and carrying out my research as well as in writing research papers.

I would like to thank my former colleagues Engineer Toomas Bernotas, Feliks Miller MSc and Madis Noppel PhD from the Laboratory of Environmental Physics at the University of Tartu for their practical advice in electrometry and theoretical physics.

I am especially grateful to Tavo Romann PhD from the Institute of Chemistry at the University of Tartu for his expert advice in electrochemistry.

I would also like to express my sincere gratitude to all my colleagues at the Institute of Forestry and Rural Engineering of the Estonian University of Life Sciences.

I would like to thank all the co-authors for their contribution to the papers. I hope that our effective cooperation will continue.

I would like to thank Mrs Tiia Kaare for the linguistic revision of the thesis.

I am grateful to my students for their help in data collection in the difficult conditions of industrial wood drying and in data analysis.

This study was partly carried out within projects supported by the Environmental Investment Centre (EIC) (project No 906) and by the Estonian University of Life Sciences (Baseline financed projects No 8–2/T9002MIMI and No 8–2/T5085MIMI05).

ORIGINAL PUBLICATIONS

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Tamme, V., Muiste, P., Mitt, R., Tamme, H. 2011. Determination of Effective Diffusion Coefficient and Mechanical Stress od Pine Wood during Convective Drying. *Baltic Forestry*, 17: 110–117.

Determination of Effective Diffusion Coefficient and Mechanical Stress of Pine Wood during Convective Drying

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Tamme, V., Muiste, P., Mitt, R. and Tamme, H. 2011. Determination of Effective Diffusion Coefficient and Mechanical Stress of Pine Wood during Convective Drying. *Baltic Forestry* 17(1): 110–117.

Abstract

The aim of this study was to find significant correlations between these three components: industrial experiments of drying, drying simulation program and laboratory experiments of drying. The study concentrates on the examination of the dependence of effective diffusion coefficient on mean moisture content in the process of convective drying of pine (*Pinus sylvestris* L.) sawn timber. The methodology of experimental determination of effective diffusion coefficient and the laboratory equipment is described and the results of the experimental determination of effective diffusion coefficient accessary measurements of the moisture content of wood were made using the method based on electrical conductance/resistance. In the course of drying processes 12 effective diffusion coefficients dependent on the moisture content of pinewood were determined on a trial basis.

Laboratory experiments were carried out to register the time of initiation of the first crack on the surface of the pinewood from the start of the drying process, and the results were compared to the maximum relative tensile stress on the surface of the pinewood simulated by the computer programme TORKSIM ver. 3.1. On the basis of the results of the simulation, the maximum relative compressive stress was determined in the core of the pinewood board subjected to drying.

One-dimensional (1D) moisture profile from the surface to the core of the board was measured and the results were compared to the simulated moisture profile of the programme TORKSIM ver. 3.1. Comparison of the moisture contents measured in the laboratory experiment and simulated by the programme showed that best matching of the moisture contents was achieved in the near-surface layer of the board sample. It was concluded that the laboratory equipment was suitable for the assessment of the accuracy of the wood drying simulation programme TORKSIM ver. 3.1 as well as for repeating the drying schedules used in industrial wood drying.

Key words: heat transport, moisture transport, effective diffusion coefficient, tensile stress, compressive stress

Introduction

The indicators of convective drying of wood (i.e. final moisture content, mechanical stresses, initiation of cracks in the wood etc.) are determined by the choice of the drying schedule. Nowadays drying control programmes for kilns are developed by mathematical modelling, i.e. computer simulation (Salin 1990, Rémond et al. 2007) of the drying process. Simulated drying schedules are carefully tested both under laboratory conditions and in industrial environment (Tronstad et al. 2005).

It can also be done conversely by taking a drying schedule which has proved to function efficiently in industrial kilns and test it under laboratory conditions. At the same time computer simulation for the drying process is performed. Certain difficulties can be encountered with the evaluation and interpretation of the results of using commercial simulation programmes, as the exact mathematical model which is the basis for the commercial programme is not known to the user. In most cases general information about the simulation programme is known – i.e. whether the model is one-, twoor three- dimensional (1D, 2D, 3D Model), isotropic or orthotropic. Sometimes background information can be obtained from other sources like a description of the mathematical model likely used in the simulation programme (Salin 1990, Rémond et al. 2007).

Direct experimental determination of the diffusion coefficient according to Fick's First Law is a widely used method. The main reason being that the method

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enables the local diffusion coefficient to be determined in different locations of the wood sample subjected to drying, dependence of the diffusion coefficient on moisture, temperature and coordinate, i.e. the function D(u, T, x) can be examined by means of experiments. The diffusion coefficient can be determined by ovendry method (Hukka 1999, Tremblay et al. 2000), computed tomography, x-ray scanning (Danvind 2005, Cai 2008) and electrical conductivity method (described in this paper). However, only the oven-dry method can be considered as an absolute method, i.e. a method which does not require any comparison or calibration with other methods.

The aim of the study has grown out of practical needs, not laboratory research. The aim was to find significant correlations between these three components: industrial experiments of drying, drying simulation program and laboratory experiments of drying.

Materials and methods

The coupled, uncoupled and diffusion-based simplified models

The coupled model to calculate the combined heat and moisture transport through a porous medium was developed by Luikov (1966), and specific to wood by Siau (1984).

The governing equation for heat transfer through wood board is as follow:

$$\rho_{c_{p}} \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(\lambda \frac{\partial T}{\partial x} \right) + \frac{\partial u}{\partial t} \rho_{w} G_{m} H_{m}, \qquad (1)$$

where x is the distance along the direction flow (m); t is the time (s); ρ is the wood density (kg m⁻³), as a function of moisture content u; c_p is the specific heat capacity of wood (J kg⁻¹ K⁻¹), as a function of temperature and moisture content u (kg kg⁻¹); T is temperature (K); λ is wood thermal conductivity (W m⁻¹ K-1), expressed as a function of temperature and moisture content u; $\rho_{\rm w}$ is the water density (kg m⁻³); $G_{\rm w}$ is the wood specific gravity (kg kg⁻¹) and H_{m} is the latent heat of moisture in wood (J kg⁻¹). The specific gravity of wood, G_m is the ratio of the density (mass of a unit volume i.e. oven-dry mass of wood) of a substance to the density (mass of the same unit volume i.e. mass of water) of a reference substance. The moisture content *u* in the wood material is expressed as the weight of water present in the wood divided by the weight of oven-dry wood substance.

The governing equation for unsteady state isothermal moisture transfer through a wood board is given as the Fick's Second Law (Crank 1956) in one dimension:

$$\frac{\partial u}{\partial t} = \frac{\partial}{\partial x} (D_t(T, u) \frac{\partial u}{\partial x}), \qquad (2)$$

where D_{r} is the transverse wood moisture diffusion coefficient (m² s⁻¹).

It is possible to simplify a coupled model and turn it into an uncoupled model on the assumption that there is no heat generation inside the wood. This assumption can be roughly implemented if the experiment fulfills certain conditions – i.e. in the case that the velocity of heat transfer process through the wood sample is over ten times higher than the velocity of mass transfer i.e. diffusion process. Another simplifying assumption would be the use of empirical formula for thermo-physical properties of wood and other non-linear transfer coefficients (Younsi et al. 2006).

In exceptional cases, the isothermal diffusion equation (i.e. Fick's Second Law) can be used to describe the process of drying wood in narrow temperature range 50-60°C with reasonable accuracy (ref. eq. 2). The prerequisite for using the extremely simplified diffusion-based model is presence of as many reference points of comparison as possible which allow us to observe the dynamics of moisture content and temperature. Such drying schedule resulting in parabolic moisture content distribution in the crosssection of the material perpendicularly to the surface was named quasi steady-state by Luikov (1966). In such case Fick's Second Law, eq. (2), can be presented in the following form:

$$D_t \frac{\partial^2 u}{\partial x^2} = const , \qquad (2a)$$

The solution of its differential equation is square root function.

Upon experimental determination of the diffusion coefficient it is very important to fulfil the assumptions of isothermal diffusion. Isothermal properties were checked by constant observation of differences in temperature on the surface of the sample and at different distances from the surface of the sample both inside the sample (Figure 1) and in the drving air.

The mathematical model being the basis for the wood drying simulation programme TORKSIM ver. 3.1 has not been disclosed in detail. However, on the basis of Salin (1990) it can be assumed that it is perfect isotropic Luikov-type coupled model.

Stress calculation model

In order to ensure quality in the process of wood drying it is necessary to calculate the strain and stress evolved in wood on the basis of previously calculated moisture profile. The mathematical model in the onedimensional isotropic case can be presented on the basis of Salin (1990). The primal equation for stress calculation:

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$$\sigma = aE(\frac{\int_{1/2}^{1/2} E\rho_b dx}{\int Edx} - \rho_b),$$
(3)

where σ - tensile stress (Pa); ρ_b -bound water content (kg m⁻³); E - modulus of elasticity (Pa); l - board thickness (m); x - coordinate from the surface of the board (m).

The governing equation for creep calculation:

$$\frac{\partial \varepsilon}{\partial t} = \frac{1}{E} \frac{\partial \sigma}{\partial t} + \frac{\partial \varepsilon_v}{\partial t} + (a + m\sigma) \frac{\partial \rho_b}{\partial t}, \qquad (4)$$

where ε – total strain; σ - tensile stress (Pa); E – modulus of elasticity (Pa); ε_{ν} – viscoelastic strain; a – unrestrained shrinkage coefficient (m³ kg⁻¹); m – mechano-sorptive creep coefficient (m³ kg⁻¹ Pa); ρ_b – bound water content (kg m³); t – time (s).

Modulus of elasticity E is not a constant, but depends on both moisture content and temperature.

Material

In this paper the data of computer simulations of two industrial experiments and one laboratory experiment of convective drying of pinewood using the 1D programme TORKSIM ver. 3.1 (Trätek 2006) are presented. Also, the approximate inverse determination of efficient diffusion coefficients is presented using the parabola method well-known from simulated moisture profiles (Kretchetov 1972). The diffusion coefficient in this context is defined as the effective diffusion coefficient of the total diffusion flux of the liquid phase and vapour phase and water and bounded water.

In an industrial kiln samples of pinewood (the length the samples 6 m, dimensions of the cross-section 150x22 mm and 150x50 mm and the sawing pattern 4EX-log), located in the middle of the pile, were dried. The samples of pinewood for the laboratory drying experiment had the cross-section of 200x56 mm and the length of 600 mm. In all three experiments the average content of heartwood was $40\% \pm 10\%$. In the laboratory experiment the ends and sides of the wood sample were covered with neutral silicone (a product of Bostik) to ensure the validity of the assumptions of the one-dimensional mathematical model in the experiment.

Experiment

In the laboratory drying test the diffusion coefficients were determined experimentally by means of electrical conductivity method Flick's First Law (Fick 1855) was used . In addition, differences in the temperature of the material subjected to drying and initiation of the first drying crack as a result of drying stress were examined. For this purpose, a forced drying schedule three times shorter than the regular schedule commonly used in industry was applied.

In the industrial chamber type convective dryer a reliable mild drying schedule was used to ensure the high final quality of wood. This method has also been successfully tested in practice before. The drying time of the 22 mm sample was 90 hours and for the 50 mm sample the corresponding time was 336 hours. The initial moisture contents were 55 $\pm 10\%$ and 36 $\pm 10\%$. respectively. The drying chamber was controlled by the relative air humidity and temperature sensor RO-TRONIC Hygro Clip-S (measuring range 0 - 100% RH, precision at 23 °C ±1.5% RH). The wood moisture content was measured by means of screw electrodes in six different points in the location of 1/3 of the thickness of the board from the surface (Figure 1). The values of moisture content were averaged and registered in the log file. Information of the average moisture content of wood was not used in the control of the drying chamber and it was saved as additional information. The average velocity of air in the drying chamber was 2 ms⁻¹. Hysteresis of automatic control in the "on-off" control of the drying chamber was adjusted to ±1.5°C in the temperature channel and to $\pm 2\%$ RH in the relative humidity channel of drying air. During the drying process a schedule with linearly rising temperature on average of 0.01°C and 0.1°C per hour was respectively used for the samples of the thickness of 22mm and 50mm. In both cases the speed of temperature rise was smaller than the fluctuation of temperature per hour caused by the automatic control system of the chamber (i.e. ±1.5°C). The drying schedules for 22 mm and 50 mm material are presented in Tables 1, 2 and 3.

The laboratory experiment with pinewood was carried out in the climate chamber FEUTRON. The forced drying schedule is presented in Tables 1 and 4. The climate chamber was operated according to relative air humidity and temperature of the same type of sensor (ROTRONIC Hygro Clip-S) as in the industrial experiment. The climate chamber was controlled

Table 1. Parameters of 22 mm pine drying schedule , 91h (industrial test), and 50 mm pine drying schedule, 336 h (industrial test), and 56 mm pine drying schedule, 121 h (laboratory experiment)

Boromotoro	22 mm, 91 h, 50 mm, 336 h ,		56 mm, 120 h,
Faiameters	industrial	industrial	laboratory
Initial MC, %	53	35	60
Dry density, kg/m ³	430	430	430
Air velocity, m/s	2	2	2
Heartwood content, %	40	40	40

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Table 2. Scheduleof 22 mm pinedrying (91 h in-	Drying time (h)	Dry bu temp (°C)	llb We ten	et bulb np (°C)	Relative humidity (RH %)
dustrial test)	0	5.4		5.4	99.5
	10	50.0		49.9	99.5
	19	50.0		50.0	94.5
	43	60.0		55.9	81.0
	67	60.0		50.2	59.0
	79	60.0		44.3	41.0
	88	60.0		45.3	43.7
	91	51.0		38.2	45.0
Table 3. Schedule		Dry	Wet		
of 50 mm pine	Drying	g bulb	bulb	Rei	ative
drying (336 h in-	time (h	n) temp	temp		
dustrial test)		(°C)	(°C)	(RF	1%)
	0	8.1	6.9	84	4.6
	15	50.0	48.7	9	2.7
	72	50.0	48.1	8	9.6
	192	55.4	51.3	8	D.1
	300	55.4	43.5	5	0.0
	336	30.0	22.7	5	3.6
Table 4. Schedule	of 56		Dry	Wet	Relative
mm pine drying (121 n		Drying	duib	DUID	humidity
laboratory experime	:11()	time (n)	temp (°C)	temp (°C)	(RH %)
		0	20.0	10.9	30
		1	50.0	48.5	92
		121	60.0	44.0	40

by "on-off" system in which the hysteresis of automatic control was adjusted to $\pm 1.5^{\circ}$ C in the temperature channel and to $\pm 2\%$ RH in the relative humidity of air channel – the same as in the case of the industrial experiments. The temperature rise in the laboratory experiment was linear, 0.08°C per hour. In the laboratory experiment the temperature range was 50°C -60°C, i.e. the same as in the industrial experiment. The difference between the laboratory experiment and the analogous industrial experiment (50 mm pinewood) was the duration of the experiment – the laboratory experiment was carried out in a time period of about 1/3 of that of the industrial experiment (120 hours and 336 hours, respectively).

Location of different sensors in the single specimen, on the surface and near the surface of the sample in the laboratory experiment is shown in Figure 1.

Differences of temperatures in the sample, on its surface and in the ambient air were registered by a thermocouple of AHLBORN (type FTA 3901, resolution of 0.1K) and the data were saved using a ninechannel data logger AHLBORN ALMEMO 2890-9. The locations of five thermocouples were 10 mm from the surface of the sample in the air, on the surface of the

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Figure 1. Location of sensors in the sample

(TC – thermocouples TC0 in air, TC1 under the surface, TC9 9 mm from the surface; TC28 28 mm from the surface; MS – moisture content sensors 4,5 mm, 9 mm and 28 mm distance from surface; DS – displacement sensor; TA – thermo-anemometer; SC - silicon coating)

sample and in the depth of 4.5 mm, 9 mm and 28 mm in the sample. The moisture content of wood was measured from the same depths using AHLBORN timber moisture sensors (type FHA 636M) and the data was saved by the data logger. Also, the moisture content of wood was measured manually at least twice in 24 hours using moisture measuring device GANN HY-DROMETTE HT 85T from the depths of 4.5 mm and 9 mm from sapwood (board thickness 56 mm) and 28 mm from heartwood. The velocity of air was registered by AHLBORN thermo-anemometer of type FVA645 TH2 at 10 mm from the surface of the sample. Strain in the surface layer of the sample and the time of initiation of the first drving crack were registered by the data logger and a displacement sensor of type FWA 025T with the resolution of 0.001 mm.

The accuracy of AHLBORN (type FHA 636MF) timber moisture sensors used in the experiment was $\pm 2\%$ (the accuracy depends on MC). The accuracy data is not presented in the manual of GANN HY-DROMETTE HT85T, which was used for manual measurement of moisture content of the wood. However, the instrument is operating on the principle of electric resistance of wood and thus, the accuracy could be considered to be the same as that of Ahlborn sensors (i.e. $\pm 2\%$). In the experiment the moisture flux and gradient were measured with the same sensor. As the diffusion coefficient was calculated as the quotient of these two indications the accuracy of the determination of the diffusion coefficient is ca $\pm 4\%$. According to Gann (2011) the accuracy of conductance type moisture sensors decreases above fibre saturation point (FSP) compared to the accuracy below FSP.

Multiple regression analysis was done in the statistical software environment of R (2011).

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Figure 2. The graphs of log file and simulation of the results of industrial drying experiment (pine board thickness 22 mm, measurements based on electrical conductance, simulated with TORKSIM ver. 3.1)



Figure 3. Results of mean moisture content simulations with the programme TORKSIM ver. 3.1 compared with industrial measurement (pine board thickness 50 mm)

Results

The graphs of log files of the results of industrial drying experiments and relevant simulations with the programme TORKSIM ver. 3.1 are shown in Figures 2 and 3.

For 22 mm material (Figure 2) the formula of linear regression was:

MC(m) = 4.43437 + 0.88278 MC(s),

Multiple R-squared: R²=0.9775, where

MC(m) - measured mean moisture content MC and

MC(s) – simulated mean moisture content MC.

Comparison of the MC(m) and MC(s) gave the standard deviation 2.94%.

For 50 mm material (Figure 3) the formula of linear regression was:

MC(m) = 0.886608 +0.853483MC(s),

Multiple R- Squared: R² =0.9919,

Comparison of the MC(m) and MC(s) gave the standard deviation 2.60%.

Maximum differences in the temperature on the surface of the sample and inside the sample did not exceed ± 0.7 °C (Figure 4). Also TORKSIM ver. 3.1 shows differences in the temperature on the surface of the sample and in the drying air near the surface of the sample. There was a good match with the differences in temperature measured in the course of the experiment (Figure 4).



Figure 4. Results of the measurements of temperatures and the simulation (laboratory experiment and simulation) Diff 0 - 1 measured temperature difference between air and

surface of board

Simulated Diff 0 - 1 simulated temperature difference between air and surface of the board

Diff 1 - 3 measured temperature difference between surface and core of board

Processing of the simulation results of industrial experiments with programme TORKSIM ver. 3.1 (22 mm pine 91 hours and 50 mm pine 336 hours) showed strong correlation with the quasi-stationary drying regime, the values of R^2 varied within the range of $R^{2=}$ 0.991 – 0.9999, 22 mm board and $R^2=0.9939$ – 0.9998, 50 mm board, respectively (example in Figure 5).



Figure 5. Example of approximation of the simulation of distribution of moisture content with the second level polynomial or parabola equation

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Correlation with the quasi-stationary drying regime was weaker in case of forced drying regime in the laboratory experiment when drying a 56 mm pine board for 121 hours. Processing of the simulation results demonstrated variation of R^2 in the range of R^2 = 0.9821 – 0.9885. Measured and simulated local MC in the depths of 4.5 mm, 9 mm and 28 m from the surface of the board were compared in the laboratory experiment using the forced drying regime (drying 56 mm pine board for 121 hours) and the results are shown in Figure 6.



Figure 6. Comparison of the moisture content measuring data of the laboratory experiment with computer simulation (depth levels 4.5 mm and 28 mm)

According to the simulation results the moisture content can also be determined by the first time derivate. These data enabled the approximate inverse determination of diffusion coefficient by means of the parabola method (Kretchetov 1972) well-known from the Fick's Second Law. Also, it was possible to define the dependence of inversely determined diffusion coefficients on the mean moisture content of wood shown on Figures 7 and 8.



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Figure 7. Dependence of inversely determined diffusion coefficients on mean moisture content of wood (pine board thickness 22 mm)



Figure 8. Dependence of inversely determined diffusion coefficients on mean moisture content of wood (pine board thickness 50 mm)

Figure 9 shows the dependence of the diffusion coefficient on approximate inverse determination of TORKSIM simulation and on the local diffusion coefficient determined directly by the laboratory experiment from the mean wood moisture content (MC).



Figure 9. The dependence of the diffusion coefficient of inverse determination (I.D.) of TORKSIM simulation (I.D. 50 mm and I.D. 22 mm) and the diffusion coefficient determined directly by the laboratory experiment from the mean moisture content (MC)

The first crack in the 56 mm thick pinewood sample emerged 82 hours after the initiation of the drying. The computer simulation of the laboratory experiment indicated maximum relative tensile stresses of the value of 0.33 units at 80 - 90 hours referring to the risk of initiation of drying cracks. Distributions of tensile and compressive stress at a defined moment of time (75, 82 and 121 hours from the start of the drying process) were found in a simulation in the laboratory experiment. The result is shown in Figure 10.

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Figure 10. Distributions of relative tensile (marked with plus) and compressive stress (marked with minus) at a defined moment in time 75, 82 and 121 hours from the start of the drying process, were determined in a simulation in the laboratory experiment

Discussions and conclusions

The accuracy of experimental determination of the diffusion coefficient in a given coordinate point was largely dependent on the resolution capacity and time stability of the measuring device. As the gradient itself is a function of the coordinate, the accuracy was also dependent on the size of the measured sample. When comparing the accuracy of experimental determination of the diffusion coefficient with the method of computed tomography $\pm 3.6\%$ (Danwind 2005) and conductivity method $(\pm 4\%)$ it appeared that the difference in accuracy was not significant. The achieved accuracy can be considered satisfactory for diffusionbased control of wood drying. The problem with the tests was that the moisture content above FSP was not verified by the reliable oven-dry method. When comparing the measuring data of the laboratory drying experiment with computer simulation, relatively good match between the measured and simulated moisture content in the layer near the surface of the sample was observed. However, the moisture content in the middle part of the sample measured at the end of the experiment was considerably higher $(39 \pm 10\%)$ than in case of simulation (18%). Probably the moisture content in the middle of the sample was measured with lower accuracy, as the accuracy of conductance type moisture sensors decreases above FSP.

During the study the approximate inverse determination of effective diffusion coefficient by the parabola method was applied. To use this approximate method for analysis, the code of the commercial simulation programme TORKSIM ver. 3.1 was not essentially needed. The results of the study showed that the electric devices for measuring moisture content can be used for experimental determination of the diffusion coefficient according to Fick's First Law.

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The results of the study indicate that there is good correlation between the moisture content of wood measured during the industrial experiment and the results of computer simulation. It was proved that by the method of electrical conductivity the effective diffusion coefficient dependent on the mean moisture content can be used as an alternative to the oven-dry method. The effective diffusion coefficients can be the basis for comparison of (industrial or laboratory) experiments and corresponding computer simulations. These outcomes are important for improving the method in the future.

During the laboratory experiment, forced drying schedule was used to test the limits of the simulation model by co-ordinate. It was found, that the simulation programme precisely predicted the appearance of first cracks during the experiment. The simulation of the industrial experiments proved, that the high quality of drying of wood can be predicted by the computer simulation of the process. This is not surprising as the simulation programme TORKSIM ver. 3. 1 has been tuned according to the results of 28 full-scale industrial experiments of drying of pine wood.

The accuracy of the results of the measurements above FSP could be influenced by compensation of systematic errors during measurement of small differences of moisture contents. Still it should be mentioned, that the range above FSP is not relevant from the point of view of quality of the drying process, as the shrinkage of wood does not take place above FSP. Usually parts of sawn timber reach FSP at different times, surface earlier than inner parts. This phenomenon was not observed as mild drying schedule was used for this experiment.

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Received 25 September 2010 Accepted 05 April 2011

ОПРЕДЕЛЕНИЕ ЭФФЕКТИВНОГО КОЭФФИЦИЕНТА ДИФФУЗИИ И МЕХАНИЧЕСКИХ НАПРЯЖЕНИЙ ДРЕВЕСИНЫ СОСНЫ ПРИ КОНВЕКТИВНОЙ СУШКЕ

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Резюме

В данной работе исследуется зависимость эффективного коэффициента диффузии древесины сосны от среднего содержания влажности при конвективной сушке. Описываются метод и лабораторное оборудование для экспериментального определения эффективного коэффициента диффузии. Результаты проведенных экспериментов сопоставлены с результатами, полученными одномерной компьютерной программой для моделирования сушки древесины TORKSIM версии 3.1. Для экспериментального определения эффективного коэффициента диффузии проведен ряд измерений влажности древесины при помощи влагомера, работающего на основе измерения электропроволности

В процессе сушки древесины сосны, была экспериментально определена зависимость эффективного коэффициента диффузии от среднего содержания влажности. Во время эксперимента было зафиксировано время появления первой трещины на поверхности образцов с начала сушки. Полученное значение времени сопоставлено с компьютерной моделью, где действуют максимальные относительные напряжения растяжения на поверхности древесины. На основе компьютерной модели были определены максимальные относительные напряжения сжатия в средней части исследуемого образца.

Во время сушки измерен также одномерный профиль влажности образцов от поверхности до середины и сравнено с профилем, полученным при помощи компьютерной программы TORKSIM.

Сопоставление результатов, полученных экспериментальным путем и компьютерным моделированием, показало, что значения локального содержания влажности в обоих методах наиболее совпадают на поверхностных слоях древесины сосны.

Эксперименты подтвердили, что использованное в экспериментах лабораторное оборудование подходит как для оценки точности результатов, полученных одномерной компьютерной программой для моделирования сушки древесины TORKSIM, так и для повтора планов сушки сушилок, используемых в промышленности.

Ключевые слова: теплопроводность, влагопроводность, эффективный коэффициент диффузии, напряжение растяжения, напряжение сжатия

2011, Vol. 17, No. 1 (32)

ISSN 1392-1355

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Tamme, V., Muiste, P., Padari, A., Tamme, H. 2014. Modelling of Resistance-Type Wood Moisture Meters for Three Deciduous Tree Species (Black Alder, Birch, Aspen) in Moisture Contents Above Fibre Saturation Point. *Baltic Forestry*, 20 (1): 157–166.

Modelling of Resistance-Type Wood Moisture Meters for Three Deciduous Tree Species (Black Alder, Birch, Aspen) in Moisture Contents Above Fibre Saturation Point

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Tamme, V., Muiste, P., Padari, A. and Tamme, H. 2014. Modelling of Resistance-Type Wood Moisture Meters for Three Deciduous Tree Species (Black Alder, Birch, Aspen) in Moisture Contents Above Fibre Saturation Point. *Baltic Forestry* 20(1): 157–166.

Abstract

For fast detection of wood moisture content (MC), handheld resistance-type and capacitance-type electrical moisture meters are widely used. In moisture contents above the fibre saturation point (FSP), the measuring accuracy and precision of resistance-type moisture meters starts to progressively decrease as the wood MC increases. The aim of this research was to quantitatively explore this well-known qualitative trend. Three different resistance-type wood moisture meters from leading manufacturers were compared. The display readings of the moisture meters were compared by using the absolute MCs determined for relevant specimens with the oven-dry method. The specimens with the dimensions of $100 \times 60 \times 60$ mm (length x width x thickness) were made of three different tree species (black alder, birch, aspen); a total of 60 specimens per each tree species were used. The specimens were dried in a climatic chamber under equal conditions (32° C and 98% RH) until the desired MC was achieved. All wood MC measurements were conducted at a room temperature of 20° C.

Modelling of experiment data confirmed the progressive increase in the absolute error in a single measurement of wood MC with resistance-type moisture meters as the average absolute MC rose. Based on the model, the absolute error on the same confidence level (95%) and for the average MC of 27% was \pm 3.9% MC, for the average MC of 60% it was \pm 10% MC, and for the average MC of 97% the absolute error was \pm 17.5% MC. The best prediction error in wood average MC predicted on the basis of the same model was \pm 1.12% MC. The same model was used to predict the resolution of resistance-type moisture meters for the above-mentioned average MCs, which was found to be approximately 1% MC.

Key words: wood drying, wood moisture meter, above FSP

Introduction

Portable electric wood moisture meters can be categorised into two groups based on their operating principle: direct current (DC) resistance-type moisture meters (Stamm 1927, James 1988, 1993) and high frequency alternate current (AC) (2-3 MHz) capacitancetype moisture meters (Ressel 2006). Capacitance-type moisture meters are contact-free, whereas a direct galvanic contact with wood is required for resistancetype moisture meters that uses two electrodes to do the measuring. The reliable measuring range of capacitance-type moisture meters is considered 2-30% MC (Ressel 2006, Bergman 2010). Historically, the main and most accurate measuring range of resistance-type moisture meters has been 7-18% in absolute MC (Norberg 1999, Forsen and Tarvainen 2000) (of dry weight), sometimes also 4-22% (Ressel 2006), and the extended measuring range has been 18-30% (Rozema 2010, Boardman et al. 2011), sometimes also 22-40% (Straube et al. 2002). The range above FSP where wood absolute MC reaches 30-100% is considered non-calibrated mostly due to the high variance in the readings of moisture meters (Vermaas 2002, ASTM Standards 2008). It has even been suggested that "... an indicated MC-reading is more or less a rough guess"(Ressel 2006) and that "readings greater than 30% must be considered only qualitative" (Bergman 2010). A more detailed qualitative assessment of resistance-type moisture meters in the range above FSP is given by manufacturers on their web pages, for example Gann (Gann 2013): "In the range above the fibre saturation point (about 30 % MC) readings become progressively less accurate, depending on the moisture content of the timber to be measured, its specific weight and temperature and the species of wood. ... whereas relatively accurate readings can be obtained with oak, beech, white afara, etc. up to range of 60-80 % mois-

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ture content". Also Brookhuis (Brookhuis 2009) stated that "Measurements below 7% and above the wood fibre saturation point are not accurate. For a precise determination beyond the measuring range, we recommend using the oven-dry method". These recommendations were taken into account in the source data used for statistical modelling in this research.

Above FSP, substantial discrepancies were documented (Karu 2011) between moisture meter readings and MCs of specimens, which were determined with the oven-dry method. It was also found that numerical differences between meter readings and relative MCs were noticeably smaller than between meter readings and absolute MCs. For wood air drying practices, the research (Tamme et al. 2012a) presented correction formulas for compensating for the differences between absolute MCs and relative MCs and moisture meter readings.

The resistance method as an affordable and reliable method is widely used for monitoring MCs in the wood drying process (Tronstad et al. 2001, Tamme et al. 2010, 2011) as well as in building envelopes (Straube et al. 2002, Onysko et al. 2008). For this reason, it is especially important to quantitatively evaluate the measuring accuracy of the resistance method for MCs above FSP.

It should be mentioned that wood fibre saturation point (FSP) may somewhat differ between tree species (Higgins 1957). If wood FSP has not been accurately determined, the approximate value of 30% MC is used as FSP (Class and Zelinka 2010).

The primary purpose of this research was to carry out a statistically reliable assessment of the progressive decrease in the measuring accuracy of resistancetype wood moisture meters as the wood MC rises above FSP (30-100% MC). In addition, the research also set out to determine the resolution of the moisture meter Gann HT 85T for both resistance (k?) and moisture content (% MC) units above FSP, and to compare the wood moisture meters used in the experiments.

The statistical models developed for wood moisture content also enable a wider interpretation (and not just a quantitative description) of the phenomenon of progressive decrease in the measuring accuracy of a single measurement with wood moisture meters. The greatest benefit of a statistical model describing wood moisture content is the opportunity to predict the average moisture content of a batch of wood with sufficient accuracy for practical needs, by a non-destructive method and within a reasonable period of time without having to use the labour- and resource-intensive oven-drying procedure every time. Sometimes the wood moisture content prediction is required for determining the mechanical properties of wood, such as the modulus of elasticity (MOE) and modulus of rupture (MOR) by a non-destructive ultrasound method (Wang et al. 2004). In fact, both the MOE and MOR are highly dependent on wood density. In turn, wood density correlates strongly with wood moisture content both above FSP and below FSP (Kretschmann 2010, Cai 2008, Carter et al. 2005).

Materials and Methods

The research made use of specimens of black alder (Alnus glutinosa), birch (Betula pendula), and European aspen (Populus tremula), each with the dimensions of 100x60x60 mm (height x width x thickness). The specimens were dried in the Feutron climatic chamber (Feutron 2013) under equal conditions (at 32°C, 98% RH and air velocity of 0.4 m/s) until the desired MC was achieved. All wood MC measurements were carried out at a room temperature of 20°C. Resistance type wood moisture meters Gann HT 85 T (Gann 2013), FMD-6 (Brookhuis 2013) and NDT James Moisture Master (Ndtjames 2013) from three different manufacturers were used. The measurement resolution of all moisture meters used in the experiments was $\pm 0.1\%$ MC. The chosen measuring depth of measuring electrodes was 1/3 of the thickness of a specimen (that is, in the case of a 60 mm specimen, the measuring depth was 20 mm of the surface of the specimen). Measuring electrodes (teflon insulated pins, 60 mm) were tapped with a Gann hammer electrode RAM- IN electrode M18 (Gann 2013) to a depth of 20 mm. The number of specimens per each tree species was n = 60.

A Kern weighing scale (Kern-sohn 2013) was used. The absolute MC of dry weight of the specimens was determined according to the standard ISO 3130:1975. The following methodological assumption was the basis for the comparison test of the moisture meters: only the average MC of the specimens was determined by the oven-dry method and the respective reading of the moisture meter was recorded. The influence of possible moisture gradients in each specimen on measuring results was not taken into account since a non-destructive method could not be used for detecting moisture gradients in the specimens.

Experiment data was statistically processed in two stages. In the first stage, statistical variables were analysed one by one by methods of descriptive statistics, and in the second stage, the variables (measurements) were examined by measures of association by methods of regression analysis.

While processing the experiment data it became clear that although the purpose was to study MCs above FSP, in some cases the trend lines that had been obtained overlapped The best prediction error in wood

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average MC predicted on the basis of the same model was $\pm 1.12\%$ MC (for example in the case of FMD-6). Due to this overlap, it can be said that the range of application of the established trend lines is even slightly wider than the MCs above FSP that was suggested in the title of this article.

In the second stage of statistical processing the experiment data, a simple univariate linear regression was used. Inverse regression was applied (Onysko et al. 2008) and therefore the moisture meter reading was chosen as the independent variable. Inverse regression was used because it is better suited for practical needs and because the purpose of this research was not calibration of moisture meters but simply their comparison under under the same conditions. A total of nine regression models were developed for three tree species and three moisture meters. Logarithmic transformation was applied to the actual MC variable. The initial variable was again used for making conclusions based on the model. For testing the reliability of the regression line that was found, the Kolmogorov-Smirnov test was used in all comparisons to evaluate the normal distribution of prediction residuals, and where possible, the Shapiro-Wilk test was also used. The tests were then doubled with visual control methods of normal distribution (a histogram with the density of probability and a Q-Q plot). Statistical modeling was done in the statistical software environment of R (r-project 2013).

Statistical modelling of wood moisture content produced the essential parameters of regression models: *R*-squared of the model, (b) standard error (SE) of the model, and (c) 95% confidence interval (CI) of the regression line. The confidence interval for individual predictors is substantially larger than the one computed above for the predicted mean (Sachs 1982). The most important external parameters in relation to regression models include the optimal number of measurements, repeatability of measurements and the repeatability (reproducibility or validation) of regmodels. Repeatability of results of measurement is defined as closeness of the agreement between the results of successive measurements of the same measuring carried out under the same conditions of measurement (ISO 3534-1:1993). Validation of regression models is based on the following property (Sachs 1982): the variance of a sum or difference of independent random variables (e. g. old and new samples) is equal to the sum of their variables, i.e.

D(X + Y) = DX + DY. (1) As rough approximation we can assume that the residual variances *DX* and *DY* of two models are equal, DX = DY, and the standard error of the new regression model *SE1* is:

$$SE1 = \sqrt{D(X + Y)} = \sqrt{2DX} \approx 1.4SE$$

(a) *R*-squared:

$$R^{2} = 1 - \frac{\sum (y_{i} - \bar{y}_{i})^{2}}{\sum y_{i} - \bar{y}_{i}^{2}}$$
(2)

The expression (2) is a measure of how well the predicted (\hat{y}_i) values fit. The less the observed values depart from the fitted line, the smaller this ratio is and the closer R^2 is to 1. Thus R^2 can be considered a measure for how well the regression line explains the observed values.

(b) Standard error of regression model (SE):

$$SE = \sqrt{\frac{\sum_{i=1}^{n} (y_i - \hat{y})^2}{n - 2}}$$
(3)

SE is a measure of the inadequacy of fit for the fitted equation $\hat{y} = a + bx$, or of the error which is made in the estimation or prediction of y from given values of x (Sachs 1982).

Wood electrical equivalent resistance method was used for experimental determination of the resolution of the moisture meter Gann HT 85T in electrical resistance units on two moisture levels selected in the beginning and end of the moisture range above FSP (30-100% MC). For simulation of equivalent wood electrical resistances in the moisture meter input, a resistance box type P4002 was used, which had the accuracy class of 0.05 and a selectable resistance range of 10 k Ω -100 $M\Omega$ (Metrosert 2013). Different resistances were used in the moisture meter input so that the reading would change \pm 0.1% MC on a selected moisture level. The divergence of the resistances in this range is corresponding to the experimentally determined resolution. For calculating the resolution according to the actual MC (that is, gravimetric MC determined with the ovendry method), the most reliable (confirmed by both the Kolmogorov-Smirnov test and the Shapiro-Wilk test) regression line was used based on the presumption of it being 100% reliable. This way, the regression line obtained a fixed transfer function, that is, it acted as a calibration curve (corrected in the comparison test). It was also noted that resolution is an idealised parameter (a certain limit value) of a measuring device, which actual measuring accuracy never achieves.

Results

Research results are presented in the following Tables and Figures. Tables 1, 2 and 3 are presenting the results of processing of the experimental data by the method of descriptive statistics for three hardwood species. Tables 4, 5 and 6 are presenting the results

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Table 1. Testing of moisture meters on black alder specimens

01-11-11	0	Moisture meter readings			
parameter	actual MC %	Gann (MC %)	NDT James (MC %)	FMD – 6 (MC %)	
Range 1	68.6 - 124.8	32.6 - 39.2	30.4 - 33.3	29.8 - 33.5	
Range 1, mean	105.7	35.9	31.6	31.8	
Range 1, st. dev.	12.154	1.299	0.819	0.908	
Range 2	52.7 - 65.5	26.8 - 30.9	25.4 - 30.7	26.4 - 28.7	
Range 2, mean	59.5	28.6	27.7	27.4	
Range 2, st. dev.	3.66	1.104	1.259	0.577	
Range 3	27.3 - 37	19.9 - 23.8	23 – 29	21.4 - 24.7	
Range 3, mean	31.56	21.8	25.6	23.1	
Range 3, st. dev.	2.031	0.793	1.52	0.718	

Table 2. Testing of moisture meters on birch specimens

Otatiati and	Oven dry	Mois	Moisture meter readings		
parameter	actual MC %	Gann (MC %)	NDT James (MC %)	FMD – 6 (MC %)	
Range 1	69.4 -91.5	38.3 - 46.8	32 - 36.6	31.3 - 36.8	
Range 1, mean	85.2	42.4	34	33.8	
Range 1, st. dev.	3.85	2.129	1.067	1.22	
Range 2	44.5 - 60.3	31.9 - 36.6	27.3 – 31.2	27.2 - 31	
Range 2, mean	49.8	34.1	29.4	28.8	
Range 2, st. dev.	4.44	1.312	1.167	0.914	
Range 3	26.4 - 37	22.5 -28.8	23.3 - 29.5	23.2 - 28.7	
Range 3, mean	30.3	24.9	26.6	25.2	
Range 3, st. dev.	2.355	1.423	1.409	1.069	

Table 3. Testing of moisture meters on aspen specimens

Otafafaal	o 1	Moisture meter readings			
parameter	actual MC %	Gann (MC %)	NDT James (MC %)	FMD – 6 (MC %)	
Range 1 Range 1, mean Range 1, st. dev. Range 2 Range 2, mean Range 2, st. dev. Range 3 Range 3, mean	86.5 - 117.2 107 7.745 39.2 - 70.5 56.5 7.866 23 - 40.6 29.9	43.7 - 50.1 47.4 1.327 33.6 - 37.6 35.9 1.07 20.5 - 29 24.3	34.8 - 39.7 37.6 1.018 28.2 - 33.4 30.5 1.251 21.8 - 29.2 25.3	31.5 - 35.7 32.6 0.885 24.9 - 30.1 27.2 1.25 20 - 29.3 23.5	
Range 3, st. dev.	3.482	1.911	1.753	1.856	

of modelling of moisture metres for three hardwood species and Figures 1, 2 and 3 display the results graphically. Figures 4 and 5 present the regression residuals histogram and Q-Q plot (propability paper) corresponding to positive Shapiro-Wilk test results. According to the Kolmogorov-Smirnov test, the distribution of prediction residuals in the regression model may be considered close to normal. The regression models used in all comparisons fit this definition. According to the Shapiro-Wilk test, the distribution of residuals in the regression model may be considered normal. The regression models in two comparisons (black alder – Gann HT 85T and birch – NDT James Moisture Master) fit this definition.

The resolution of the moisture meter Gann HT 85T according to resistance was experimentally determined (the wood group switch was in the 3^{rd} position and the temperature switch in the 20°C position) with the resistance simulation method for two wood MCs: wood MC of 85.4% ± 0.1% MC produced the resolution of

Table 4. Mode	lling of moisture meters on black alder spec-
imens. Indepen	dent variable in regression model is moisture
meter reading	in MC %

		Meter			
Parameter	Gann HT 85T	NDT James	FMD-6		
No. of observations	134	133	134		
Intercept (Int.)	0.711	-0.358	0.573		
Lower 95% of Int.	0.675	-0.514	0.458		
Upper 95% of Int.	0.748	-0.201	0.688		
p-value of Intercept	p < 0.001	p < 0.001	p < 0.001		
Slope (Sl.)	0.03640	0.0743	0.0427		
Lower 95% of SI.	0.0352	0.0688	0.0387		
Upper 95% of SI.	0.0376	0,0798	0.0468		
p-value of Slope	p < 0.001	p<0.001	p < 0.001		
R-squared (R ²)	0.963	0.847	0.765		
Standard Error of	0.04836	0.09783	0.1213		
Regmodel (SE)					
p-value of	p < 0.001	p < 0.001	p < 0.001		
Regmodel	·				
*) ks. test of	p = 0.5412 > 0.05	p = 0.4156>0.05	p = 0.1224>0.05		
residuals, p-value					
Shapiro-Wilk test of residuals, <i>p</i> -value	<i>p</i> = 0.2274 > 0.05	<i>p</i> = 0.0177<0.05	p=3.0e-5<0.05		

*) ks.test - Kolmogorov-Smirnov test of residuals

Table 5. Modelling of moisture meters on birch specimens. Independent variable in regression model is moisture meter reading in MC %

		Meter			
Parameter	Gann HT 85T	NDT James	FMD-6		
No. of observations	134	134	134		
Intercept (Int.)	0.8673	0.0437	0.2468		
Slope (SI.)	0.0249	0.05504	0.0495		
R-squared (R ²)	0.968	0.920	0.942		
Standard Error of Regmodel (SE)	0. 0385	0.0604	0.0514		
ks. test of residuals, p-value	p = 0.1069 > 0.05	<i>p</i> = 0.7638 > 0.05	p = 0.1995 > 0.05		

Table 6. Modelling of moisture meters on aspen specimens. Independent variable in regression model is moisture meter reading in MC %

Parameter		Meter			
alameter	Gann HT 85T	NDT James	FMD-6		
No. of observations	140	140	140		
Intercept (Int.)	0.894	0.399	0.155		
Slope (SI.)	0.0239	0.0432	0.0568		
R-squared (R ²)	0.982	0.946	0.936		
Standard Error of Regmodel (SE)	0.0361	0.0619	0.0676		
ks. test of residuals, <i>p</i> -value	p = 0.4579 > 0.05	p = 0.1469 > 0.05	p = 0.09119 > 0.05		

 $10 \text{ k}\Omega \pm 0.104 \text{ k}\Omega$, b) wood MC of $30\% \pm 0.1\%$ MC produced the resolution of $370 \text{ k}\Omega \pm 5 \text{ k}\Omega$. The resolution of a wood moisture meter according to resistance is significant when a resistance meter is added in the moisture meter comparison test.

According to a statistical model, the resolution of the moisture meter Gann HT 85T was calculated at wood actual MC as follows: a) 97% MC produced the resolution of 0.82% MC, and b) 27% MC produced the resolution of 0.23% MC. According to the actual MC





Figure 1. Regression lines of testing wood moisture meters Gann HT 85T, NDT James Moisture Master and Brookhuis FMD-6 on black alder specimen with their 95% confidence and tolerance bands (LOG(MC actual %) – actual MC in \log_{10} ; Gann, NDT, FMD – meter reading MC, %)

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Figure 2. Regression lines of testing wood moisture meters Gann HT 85T, NDT James Moisture Master and Brookhuis FMD-6 on birch specimen with their 95% confidence and tolerance bands (LOG(MC actual %) – actual MC in \log_{10} ; Gann, NDT, FMD – meter reading MC, %)

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Figure 3. Regression lines of testing wood moisture meters Gann HT 85T, NDT James Moisture Master and Brookhuis FMD-6 on aspen specimen with their 95% confidence and tolerance bands (LOG(MC actual %) – actual MC in \log_{10} ; Gann, NDT, FMD – meter reading MC, %)

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Figure 4. Black alder – Gann HT 85T histogram of residuals with curve of density of probability of normal distribution and Q-Q plot of regression model

Theoretical Quantiles

above FSP, the total resolution based on a robust approach, is approximately 1% MC.

From a practical standpoint, it is vital to answer the question of up to which MC the moisture meters can reliably be used. In using moisture meters, the upper MC limit has been determined with an electrical resistance value at which the meter still displays a stable reading. Depending on the tree species, this value may be different. For example, the research (Tamme et al. 2012b) found the minimum electrical resistance to be 10 kŁ, which corresponded to the average oven-dry MC of 146% of the pine sapwood specimen.

Statistical modelling of experiment data confirmed the progressive increase in the absolute error in a single measurement of wood actual MC as the average absolute MC rose. The absolute error found by the moisture meter Gann HT 85T on the basis of the modMODELLING OF RESISTANCE-TYPE WOOD MOISTURE METERS /.../



Figure 5. Birch – NDT James Moisture Master histogram of residuals with curve of density of probability of normal distribution and Q-Q plot of regression model

el on the same confidence level (95%) and for the average MC of 27% was \pm 3.9% MC; for the average MC of 60% it was \pm 10% MC and for the average MC of 97% the absolute error was \pm 17.5% MC. The absolute error with a "+" sign was determined on the basis of the upper 95% confidence limit of regression parameters and the absolute error with a "-" sign was determined on the basis of the lower 95% confidence limit of regression parameters.

When comparing the moisture meters used in the experiments, it can be suggested based on tables 4, 5 and 6 that the SEs of the regression models developed for the moisture meter Gann HT 85T are, in the case of all three tree species, approximately two times smaller than those for moisture meters NDT James and FMD-6. Moreover, R-squared values of the models produced for Gann HT 85T are the greatest in the case

of all three tree species compared to the other two moisture meters NDT James and FMD-6 used in the experiments.

Discussion

The standard deviation (st. dev.) in the empirical distribution of the MCs determined in the specimens with the oven-dry method indicates a downward trend when the specimens are slowly dried under equal conditions (see Tables 1, 2 and 3). This trend emerged in case of the specimens of all three dried tree species. The fact that a slow drying mode evens out the differences between the average MCs of the specimens is, most likely, one of the manifestations of Fick's law.

Modelling of wood MC above FSP improves the accuracy of determining wood MC. This quality can be best illustrated by (figuratively) transferring the modelling results obtained in this research to a real production situation (in sawmills, treatment plants and so on).

Example (of a figurative construction): 60 boards (i.e. the sample) have been randomly selected from a batch of black alder boards (e.g. the population). The task is to estimate the average moisture content of the batch of wood based on the sample. First, the ovendrying method is used. If the moisture content of a single board is determined by oven-drying, the average possible error is 12.2% (see row "St. dev. of range 1" Table 1 in the case of an average MC of 97% chosen for the example calculated by the parameters of regression model described in the Table 4). However, determining the moisture content of all 60 boards by ovendrying enables detecting the average moisture content (97% MC in this specific example) with great accuracy (see Table 1 data, at least 1.58% MC). In spite of the high accuracy of the oven-dry method, it also has a few flaws. Firstly, it will take a few days to get the results. During this time, the wet wood has continued to dry on the storage site and thus, the results may no longer be reliable. Secondly, this method is a destructive one as sawing the specimens out of the boards reduces the quality of the remaining board parts. Thirdly, the cost of electric energy, labour force and equipment (highquality drying chambers and precise scales) is high. All in all, using the oven-dry method in practice is only justifiable in certain cases and for the purpose of developing statistical models. In the following segment, the average moisture content of 60 boards (sample) is predicted on conditions equal to those of the oven-dry method. Data in Table 1 is applied separately to variables and data in Table 3 is used for the regression model of the moisture meter Gann HT 85T. By measuring the moisture content of a single randomly selected board with the moisture meter Gann HT 85T, the average pos-

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sible margin of error is 17.5% MC (the margin of error, in this case, is somewhat larger than in the case of the oven-dry method where it is 12.2% MC). If the average moisture content of the 60 boards (which as in the previous examples was 97% MC) is predicted on the basis of the SE of the regression model, the possible average margin of error is 1.12% MC. Therefore, the prediction of the average moisture content of the wood batch based on the model has proven to be surprisingly accurate. To confirm this prediction, the 60 boards of the sample must be actually measured. If a wood moisture meter has a memory for recording readings (as in FMD-6 or Gann model 2050), the actual measuring speed is two measurements per minute and the entire procedure would take half an hour. Let us assume that instead of 60 measurements we limit ourselves to 30 measurements, thereby halving the number of measurements. In that case, the SE of the model would increase by four times based on the equation (3), and in predicting the average moisture content, we would have on average a margin of error of 1.56% MC. According to the model repeatability principle, the measuring series of the next 60 measurements (that is, the new sample) should result in a 1.4 time increase in the SE. This would mean that in the next moisture content prediction based on the previous model and new measuring data, we would on average have a margin of error of 1.17% MC.

In the modelling of wood moisture content, it is important to keep in mind that there is no real need to use calibrated measuring instruments such as wood moisture meters. Therefore, the metrological parameters that were significant for calibrated wood moisture meters (e.g. accuracy, repeatability, reliable measuring range, etc.) take on a new meaning in a statistical model even from the point of view of the model as a whole. For example, an analogy could be drawn between the terms accuracy and quality of the model and confidence interval of estimated regression parameters, and reliable measuring range could be similar to reliable model domain, and repeatability could be compared to model reproducibility, etc. If this research had made use of three electrical resistance meters (that is, if their scale readings would have been given in $k\Omega$) instead of three calibrated wood moisture meters, there would probably not have been any noticeable changes in the obtained regression model parameters. The only prerequisite for measuring instruments used for modelling is their sufficient moisture sensitivity (that is, upon a single-unit change in wood moisture content their output signal should be reliably detected). Linearity of the output signal of the used measuring instruments as well as time stability would also improve the essential parameters of the statistical model. The methodology applied in this

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research does not require according to Vermaas (Vermaas 2002) "attempts to "calibrate" resistance moisture meters for use above f. s. p.".

Conclusions

To acquire the calibration curve, the *in situ* calibration method is used, which is characteristic of nondestructive methods. It is presumed that an individual measurement is non-reliable; essentially the precalibration of the moisture meters by the manufacturer is also ignored. The statistics procedure automatically adapts to the pre-calibration and produces correct parameters of the model. With this, the pre-calibrated wood moisture meters are recalibrated. The recalibration curve is found for the average values of a large number of measurements, as is the practice in non-destructive methods.

Modelling of experiment data confirmed the progressive increase in the absolute error in a single measurement of wood MC with resistance-type moisture meters as the average absolute of MC rose. Based on the model, the absolute error on the same confidence level (95%) and for the average MC of 27% was $\pm 3.9\%$ MC, for the average MC of 60% it was \pm 10% MC and for the average MC of 97% the absolute error was \pm 17.5% MC. The predicted error of wood average MC by the same model was $\pm 1.12\%$ MC. The model was also used to predict the resolution of resistance-type moisture meters, which was found to be approximately 1% MC. It was found that in the modelling of wood moisture content, utilization of measuring instruments previously calibrated into wood moisture meters is not necessary. Necessary prerequisites include moisture sensitivity of measuring instruments, linearity of output signal, and time stability.

Acknowledgements

This research was supported by grant No. 906 of the Environmental Investment Centre of Estonia.

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Received 12 July 2013 Accepted 15 May 2014

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МОДЕЛИРОВАНИЕ ПРИБОРОВ ИЗМЕРЕНИЯ ВЛАЖНОСТИ ПО СОПРОТИВЛЕНИЮ НА ПРИМЕРЕ ТРЕХ ЛИСТВЕННЫХ ДЕРЕВЬЕВ (ОЛЬХА ЧЕРНАЯ, БЕРЕЗА, ОСИНА) ПРИ СОДЕРЖАНИИ ВЛАЖНОСТИ ВЫШЕ ТОЧКИ НАСЫЩЕНИЯ ОДНОГО ВОЛОКНА

В. Тамме, П. Муйсте, А. Падари и Х. Тамме

Резюме

Для измерения содержания влажности древесины широко используются портативные электрические приборы сопротивления. При увеличении влажности древесины в пределах выше точки насыщения волокна точность измерения таких приборов начинает прогрессивно снижаться. В данной работе представлена квантитавная характеристика даннои хорошо известной квалитативной тенденции. Показания трех приборов от ведущих производителей для измерения влажности сравнивали с абсолютной влажностью определенной методом взвешивания высушеных образцов. Из каждого вида древесины (ольха черная, береза, осина) были изготовлены 60 образцов с размерами (60×60×100) мм³. Образцы сушили до желаемого содержания влажности в климатической камере при одинаковых условиях (температура 32°С и относительная влажность воздуха RH 98%). Все измерения проводили при комнатной температуре 20 °С.

На основе статистического моделирования при одиночном измерении MC (moisture content) древесины подтвердилось прогрессивное снижение точности измерения приборов влажности на основе измерения сопротивления при увеличении средней абсолютной влажности древесины. При 95% доверительном уровне и при среднем MC=27% абсолютная ошибка измерения статистической модели была $\pm 3.9\%$ MC, при среднем MC=60% абсолютная ошибка измерения статистической модели была $\pm 3.9\%$ MC, при среднем MC=60% абсолютная ошибка измерения статистической модели была $\pm 3.9\%$ MC, при среднем MC=60% абсолютная ошибка измерения статистической модели была $\pm 1.0\%$ MC и при среднем MC=97% абсолютная ошибка измерения статистической модели была $\pm 11.0\%$ MC. Наименьшая ошибка прогноза среднего MC для древесины по данной модели была $\pm 1.12\%$ MC. На основе представленной модели был составлен прогноз предела разрешения использованных приборов для изученной древесины при средних MC, соответствующее значение получили в пределах 1% MC.

Ключевые слова: сушка древесины, измеритель влажности древесины, выше FSP.

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Experimental study of electrode effects of resistance type electrodes for monitoring wood drying process above fibre saturation point

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Tamme, V., Muiste, P., Kask, R., Padari, A., Tamme, H. 2012. Experimental study of electrode effects of resistance type electrodes for monitoring wood drying process above fibre saturation point. – Forestry Studies | Metsanduslikud Uurimused 56, 42–55. ISSN 1406-9954.

Abstract. Due to practical need to improve the accuracy of measurement of wood electrical resistance via creating multivariate calibration models the study of electrode effects was carried out. The experiments were made in DC mode and five types of electrode effects were measured: electrical resistance of the galvanic contact between the measuring electrode and wood, corrosion of measuring electrodes in wet wood, polarization and depolarization of the double layer forming on the surface of the measuring electrodes, residual polarization voltage between measuring electrodes and effect of repeated use of measuring electrodes. For multi-channel measurement mode effect of neighbouring electrodes was measured. Behind each of these factors is a complex transfer mechanism of free and bounded charge carriers from wood to measuring electrodes. Among the factors, polarization and depolarization have an immediate effect (duration of the processes in seconds) while resistance of electrode/wood contact, corrosion of electrodes and residual polarization have a slow effect (duration of processes in hours). For pine sapwood, coefficients of the Stamm formula were found in moisture contents (MC) above the fibre saturation point. It was determined that wood polarization and depolarization indicators are dependent on wood moisture content as is the case with wood electrical resistance. Compared to wood electrical resistance, wood polarization proved approximately four times and depolarization approximately eight times less sensitive to moisture content variation.

Key words: wood drying, above FSP, corrosion, polarization.

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Introduction

The electrical resistance method is widely used for monitoring wood drying since the method is economical and reliable (Tronstad *et al.*, 2001, Onysko *et al.*, 2008). But above fiber saturation point FSP (MC 30%) the measurement is complicated as the measuring accuracy is insufficient and starts decreasing correspondingly to an increase in wood moisture content (Edwards, 1974; Vermaas, 2002). The decrease in measuring accuracy can also be described quantitatively, by the 2S-value (Rozema, 2010). Insufficient measuring accuracy above FSP also reduces the accuracy of effective diffusion coefficient monitoring in the process of wood drying (Tamme *et al.*, 2010, 2011). For precise determination of moisture content it is recommended to combine moisture meter measurements with the oven dry method for the measuring range above FSP (2)

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(Brookhuis, 2009). This recommendation has been taken into account in this study.

The main empirical relationships and definitions of the resistance method have been given in studies (Stamm, 1927; Norberg, 1999).

The moisture content MC of wood, M, is generally defined as the ratio of the mass of water to the mass of dry wood:

$$M = m_{water}/m_{dw'}$$
(1)

where

m_{water} – mass of water

 $\rm m_{dw}$ – mass of dry wood.

w = 100M,

where

w - moisture content, MC, in [%].

The common relationship, originally proposed by Stamm (Stamm, 1927) expresses the effect of the wood MC on resistivity at a constant temperature:

 $\log \rho = C + D\log M, \tag{3}$

where

 ρ – resistivity

M – moisture content, MC

C and D – constants at constant temperature.

This expression (3) was found to work satisfactorily within the typical range of the MC, where electric moisture metes are used, *i.e.* 7–30%.

In the case of electrical moisture meters, the MC is related to the resistance obtained for the given electrode configuration and for the given equivalent DC circuit.

For a given electrode configuration in the first approach:

 $R = \rho (L/A) = K \rho, \qquad (4)$

where

R – resistance

K – cell constant, or measuring volume constant

L/A – cell constant for the particular electrode configuration

A – electrode/wood contact area, mm²

L – length between two electrodes.

Taking into account formulas (2), (3) and (4), ratio (3) may be replaced with an equivalent, yet a more convenient ratio for practical use:

$$\log R = C_1 + D\log w,$$
(5)
there

D – constant at a constant temperature and the modified constant at a constant temperature

$$C_1 = C + \log K - 2D \tag{6}$$

MC up to 40% of Douglas Fir can be calculated by the following equation (Straube *et al.*, 2002):

$$\log w = 2.99 - 2.113(\log (\log R)),$$
 (5a)
where

w – moisture content in mass %

R – resistance in Ohms (Ω).

In the measurement of wood DC resistance, the following factors are important:

a) electrical resistance of the galvanic contact between the measuring electrode and wood, b) corrosion of measuring electrodes in wet wood, c) polarization and depolarization of the double layer forming on the surface of the measuring electrodes (Metrohm Autolab B.V., Eco Chemie, http:// www.ecochemie.nl) and of the polar molecules of wood itself, d) residual polarization voltage between measuring electrodes, and lastly, e) effect of repeated application (repeated measurements) of measuring electrodes. Behind each of these factors is a complex transfer mechanism of free and bounded charge carriers from wood to measuring electrodes (James, 1975). Among the factors, polarization and depolarization have an immediate effect (duration of processes can be measured in seconds); whereas resistance of electrode/wood contact, corrosion of electrodes and residual polarization have a slow effect (duration of processes can be measured in hours).

These mechanisms can be examined in more detail with the help of certain alternate current (AC) equivalent circuits using electrical impedance spectrometry (EIS) (Zelinka, 2006, 2007; Metrohm Autolab B.V., Eco Chemie, http://www.ecochemie.nl)). The experience showed that the electrode effects involved in measuring wood electrical resistance should be observed in DC mode in order to avoid problems in using results obtained in AC mode for DC circuits.

Several experiments in DC mode were carried out during this research, the purpose of which was to assess five types of electrode effects both in terms of quantity and quality, mostly due to the practical need to minimise measurement errors in wood electrical resistance and also for creating multivariate calibration models.

Material and Methods

In studying electrode effects on a raw data file (set of unprocessed measurement data with numeric or analogous filters) of electrical resistance measurement (Fig. 4), a pine sapwood specimen with the dimensions of 100×60×60 mm (length × width × thickness) was used. It was dried in the climatic test chamber Feutron (Feutron, http://www.feutron.de) at room temperature (20 °C, 96% RH) in the stationary air for 90 hours and thereafter at 32 °C and 96% RH at the air velocity 0.4 m s⁻¹ for another 244 hours. The specimen drying process lasted a total of 334 hours during which the wood MC was reduced from 146% to 36.4%.

For slow processes like the resistance of electrode/wood contact and corrosion, the resistance meter Scanntronik Material Moisture Gigamodule with Scanntronik Thermofox data logger was used (Scanntronik Mugrauer Gmb, www. scanntronik.de). For measuring the voltage of the slow process of residual polarization, the profi-tester Meterman 38 XR was used (The Test Equipment Depot, http:// www.testequipmentdepot.com/meterman/dmm/38xr.htm/). In measuring slow processes, the minimum interval between two measurements was 1 hour. For measuring the fast process of polarization resistance, the resistance meter AlphaLab Inc. (http://www.trifield.com) was used within the measurement range of 0–20 M Ω , with a measuring accuracy of \pm 2%. For saving measurement data in the data logger

Ahlborn (Type ALMEMO 2590-9) (Ahlborn, http://www.ahlborn.com/), an analogue output (with a range of 0-1 V), which had a linear connection with the resistance measuring range of 0–20 M Ω , was attached to the resistance meter AlphaLab. Readings of the analogue output and Alphalab display were related by the following formula: 10 (analogue output reading in volts (V) = display reading in megaohms (M Ω). The voltage of the analogue output could be measured at a speed of 10 measurements per second or 1 measurement per second by using the Ahlborn DC voltmeter ZA9000FS3. For measuring the voltage of another fast process, depolarization, the Meterman 38 XR profi-tester with an input resistance of 10 $M\Omega$ was used, which enabled the measurement speed of 1 measurement per second. For the measurement during the experiments insulated pin electrodes were used (ram-in electrode M18, 60 mm, manufactured by Gann).

In the measurement of electrical resistance, polarization resistance, depolarization process and residual polarization, electrodes e1 and e2 were used at a distance of L = 30 mm (measuring direction was across the grain) (see Fig. 2). In examining electrode/wood resistance R_c and electrode corrosion, electrodes e1 and e2 as well as an extra pair of electrodes e3 and e4 were used at a distance of L/2 = 15 mm. The distance between the main measuring electrodes (e1, e2) and extra electrodes (e3, e4) was D = 40 mm. Distance D was experimentally selected in order to avoid interaction between neighbouring electrodes, i.e. transmission of electrical potential by wet (143% MC) wood. The difference between transmitted potentials (electrical voltage) was measured in dead neighbouring electrodes, which was below 1/30 measuring voltage in the case of D = 40 mm.

The specimen was weighed with a scale manufactured by KERN (Model KERN EW B 620-M, resolution 0.01 g) (Kern & Sohn GmbH, http://www.kern-sohn.com) at 24hour intervals on average; the oven dry Figure 1. Experimental setup.

- 1 AlphaLab resistance meter,
- 2 Scanntronic 8 channel
- resistance meter,
- 3 box of reference resistances,
- 4 switching box,
- 5 climatic chamber Feutron.

Joonis 1. Eksperimendi aparatuur.

- 1 AlphaLab takistusmõõtja,
- 2 Scanntronic 8 kanaliga takistusmõõtja,
- 3 võrdlustakistite salv,
- 4 lülituspaneel,
- 5 kliimakamber Feutron.





Figure 2. Arrangement of measuring electrodes on the examined specimen. All electrodes were placed at a depth of 20 mm from the surface of the specimen.

Joonis 2. Mõõteelektroodide paigutus uuritaval katsekehal. Kõik elektroodid olid paigutatud 20 mm sügavusele katsekeha pinnast.

weight of the specimen was determined with the same scale.

Gann HT 85 T (Gann Mess- u. Regeltechnik GmbH, http://www.gann.de), NDT James Moisture Master (James Instruments Inc., http://www.ndtjames. com) and Brookhuis FMD-6 (Brookhuis, http://www.brookhuis.com) resistance type wood moisture meters were used in the experiments.

Unfortunately, the electrical resistance measurement raw file volume and internal data processing algorithm in the resistance meter Scanntronic Gigamodule and in the resistance type wood moisture meters used in the experiments remained hidden from the user. The experimental setup is depicted in Figure 1.

Interaction registration methodology

The methodological prerequisite for the registration of interactions in the research was the measurement of all examined interactions by DC measuring instruments with the same operating principle. That methodological restriction was necessary to work out a novel precise calibration method of a DC measuring instrument (but not an AC measuring instrument such as the EIS) of wood moisture:

a) resistance of electrode/wood contact on DC and b) electrode corrosion. According to studies (Norberg, 1999) and (James, 1993), $R_{c'}$ resistance associated with the wood/electrode interface, and $R_{v'}$ volume resistance, are series connected. By varying the distance L between electrodes (see Fig. 2), a linear system of equation (7) may be proposed for the four-electrode measuring system:

 $R_{1} = 2R_{c} + R_{v}$ $R_{2} = 2R_{c} + 0.5R_{v}$ (7)
the solutions of which are:

 $R_c = R_2 - 0.5R_1$, (7a) and

 $R_v = 2(R_1 - R_2),$ (7b) where

 R_1 – resistance measured between electrodes (e1) and (e2) (see Fig. 2)

 R_2 – resistance measured between electrodes (e3) and (e4)

 R_c – resistance of a single electrode/wood contact

R_v – volume resistance.

The solution (7b) may also be the corrosion indicator. Analysis of solution behaviour is given in discussion.





- Figure 3. Simplified equivalent circuit for describing the processes of polarization and depolarization.
- Joonis 3. Lihtsustatud ekvivalentskeem polarisatsiooni ja depolarisatsiooni protsesside kirjeldamiseks.

c) polarization and depolarization. The polarization of the double surface layer of the electrodes and wood polarization are relaxation processes the simplified equivalent circuit of which is adapted (Onysko, 2008) and given in Figure 3. Polarization is expressed in the DC circuit by an increase in polarization resistance over time. As part of the experiment, the resistance meter AlphaLab allowed to measure it directly. Measuring polarization resistance enables the approximate assessment of the maximum measurement error in wood electrical resistance depending on wood moisture content. In depolarization process, voltage between the electrodes is the easiest to measure with the help of a DC voltmeter with high input resistance. This measuring method is also called a chronopotentiometric measurement (Metrohm Autolab B.V., Eco Chemie, http://www.ecochemie.nl). Measuring depolarization voltage allows a practical assessment of the time interval that is safe for beginning the next resistance measurement without risking the occurrence of a measurement error. Initial voltage relation U_1/U_0 (where U_0 is the input voltage of the resistance meter and U_1 is the initial voltage of the depolarization process) and T_{max}, the duration of the depolarization process, may also hypothetically be related to wood moisture content.

d) Residual polarization voltage between the measuring electrodes may hypothetically be caused by the difference between the corrosion potentials of the electrodes. Corrosion potential is usually measured with regard to a reference electrode used in electrochemistry (e.g. the Ag.AgCl reference electrode (Metrohm Autolab B.V., http://www.metrohm-autolab.com/Products/Echem/Accessories/accessories. html). Thus, residual polarization could be treated as the combined effect of the corrosion potential, measurement voltage of electrodes and the varied electrical mobility of positive and negative charge carriers. From a practical viewpoint, residual polarization voltage would mean the occurrence



Figure 4. Example of polarization resistance measurement raw file at pine wood MC 146% and 20 °C. Joonis 4. Näide polarisatsiooni takistuse mõõtmise toorfailist männi maltspuidus 146% NS ja 20 °C.

of a certain systematic error in measuring wood electrical resistance with a DC measuring device.

e) Effect of repeated application (repeated measurements) of pin electrodes.

The experiments made use of specimens made of five different tree species (Scots pine, Norway spruce, Grey alder, Birch and European aspen) with dimensions ($100 \times 60 \times 60$ mm) and resistance type wood moisture meters by three different manufacturers (Gann HT 85 T, FMD-6, and NDT James). Measuring electrodes (teflon insulated pins, 60 mm) were tapped with a Gann hammer electrode (RAM- IN electrode M18) to a depth of 20 mm (1/3 of the thickness of the specimen). The number of specimens per each tree species was n = 60.

These types of measurements have a significant practical value in terms of moisture meter calibration, yet at the same time, while conducting measurements, several random factors need to be considered (for example, wood structure inhomogenity, moisture content gradients, etc.). The effect of the repeated use of electrodes is revealed in the moisture meters comparison test. It was assumed that electrode corrosion may be neglected when conducting short-term measurements of wood moisture content. In distributing moisture meters into accuracy classes by the 2S-value (Rozema, 2010), all other random factors must be kept at an equal a level as possible. The 2S-value indicates the range within which, with a 95% certainty, the actual moisture content will fall if determined according to the oven dry method (Brookhuis, 2009).

In the test of moisture meters the electrical resistance of wood/electrode contact was measured indirectly via calibration and algorithm of processing the raw file of the electrical resistance measurement.

Results and Discussion

By comparing Figures 5 and 7, the figures seem to have a relatively similar shape. Based on this similarity, a hypothesis may be proposed that the resistance method owes its moisture sensitivity mainly to the resistance of the electrode/wood contact.

Figure 6 shows that during the drying process, wood volume resistance R_v turns negative for a certain time period, and then back to positive, and maintains relatively stable (in a logarithmic scale). Reasonably



Figure 5. Monitoring of electrical resistance of pine sapwood specimen by using a four-electrode measuring system and Scanntronik Gigamodule resistance meter.

Joonis 5. Elektritakistuse monitooring männi maltspuidus, kasutades nelja elektroodiga mõõtesüsteemi ja Scanntronik Gigamodule takistusmõõtjat.

Figure 6. Behaviour of volume resistance of wood. *Joonis 6. Puidu ruumtakistuse*





Figure 7. Behaviour of electrode/wood contact resistance.

Joonis 7. Elektrood/puit kontakti takistuse muutumine ajas. (proceeding from general physical considerations), wood volume resistance cannot be effectively zero. This phenomenon may be explained by the following qualitative model of temperature dependent electrode corrosion affecting electrical resistance.

Step 1. For some random reason, accelerating corrosion occurs in one of the electrodes, or a pair of electrodes, and according to the measurement data of four-electrode measuring system it seems that wood volume resistance (in a logarithmic scale) turns negative.

Step 2. Logically, it may be assumed that accelerating corrosion in some electrodes cannot last for very long. Indeed, a more intense corrosion should result in an increase in the resistance of the electrode/ wood contact, and thus, current intensity through the more corroded pair of electrodes would decrease.

Step 3. Raising the temperature by $12 \,^{\circ}$ C in the experiment intensified corrosion also in the rest of the electrodes and restored the equal speed of corrosion in all electrodes, thereby turning wood volume resistance back to positive.

When conducting measurements in a four-electrode measuring system (see Fig. 2) in DC mode, experiment results indicate a problem that volume resistance R_v (values calculated according to formula (7b) may be somewhat underestimated. Therefore, volume resistance R_v time graph (see Fig. 6) functions better as an indicator of corrosion. For a more accurate determination of volume resistance R_v , measurements of resistances R_1 and R_2 should be carried out in parallel both in DC mode and AC mode.

Figure 8 suggests that in wood moisture contents above FSP, the relative increment of polarization resistance may reach 120%. If the initial resistance R_0 is viewed as approximately equal to wood resistance, the maximum wood resistance measurement error, upon not considering polarization resistance, may also reach 120%. Figure 8 also shows that polarization resistance as well as the relative increment of polarization resistance, depending on time, is rather well-fitted with the logarithmic function. When measuring resistance in a logarithmic scale, polarization resistance can be quite easily subtracted from wood resistance by using instead of R_0 its calculated value R_{0calc} (regression curve value at time 0.1 sec (t = 0.1 sec)). R_{0calc} values given in Table 1 have been calculated on the basis of regression curves adjusted for 60-second intervals. For obtaining R_{0calc} more frequent measurements could be carried out during the first second of the polarization process as an alternative (e.g. 10-100 measurements per second) and then R_{0calc} may be calculated on the basis of a linear function fitted to measured data.

Table 1 shows intervals of wood resistance plus polarization resistance values in comparison to data measured at the same time with Scanntronik Gigamodule, which have been recalculated from the logarithmic scale 10logR into resistance R, given in megaohms. According to Table 1, coordination between data on different resistance meters is satisfactory. Yet, it is unknown how the polarization resistance raw file in the Scanntronik Gigamodule resistance meter has been processed and whether and in which manner residual polarization volt-

Table 1. Quantities characteristic of the polarization process.Tabel 1. Polarisatsiooni protsessi iseloomustavad suurused.

Wood moisture Puidu niiskus (%)	R0, $(M\Omega)$, t = 0 sec	$R_{max} (M\Omega), t = \sim 600 sec$	$RO_{calc} (M\Omega), t = 60 sec$	Gigamodule, R (MΩ)	((R-R0) / R0) 100%
146	0.017	0.043	0.011	0.054	126
97.2	0.172	0.227	0.147	0.151	32.6
36.4	0.59	0.73	0.52	0.56	23.7



Figure 8. Relative increment of polarization resistance in pine sapwood at three different moisture contents: MC1 = 146%; MC2 = 97.2% and MC3 = 36.4%

Joonis 8. Polarisatsiooni takistuse suhteline juurdekasv männi maltspuidus erinevatel niiskussisaldustel: NS1 = 146%; NS2 = 97.2% ja NS3 = 36.4%.

age as a possible source of a systematic error has been considered.

Figures 9 and 10 depicts that depolarization lasts longer in the case of wet wood, for ca 600 seconds. Measurements should continue only after the end of the depolarization process. Depolarization initial voltage relation U_1/U_0 and $T_{max'}$ the duration of the depolarization process also depend on wood moisture content according to Figure 9 and Table 2. Instead of using initial voltage U₁, more accurate calculations would be obtained by using its calculated value U_{1calc} (that is, the extrapolated value of the regression curve at t = 0.1 sec). Based on the above, it may be assumed that in the case of an equivalent circuit highly descriptive of the depolarization process, this process could also be used for measuring wood electrical resistance and wood moisture content.

Figure 11 shows that residual polarization voltage between measuring electrodes is relatively stable (average –28.5 mV), but suggests a downward trend as wood moisture content decreases. For example, residual polarization voltage may lead to a systematic measurement error when measuring wood resistance at input measuring voltage 3 V of the resistance meter, depending on measuring voltage polarity up to \pm 1%, and at measuring voltage 1 V up to \pm 3%, also in resistance measuring units, because the resistance scale has a linear connection with the input voltage.

Linear dependence (in a logarithmic scale) between moisture content and wood electrical resistance in the pine specimen is shown in Figure 12. Based on data given in Figure 12, it may be suggested that Stamm's well-known empirical relation (formulas 3 and 5) applies also in pine wood moisture contents above FSP (above MC 30%). According to a common belief, this pattern is satisfactory only in the MC range of 7-30% (Norberg 1999). In the future, further research should determine the role played by the specific drying regime used in the experiment and tree species as well as the general (selected independently from the drying regime) empirical relation between wood electrical resistance and moisture content in the area above FSP in producing this quite a surprising result.



Figure 9. Changes in depolarization voltage of pine sapwood at three different moisture contents: MC1 = 146%; MC2 = 97.2% and MC3 = 42.8%





Figure 10. Depolarization initial voltage relation U_1/U_0 (initial voltages U_1 and U_0). Joonis 10. Depolarisatsiooni algpingete U_1/U_0 suhe (algpinged U_1 ja U_0).

Table 2.	Quantities characteristic of the depolarization process.
Tabel 2.	Depolarisatsiooni protsessi iseloomustavad suurused.

Wood moisture Puidu niiskus, (%)	$(U_1 / U_0) 100\%$	$T_{max}(U_1 = 0),$ sec	(U _{1,calc} / U ₀) 100%, t = 60 sec	$(U_1 / U_0)T_{max}$, sec
146	7.4	600	10.37	44.4
97.2	4.5	488	6.66	21.96
42.8	2.5	335	3.33	8.38



Figure 11. Changes (variation) in residual polarization voltage measured in the specimen during drying. The moisture content of the specimen varied respectively from 146% to 36.4%.

Joonis 11. Männi maltspuidust katsekeha kuivatamise käigus mõõdetud jääkpolarisatsiooni pinged. Katsekeha niiskussisaldus (NS) muutus alates 146% kuni 36.4%.





Joonis 12. Empiiriline seos männi maltspuidu niiskussisalduse ja elektritakistuse vahel temperatuuril 32 °C. Katsekeha niiskussisaldus vähenes 141% kuni 36.4%.

For the specific drying regime and type of pin electrodes used in the experiments, the average electrode/wood contact area of both electrodes was A = 94.8 mm² and distance between electrodes was L = 30 mm. Based on this data, cell constant K, and based on Figure 1, also constants in formula (5) could be calculated: $C_1 = 7.7783$ and D = -1.3233. For the Stamm formula (3), respec-

tive values of the constants are the following: C = 5.63 and D = -1.3233. Formula (6) was applied for calculating the value of constant C. All constants have been determined for pine sapwood and the constant temperature of 32 °C.

By comparing the figures given in Tables 1 and 2, the estimated average moisture sensitivity of different measuring processes

Table 3. 2S values of MC readings (acc. to Rozema, 2010) of wet and dry wood (above FSP and below FSP) measured with different moisture meters. Number of specimens n = 60x5.

Tabel 3.	2S väärtused	erinevate	niiskusmõõtjatega	saadud	NS	lugemitele	(vastavalt	Rozema,	2010	artik-
	lile).									

Type of Mois- ture meter Niiskusmõõtja tüüp	Scots pine heart- wood, average actual MC wet 34.2% dry 27% Männi lülipuit keskmine tegelik niiskussisaldus märg kuiv	Norway spruce heartwood, aver- age actual MC wet 31.3% dry 27.1% Kuuse lülipuit kesmine tegelik niiskussisaldus märg kuiv	Grey alder, av- erage actual MC wet 105.7% dry 31.6% Hall lepp kesmine tegelik miiskussisaldus märg kuiv	Birch, average actual MC wet 85.2% dry 30.3% Kask keskmine tegelik niiskus- sisaldus märg kuiv	European aspen, average actual MC wet 107% dry 29.9% Haab keskmine tegelik niiskus- sisaldus märg kuiv
GANN HT 85 T 2S-value	3.8 2.4	2.4 1.0	2.6 1.6	4.2 2.8	2.6 3.5
FMD-6 2S-value	3.0 2.2	1.8 1.0	7.4 1.4	2.4 2.2	1.8 3.5
NDT James,	2.2 2.6	3.2 2.6	2.2 2.8	2.2 2.8	2.0 2.7

Bold numbers – Not measurable, if 2S-value > 3.5%.(acc. to Rozema, 2010).

MC - moisture content (%), determined by indirect (electrical resistance) and direct (oven-dry) methods.

Paksud numbrid – Mitte mõõdetav, kui 2Š-väärtus > 3.5% (vastavalt Rozema, 2010 artiklile).

NS – niiskusisalduse (%), mis on määratud kaudsel (elektritakistuse) ja otsesel (kuivkaalumise) meetodil.

may be determined. A two-fold increase in wood moisture content (97.2% up to 146%) brought about a corresponding increase in depolarization initial voltage relation $((U_1/U_0)100\%)$, a four-fold increase in the relative increment of polarization resistance ((R-R0)/R0)100%), and an eight-fold increase in the measured electrical resistance R0. Thus, the measurement of electrical resistance seems to involve the highest moisture sensitivity, followed by the measurement of the relative increment of polarization resistance, and lastly, the measurement of depolarization initial voltage relation. In the practical use of the methods for measuring wood moisture content, the moisture sensitivity of a method is not the only selection criterion. It is also essential to minimize the variance of the measurement data recorded. A more detailed research of moisture sensitivity and variance would require more measurement data.

A comparison of the values of quantity 2S by tree species in Table 3 indicates a trend in all tree species that 2S values decrease in accordance with a decrease in wood moisture content. Aspen wood is the only exception with its increasing 2S values. It might be that drying results in larger moisture gradients in aspen wood compared to other tree species. This phenomenon with aspen wood requires in-depth future research.

The analysis by DC measuring mode enables adding the following variables in the multivariate statistical model for calibration: 1) gravimetric wood MC, 2) wood/ electrode contact resistance, 3) volumetric resistance of wood, 4) relative increment of polarization resistance, 5) time constant of the polarization process at given wood moisture levels, 6) depolarization initial voltage relation, 7) time constant of the depolarization process at given wood moisture levels, 8) multiplication of the depolarization initial voltage relation and total process time, 9) residual polarization voltage (or charge). All variables are defined on the assumption that the temperature is constant.

Conclusions

Knowledge of electrode effects enables improved consideration and prevention of random factors reducing calibration quality of resistance type wood moisture meters in wood moisture contents above FSP.

To improve the accuracy of measurement of wood electrical resistance the study presented a novel approach. The additional variables were generated for the multivariate statistical model for calibration. Application of these variables presumably improves the R^2 value of the model and minimises its standard error compared to the simple calibration model with one independent variable ($y \sim x$).

The outcomes of the study were approximate error estimates and suggestions for five different types of electrode effects, which should be considered when organising the calibration process of resistance type wood moisture meters in the range above FSP.

Acknowledgements. The work was supported by the Environmental Investment Centre of Estonia (Grant No. 906).

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Elektroodefektide uurimine puidu kuivatamisel niiskussisaldustel üle kiu küllastuspunkti

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Kokkuvõte

Puidu kuivatuse monitooringul on elektriline takistusmeetod laialdaselt kasutusel. Kuid probleeme tekitab puidu niiskussisalduse (NS) määramine üle kiu küllastuspunkti (s.o. NS vahemikus 30% kuni 170% puidu kuivkaalu suhtes), kus on põhilisteks probleemideks ebapiisav mõõtetäpsus ja tulemuste vähene korratavus, mille tõttu pole seni õnnestunud saada sellele meetodile mainitud piirkonnas rahuldavat kalibreerimiskõverat. Selle artikli eesmärgiks oli eksperimentaalselt uurida puidu ja mõõteelektroodide vastastikust toimet. Elektroodefekte uuriti puidu kuivatamisel üle kiu küllastuspunkti, madalatel temperatuuridel (20 °C ja 30 °C) ja leebetel kuivatusrežiimidel (98% RH ja 96% RH õhu suhtelisel niiskussisaldusel). Katsetes kasutati kahe erineva tootja elektrilise takistuse mõõtjat ja kolme erineva tootja takistus-tüüpi puidu niiskuse mõõtjat. Ühesugustes tingimustes (32 °C ja 96% RH ja puidu NS piirkonnas üle kiu küllastuspunkti), katsekehi kuivatati ja teatud ajavahemike järel mõõdeti niiskussisaldusi viiest erinevast puiduliigist (Pinus sylvestris, Picea abies, Alnus glutinosa, Betula pendula, Populus tremula) valmistatud katsekehadel mõõtmetega 100×60×60 mm (pikkus, laius, paksus). Kokku oli eksperimendis 500 katsekeha. Statistiliselt analüüsiti elektroodide korduva paigaldamise mõju mõõtmistulemuste korratavusele. Männi maltspuidus (niiskussisalduste piirkonnas üle

kiu küllastuspunkti) uuriti lisaks ka mõõteelektroodide korrosiooni, polarisatsiooni ja depolarisatsiooni protsesside mõju elektritakistuse mõõtetulemustele. Selle uurimuse peamiste tulemustena võib esile tõsta järgmisi tulemusi: 1) Määrati eksperimentaalselt Stamm'i valemi koefitsiendid uuritava (üle kiu küllastuspunkti) puidu niiskuste vahemiku jaoks. 2) Stamm'i valem annab funktsionaalse sõltuvuse puidu niiskussisalduse ja puidu elektritakistuse vahel. Lisaks sellele sõltuvusele (nn. Stamm'i protsess), leiti veel kaks protsessi (polarisatsiooni ja depolarisatsiooni protsessid), mis samuti osutusid puidu niiskusest sõltuvaiks. Kõigi kolme protsessi jaoks leiti ligikaudsed niiskustundlikkuste suhtarvud. 3) Leiti lihtne protseduur puit/elektrood kontakti takistuse ja puidu ruumtakistuse eraldamiseks (e. separeerimiseks). 4) Katsetulemuste alusel valiti välja olulisemad füüsikalised parameetrid, mis võivad edaspidi leida rakendust puidu niiskusmõõtjate kalibreerimisel mitme muutujaga statistilistes mudelites. Selles uurimuse tulemuste interpreteerimise käigus püstitati ka kaks hüpoteesi üks jääkpolarisatsiooni nähtuse selgituseks, ja teine Stamm'i protsessi niiskustundlikkuse selgituseks. Selle uurimuse tulemusi on võimalik kasutada takistus-tüüpi puidu niiskusmõõtjate kalibreerimise protseduuri organiseerimisel puidu niiskussisaldustel üle kiu küllastuspunkti.

Received August 28, 2013, revised September 5, 2013, accepted September 16, 2013

IV

Tamme, V., Muiste, P., Tamme, H. 2013. Experimental study of resistance type wood moisture sensors for monitoring wood drying process above fibre saturation point. *Forestry Studies*, 59: 28–44.



Experimental study of resistance type wood moisture sensors for monitoring wood drying process above fibre saturation point

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Tamme, V., Muiste, P., Tamme, H. 2013. Experimental study of resistance type wood moisture sensors for monitoring wood drying process above fibre saturation point. – Forestry Studies | Metsanduslikud Uurimused 59, 28–44. ISSN 1406-9954. Journal homepage: http://mi.emu.ee/forestry.studies

Abstract. In wood moisture contents above fibre saturation point (FSP) the measuring accuracy of resistance type sensors starts decreasing and several side effects related to measuring wood moisture begin to occur. These side effects can be taken into account in the measuring process and can be eliminated. This research examined wood charging and discharging in the process of measurements, phenomena related to repeated measurements in both a single electrode insertion spot and different insertion spots in the specimen made of pine (Pinus sylvestris L.) sapwood and black alder (Alnus glutinosa (L.) Gaertn.). The research presented approaching equations separately for the voltage and current of the polarization and depolarization process for the initial phase of the process. Empirical equations were found for wood electrical resistance and electrical capacitance in the polarization process. For black alder possibilities for calibration of a resistance meter and resistance type electrodes were explored. It was found that to improve the calibration accuracy of resistance type measuring electrodes an additional individual calibration with regard to the average moisture content of the specimen may be carried out with the electrodes above FSP. Research results can be implemented in monitoring the wood drying process using resistance type wood moisture sensors, but also in determining the electrical parameters of growing trees.

Key words: wood drying, above FSP, corrosion, polarization.

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Introduction

The electrical resistance method is widely used for monitoring wood drying since the method is economical and reliable (Tronstad *et al.*, 2001; Onysko *et al.*, 2008). However, the method is deficient due to the fact that above fibre saturation point (FSP) measuring accuracy is insufficient and starts decreasing correspondingly to the increasing wood moisture content (Edwards, 1974; James, 1993; Vermaas, 2002). The decrease in measuring accuracy can also be described quantitatively, with the 2S-value (Rozema, 2010; Tamme *et al.*, 2012) or by statistical modelling (Tamme *et al.*, 2013). Insufficient measuring accuracy above FSP may also reduce the accuracy of effective diffusion coefficient monitoring in the process of wood drying (Tamme *et al.*, 2010, 2011).

In the measurement of wood direct current (DC) resistance, the following factors are important (Tamme *et al.*, 2012): (a) electrical resistance of the galvanic contact between the measuring electrode and wood, (b) corrosion of measuring electrodes in wet wood, (c) polarization and depolarization of the double layer forming on the surface of the measuring electrodes and of the polar molecules of wood itself, (d) residual polarization voltage between measuring electrodes, and lastly, (e) effect of repeated application (repeated measurements) of measuring electrodes.

(a) electrical resistance of the galvanic contact between the measuring electrode and wood.

The main empirical relationships and definitions of DC have been presented in studies by Norberg (1999), Stamm (1927) and Straube *et al.* (2002) and modified in the study by Tamme *et al.* (2012).

(b) Corrosion of measuring electrodes in wet (MC above FSP) wood.

According to Eco Chemie (2013) "The corrosion of iron [measuring electrodes] to form rust proceeds according to the overall reaction:

$$2 Fe + H_2O + O_2 -> 2 Fe(OH)_2$$
 (1)

This reaction includes the dissolution of iron, the reduction of oxygen and formation of rust:

$$Fe - > Fe^{2+} + 2e^{-}$$
(anodic) (2)

$$2 H_2O + O_2 + 4 e^{-} - > 4 OH^{-}$$
(cathodic) (3)

$$Fe^{2+} + 2 OH^{-} - > Fe(OH),$$
(chemical) (4)

The corrosion of measuring electrodes in wet wood may be described by the Butler-Volmer equation (Ecochemie, 2013):

$$i = i_{corr} \left(e^{\frac{2.303 \frac{\eta}{b_a}}{b_a}} - e^{\frac{2.303 \frac{\eta}{b_c}}{b_c}} \right)$$
(5)

where
$$\eta = E - E_{corr}$$
 (6)"

The Tafel constants b_a and b_c in the Butler-Volmer equation and corrosion current i_{corr} are usually determined in alternate current (AC) mode with an electrical impedance spectrometer (EIS).

Metal corrosion in wood has been examined by the electrical impedance spectrometry (EIS) method in the research by Zelinka & Rammer (2006). For measuring wood MC in DC mode and above FSP, the research by Tamme *et al.* (2012) gives a qualitative description of the corrosion of measuring electrodes for a measurement system with four electrodes. It was offered using the measurement system with four electrodes as an indicator of the corrosion of measuring electrodes. The intersection of wood MC monitoring graphs as a characteristic feature of the corrosion of measuring electrodes is presented also by Lazarescu *et al.* (2010) (see Figure 9) which also applied the measurement system with four electrodes.

(c) polarization and depolarization of the double layer forming on the surface of the measuring electrodes (Zelinka et al., 2007; Eco Chemie, 2013) and of the polar molecules of wood itself.

In measuring wood electrical resistance, electrical voltage (difference between potentials) is applied to the measuring electrodes. The applied voltage generates an electrical field between the electrodes in wood, causing the polar molecules of wood and water to orient toward the electrical field and free charge carriers with different polarity to migrate. In addition, a double layer with a thickness of a few nanometers is formed on the surface of the electrodes (Eco Chemie, 2013). To put it in simple terms, wood is electrically charging during the measuring cycle of electrical resistance. This is called the wood polarization phase or polarization process. The voltage applied to the measuring electrodes at the end of the electrical resistance measuring cycle is switched off and this marks the start of the discharging phase of the electrically charged wood, which is called the wood depolarization process. Both processes, polarization and depolarization, can be measured using relatively simple methodology in the experiment (Figure 1).

Using Ohm's law, the voltage drop (Tamme *et al.*, 2012; Eco Chemie, 2013) for black alder *Alnus glutinosa* (L.) Gaertn. by Figures 7:

$$\Delta E_{ohmic} = iR_{\Omega} \tag{7}$$

$$E_{actual} = E_{applied} - iR_{\Omega} \tag{8}$$

When monitoring the potential (Tamme *et al.*, 2012) the true potential lags the applied potential according to the following equation:

$$E_{actual} = E_{applied} \left(1 - e^{-\frac{t}{R_{\Omega}C_{dl}}} \right),$$
(9)

where R_{Ω} is the Ohmic resistance, C_{dl} is the double layer capacitance, and *t* is the time, at which the measurement is taken.

For depolarization process, where $E_{applied} = 0$, the potential lags according to the following equation:

$$E_{actual} = E_0 e^{\frac{-t}{RC}},\tag{9a}$$

where E_0 – potential at starting moment (t = 0) of the depolarization process.

The study by Tamme *et al.* (2012) obtained measurement results with intervals of 0.1 second and 1 second. Either the moment of applying voltage to the measuring electrodes (when measuring the polarization process) or switching off the voltage (when measuring the depolarization process) was chosen as the starting point (t = 0) of the measurements.

Assuming a pure RC circuit (see Figure 1, test scheme) for the capacitor the *ln(i)* plot will give a straight line according to:

$$i = \frac{\Delta E e^{\frac{-t}{RC}}}{R} \tag{10}$$

$$\ln(i) = \ln(\frac{\Delta E}{R}) - (\frac{1}{RC})t \tag{11}$$

The multiplication *RC* of resistance and capacitance, which has a time dimension, is called the time factor τ (tau) of the charging-discharging process. In general, time factors are different in the charging and discharging process (Tamme *et al.*, 2012).





Puidu takistus

Wood Resistance

of Double Layer of Electrodes Figure 1. Experimental setup circuit diagram. V –

- DC Ahlborn voltmeter, type ZA9000-FS3, \pm 2.6 V. E₁ and E₂ – electrometer Keithley Model 6517B, Switches S₁, S₂, and S₃ – relay contacts.
- Joonis 1. Katseseadme põhimõtteskeem. V alalisvoolu voltmeeter "Ahlborn", tüüp ZA9000-FS3, \pm 2.6 V. E_1 and E_2 – elektromeeter "Keithley Model 6517B", S_1 , S_2 , and S_3 – lülitid (relee kontaktid).

Polarization and depolarization processes in wood have a complex physical background. James (1975) has named three classes of mechanisms to explain the dielectric properties of wood: (a) mechanisms with short relaxation times including electronic, atomic, and fast molecular polarizations; (b) mechanisms with intermediate relaxation times including slow molecular, fixed dipole, and fast interfacial polarizations; and (c) mechanisms with long relaxation times including slow dipole and interfacial polarizations. James' analysis was based on different polarization times of molecules. The general description of dielectric polarization has been given in

a course on theoretical physics (e.g. Save-lyev, 1975).

(d) residual polarization voltage between measuring electrodes.

Tamme *et al.* (2012) determined the value of residual polarization voltage and considered the different electrical mobility of positive and negative charge carriers in wood as well as the difference between corrosion potentials as the possible root cause for residual polarization voltage.

(e) effect of repeated application (repeated measurements) of measuring electrodes.

The effect of repeated relocation of insulated pin electrodes in new measuring areas when measuring wood electrical resistance has been quantitatively analysed with the S-value (Rozema, 2010; Tamme *et al.*, 2012) and the statistical modelling method (Tamme *et al.*, 2013). The last study quantitatively evaluated the progressive decrease in the measuring accuracy of some widely used wood moisture meters upon an increasing wood average moisture content from 30% up to 100% (above FSP).

Two hypotheses were proposed for this research:

- 1. *Hypothesis H1*. It is possible to qualitatively describe the equivalent circuit of a resistance-type wood moisture sensor by making measurements and calculations (see Figure 1).
- 2. *Hypothesis H2*. The statistical model enables predicting wood average moisture content for a specific and accurately repeated drying schedule between wood moisture contents of 26% MC and 90% MC.

Material and Methods

Hypothesis H1 testing methodology

Electrical resistance is determined for an equivalent circuit directly with a resistance meter, as described in the research by Tamme *et al.* (2012). If the applied working

voltage is known, the current strength flowing through wood can be calculated using Ohm's law. Another option is to measure the voltage applied to measuring electrodes and current strength flowing through wood, and then calculate wood electrical resistance using Ohm's law. The sum of (different) electrical capacitances in the equivalent circuit is calculated using measurement results and empirical equations (12 and 13).

To examine wood polarization and depolarization processes on the same black alder specimen (20 °C, 96% RH) an experimental setup was used, the circuit diagram of which is given on Figure 1. The Ahlborn 2.6 V DC voltmeter (type ZA9000FS3) with an input resistance of 10 MegaOhms was used in the experimental setup. As electrometers E_1 and E_2 the Keithley Model 6517B electrometer was used according to the positions of switches S_1 , S_2 and S_3 (Keithley, 2013). The Keithley Model 6517B electrometer enables selecting between four measuring modes: voltage U, current I, resistance Ω , and charge Q.

Hypothesis H2 testing methodology

The testing method involves producing a statistical prediction model for a specific tree species and specimen dimensions, given drying schedule and average moisture content range of the specimen. The reliability of the model is assessed with tests on the normality of regression residuals and visually by using the probability paper. Model testing may be supported by the individual calibration method for pin electrodes, as seen in researches by Tamme *et al.* (2012) and Lazarescu *et al.* (2010). Details on model repeatability conditions (i.e. validation) have been analysed in the research by Tamme *et al.* (2013).

The experiments made use of a black alder specimen with the dimensions of $500 \times 120 \times 35$ mm (length x width x thickness), which was conditioned in the Feutron climatic chamber (Feutron, 2013) at a room temperature of 20 °C and relative humidity of 96% RH for 72 hours and there-

Kuivatuse aeg /	Kuivatuskambri temperatuur /	Kuivatuskambris suhteline õhuniiskus / Relative humidity (RH %)
Drying time, (ii)		Retacive numberly, (RT 70)
0	20	60
12	47	95
24	50	90
48	52	80
72	52	69
96	52	59

Table 1. Drying schedule of 35 mm black alder drying (96 h test).

Tabel 1.	Kuivatusplaan	35 mm	paksuseaa	musta lena	materiali	kuivatamiseks	(96	tunnine	katse)
abel 1.	Nurvacusptuuri	<i>55</i> mm	punsuscyu	mustu iepu	materjati	Ranvacannischs	(20	cumme	nucse)	٠.

after at an average temperature of 50 °C for another 96 hours according to the drying schedule (Table 1). The ends ($120 \times 35 \text{ mm}$) and sides ($500 \times 35 \text{ mm}$) of the specimen were coated with nitrocellulose lacquer to avoid water vaporisation. By adopting this measure, one-dimensional (1D) moisture distribution was ensured in the specimen.

The specimen had been sawn from the black alder log in a way that the length of the specimen was oriented along the grain, the width across the grain in tangential direction and the thickness across the grain in radial direction. The sawn specimen was located approximately 8 cm from the pith of the log.

The average air velocity in the conditioning phase (that is, the first 72 hours and at 20 °C room temperature) was the natural convection air velocity in the chamber, whereas when drying according to the drying schedule, the average air velocity was 2 m s⁻¹. Wood electrical resistance measurements were conducted with one specimen in 80 different pin electrode insertion spots and at an equal measuring depth of 12 mm of the surface of the specimen (that is, 1/3of the thickness of the specimen). Due to the short duration of measurements (not more than one minute in the spot of application of the electrodes), the effect of electrode corrosion was neglected.

The specimen was weighed on a scale (Mettler-Toledo, type PB3002-S/Fact, readability \pm 0.01 g) (Mettler-Toledo, 2013) after each electrical resistance measure-

ment. The specimen was oven-dried and weighed using the same Mettler-Toledo scale. The relative moisture content of the specimen according to dry mass was determined in compliance with the standard ISO 3130:1975. Based on dry weighing and electrical resistance measurement data a statistical regression model was later produced for the specimen dried according to a specific drying schedule. Teflon insulated pin electrodes (ram-in electrodes M18) with a length of 40 mm manufactured by Gann (Gann, 2013) were used as measuring electrodes in the experiments. A resistance meter "Scanntronik Material Moisture Gigamodule" (Scanntronik, 2013) was used to measure wood electrical resistance. A Rotronic sensor (type Rotronic Hygro Clip-S, measuring range 0 - 100%, precision at 23 °C ± 1.5%) (Rotronic, 2013) was used to check the drying air humidity and temperature. An Ahlborn air velocity sensor (type FVA935-TH4, measuring range 0 – 2 m s⁻¹) (Ahlborn, 2013) was used to check the velocity of drying air.

Results and Discussion

Hypothesis H1 testing results and discussion

(*a*) Results of testing pine (Pinus sylvestris L.) sapwood.

Measurement data in the research by Tamme *et al.* (2012) (see Figure 4) were used to determine the polarization current

in the polarization (or wood charging) process, the time dependence of which is given in Figure 2. The polarization current of the pine sapwood specimen was determined at the average moisture content level of 146% and temperature of 20 °C.

The polarization process of the specimen was examined in more detail for 20 seconds starting from the beginning of the process (that is, the initial phase of the polarization process). The observation period of 20 seconds was selected because the resistance meter Scanntronik (Scanntronic, 2013) uses a measuring cycle of the same duration. Since the theoretical equation (10) fails to approach a 20-second polarization process with sufficient accuracy, the empirical equation

$$I = I_0 \exp(\frac{-t}{\tau_0 + 0.17t}), \qquad (12)$$

where $\tau_0 = 5.15$ s and $I_0 = 65.81$ µA, was found to approach the process more accurately. Graphs showing the experimentally determined polarization current, the polarization current calculated with the theoretical equation (10) and the polarization current calculated with the empirical equation (12) are given in Figure 3 in the same scale. The dependence of the time factor τ = RC (see equations 10 and 12) of the polarization process on time is given in Figure 4. The figure shows that the dependence of the time factor on the polarization time, from the starting moment, is practically linear (R-squared 0.99). Taking into account that polarization resistance had been experimentally determined by direct measurement, the time factor definition equation enables to determine also the dependence of the summarised electrical capacitance C of 146% MC pine sapwood and double layer of electrodes on time, which is shown in Figure 4.



Figure 2. Pine sapwood polarization current at 146% MC of the specimen and temperature of 20 °C.

Joonis 2. Männi maltspuidu polarisatsiooni vool katsekeha keskmisel niiskussisaldusel 146% ja temperatuuril 20 °C.



Figure 3. Graphs of 146% MC pine sapwood showing the experimentally determined polarization current (I(μ A)), the polarization current (Ipol(tt)) calculated with the theoretical equation (10) and the polarization current (Ipol(tau(t))) calculated with the empirical equation (12) in a single scale.

Joonis 3. Eksperimendist määratud polarisatsiooni voolu I (μA), teoreetilise valemi (10) alusel arvutatud polarisatsiooni voolu Ipol(tt) ja empiirilise valemi (12) alusel arvutatud polarisatsiooni voolu Ipol(tau(t)) graafikud samas teljestikus.

Physical considerations make it obvious that neither polarization resistance R, polarization capacitance C nor the time factor τ (tau) can increase limitlessly; these quantities must approach asymptotically to a certain limit value or plateau.

The research (Tamme *et al.*, 2012) (see Figure 9) presents also pine sapwood depolarization process graphs at 20 °C. A more detailed analysis of the depolarization process at 146% MC (i.e. graph corresponding to MC1 in Figure 9) allowed to determine the empirical equation:

$$U = U_0 \exp(\frac{-t}{\tau_0 + 0.6t})$$
 (13)

where $\tau_0 = 4.685$ s and $U_0 = 0.411$ V for the 20 seconds of the initial phase of this process,



Figure 4. Time dependence of the summarised electrical capacitance C of 146% MC pine sapwood and double layer of electrodes of the depolarization process.

Joonis 4. 146% niiskussisaldusega männi maltspuidu ja elektroodide kaksikkihi summaarse elektrimahtuvuse C sõltuvus ajast.

which approaches the time dependence of depolarization voltage better than the theoretical equation (9a). The experimentally determined depolarization voltage U, voltage calculated with the theoretical equation (9a) and depolarization voltages calculated with the empirical equation (13) for 146% MC pine sapwood are given in the same scale in Figure 5. Figure 6 shows the dependence of the time factor of the same depolarization process (the one in Figure 5) on time, which proved practically linear in the initial phase of the depolarization process. Physical considerations make it obvious that also the increase in the time factor of the depolarization process needs to slow down, and the time factor has to asymptotically approach a certain limit value or plateau. As depolarization current was not measured in the research (Tamme et al., 2012), the summarised electrical capacitance C of wood and electrodes based on the found time factor unfortunately deemed impossible in the depolarization process.



- Figure 5. The experimentally determined depolarization voltage U, -legend (V(MC1)), voltage calculated with the theoretical equation (9a) (Ucalc(tt)) and depolarization voltages calculated with the empirical equation (13) (Ucalc(t1)) for 146% MC pine sapwood in a single scale.
- Joonis 5. Eksperimendist määratud depolarisatsiooni pinge U (-legend V(MC1)), teoreetilise valemiga arvutatud pinge (9a) (-legend Ucalc(tt)) ja empiirilise valemiga (13) arvutatud depolarisatsiooni pinged (-legend Ucalc(t1)) 146% männi maltspuidu jaoks samas teljestikus.



Figure 6. Dependence of the depolarization time factor (shown in Figure 5) of 146% MC pine sapwood on time.

Joonis 6. 146% niiskussisaldusega männi maltspuidu depolarisatsiooni protsessi (vt. joonis 5.) ajateguri sõltuvus ajast.



Figure 7. General shape of the polarization-depolarization measuring cycle in the voltage U scale for black alder (90% MC and 20 °C).

Joonis 7. 90% niiskussisaldusel ja temperatuuril 20 °C sanglepa puidu jaoks polarisatsiooni – depolarisatsiooni mõõtetsükli üldkuju pinge U teljestikus.

(a) Results of testing black alder.

To examine wood polarization (electrical charging) and depolarization (electrical discharging) processes and improve the measuring methodology a few experiments were carried out involving another tree species (black alder) at 90% MC and temperature of 20 °C. The general shape of the polarization-depolarization measuring cycle in the voltage graph for black alder is given in Figure 7. The transition from the polarization phase to the depolarization phase shown in Figure 7 involves a voltage drop or the Ohmic drop which can be described with the equation (9). Figure 8 shows the dependence of polarization current on time, which was measured directly with the electrometer E_1 . Figure 9 shows the dependence of polarization resistance on time for black alder (90% MC and 20 °C).





Joonis 8. Elektromeetriga E, mõõdetud sanglepa puidu polarisatsiooni voolu sõltuvus ajast 90% niiskussisaldusel ja temperatuuril 20 °C.

During the measurement switches S_1 and S₂ were closed and S₃ was in the off position (see Figure 1). Figure 10 shows the two consecutive measurements of depolarization current and depolarization voltage, which are depicted in a single scale. During these measurements, switch S₁ was open and switches S₂ and S₃ were in the closed position. Based on the depolarization voltage and depolarization current measurement data, Figure 11 shows the dependence of black alder electrical resistance, calculated according to the Ohm's law, on time in the depolarization process. Figure 11 shows that the electrical resistance of the black alder specimen with 90% MC increases in the initial phase of the depolarization process but later approaches a certain value. In the final phase of the process, current and voltage measurement errors also become major since choosing a more sensitive measuring area for the electrometer is not possible without interrupting the process.

Some technical considerations regarding the methodology for measuring the processes of polarization and depolarization:

(a) Depolarization current and depolarization voltage should be measured simultaneously and in a similar magnitude to that of measuring instruments with input resistance. The logical solution would be to measure depolarization current indirectly, that is, use the electrometer E_2 in the charge Q measuring mode, and calculate depolarization current with the following equation:

$$I(t)_{dpol} = \Delta Q / \Delta t \tag{14}$$

Unfortunately the largest measuring range of the measuring mode of the charge of the electrometer E_2 used in the experiments proved too sensitive for the specific experiment.



Figure 9. Time dependence of polarization resistance for black alder (90% MC and 20 °C).

Joonis 9. Polarisatsiooni takistuse sõltuvus ajast sanglepa puidu jaoks 90% niiskussisaldusel ja temperatuuril 20 °C.



- Figure 10. Two consecutive measurements of depolarization current and depolarization voltage for black alder (90% MC and 20 °C), depicted in a single scale.
- Joonis 10. Depolarisatsiooni voolu ja depolarisatsiooni pinge kaks järjestikust mõõtmist sanglepa puidu jaoks (90 % NS ja 20 °C), mis on kujutatud ühises teljestikus.



- Figure 11. Time dependence of black alder (90% MC and 20 °C) electrical resistance in the depolarization process, calculated according to the Ohm's law based on the depolarization voltage and depolarization current measurement data.
- Joonis 11. Depolarisatsiooni pinge ja depolarisatsiooni voolu mõõtmisandmete alusel Ohmi seaduse järgi arvutatud sanglepa puidu elektritakistuse sõltuvus ajast depolarisatsiooni protsessis 90% niiskussisaldusel ja temperatuuril 20 °C.

(b) The residual polarization voltage mentioned in the research (Tamme *et al.*, 2012) occurred in the electrodes also in shortterm measurements where the influence of electrode corrosion may be disregarded. The more likely reason for residual polarization seems to be that electrical mobility is different for charge carriers of different polarity. The influence of residual polarization helped to significantly reduce the correctly completed depolarization (wood discharging) process.

(c) Numerous repetitions of polarizationdepolarization cycles may result in reduced concentration of charge carriers (partial exhaustion of charge carriers) in the electrode gap. Exhaustion of charge carriers may result in greater wood electrical resistance values in later measurements than with the same moisture content and electrical measurements in the case of electrodes yet undisturbed or inserted in the given spot for the first time. The abovementioned situation did not yet come about in two consecutive measurements: however, after six consecutive measurements an apparent rise occurred in black alder electrical resistance.

Hypothesis H2 testing results and discussion

Figure 12 shows a graph for individually calibrated needle electrodes for pine sapwood based on the data of the research (Tamme *et al.*, 2012). The parameters of this graph (regression line) are given in Table 2.

Individual calibration of pin electrodes may give satisfactory measuring accuracy also in wood MCs above FSP. A significant deficiency of the individual calibration method is, however, the need to carry out calibration (by the dry weighing method according to the standard ISO 3130:1975) in a new pin electrode insertion spot in wood every time and separately for each pair of electrodes, which, altogether, is a very labour-intensive procedure.



- Figure 12. Regression curve received for the black alder specimen based on 80 measurements at 50 °C, which, according to the average moisture content (MC%) of the specimen, enables predicting the average reading of the resistance meter.
- Joonis 12. Musta lepa katsekeha jaoks 80 mõõtmise alusel ja 50 °C temperatuuril saadud regressioonkõver, mille abil on katsekeha keskmise niiskussisalduse (NS%) järgi võimalik prognoosida takistusmõõtja keskmist näitu.

As an alternative to individual calibration of pin electrodes, the so-called prediction (or average measurement result prognosis) method is used. The prediction method involves statistical modelling or regression analysis of the relationship between two or more parameters. In the case of regression analysis, it is recommended to verify the reliability of the received model with regression diagnostics methods.

Figure 13 shows the regression curve received for the black alder specimen at 50 °C. The given regression model describes 95% of the variability (R-squared 0.95) of black alder wood electrical resistance (in units 10logR). In comparison, Figure 14 depicts the inverse regression curve received on the same conditions based on the average of 20 resistance meter readings for predicting the average moisture content of the black alder specimen. The reliability of the model has been verified by the conformity of prognosis residuals to normal distribution: visually, with the Q-Q

- Table 2. Regression parameters (for model 1 and model 2) and descriptive statistics parameters (for model 2).
- Tabel 2.
 Regressiooni parameetrid (mudeli 1 ja mudeli 2 jaoks) ja mõned kirjeldava statistika parameetrid mudeli 2 jaoks.

Parameeter / Parameter	Mudel / Model				
	Individuaalsel elektroodid NS Individually calib (according to Tar fig.	t kalibreeritud määramiseks / rated, (model 1) nme et al., 2012, 12)	Niiskussisaldust regressioon MC predicting re (fig.	(NS) prognoosiv nmudel 2 / gression model 2 12)	
Mõõtmiste arv / Observations	1	0	80		
Determinatsioonikordaja / R Squared	0.	0.99		0.95	
Mudeli standardviga / Standard error of regmodel	0.0	26	0.816		
Mudeli prognoosijääkide Kolmogorov-Smirnovi test / Kolmogorov-Smirnov test of residuals of regmodel			p-value :	= 0.6558	
Mudeli prognoosi jääkide Shapiro-Wilk test / Shapiro-Wilk test of residuals of regmodel	-	-	p-value	= 0.8622	
Mudel 2 jaoks tehtud mõõtmiste arv (4 x 20 mõõtmist) / Number of measurements (model 2) NS / MC% 10logR	20 20	20 20	20 20	20 20	
Mõõteseeriate aritmeetilised keskmised / Mean of NS / MC% 10logR	78.49 % 47.84	62.37 % 48.715	39.16 % 50.995	26.29 % 56.65	
Keskmiste standardvead / Standard error of NS / MC% 10logR	0.0437 % 0.1836	0.109 % 0.2094	0.073 % 0.219	0.026 % 0.112	
<i>Standardhälbed /</i> Standard deviation of NS / MC% 10logR	0.2 % 0.842	0.489 % 0.9366	0.327 % 0.98	0.1162 % 0.502	
Vahemikud /Range of NS / MC% 10logR	0.9 % 3.6	2.02 % 3.8	1.0 % 3.4	0.4 % 2.3	

plot (Figure 15), Kolmogorov-Smirnov test (Kolmogorov-Smirnov test, short ks. test) and Shapiro-Wilk test (short shapiro.test) using the statistics program R (R-project, 2013). Parameters of the regression model produced for black alder at 50 °C and some important descriptive statistics parameters are summarised in Table 2. The comparison between the individual calibration method (model 1) and prediction method (model 2) based on Table 2 shows that Rsquared of the regression model is considerably larger in the individual calibration model and the standard error considerably smaller than in the statistical model of the prediction method.

Note: Has been selected as the null hypothesis in the Kolmogorov-Smirnov test and Shapiro-Wilk test, stating that the examined distribution is not normal distribution. If the significance probability of the null hypothesis (p-value) is p > 0.05, the contrary hypothesis will apply.



- Figure 13. Inverse regression curve received for the black alder specimen based on a total of 80 measurements at 50 °C, which, according to the average reading of the resistance meter, enables predicting the average moisture content of the specimen. Based on the resistance meter (4 x 20) reading, this curve allows predicting average moisture contents of the specimen.
- Joonis 13. Musta lepa katsekeha jaoks kokku 80 mõõtmise alusel ja 50 °C temperatuuril saadud pöörd-regressioonkõver. Selle kõvera abil on takistusmõõtja (4 x 20) lugemi alusel võimalik prognoosida katsekeha keskmist niiskussisaldust.

To compare the details (e.g. for examining the variability of coefficients of the Stamm equation (3), etc.) of the individual calibration method and prediction method of insulated pin electrodes, a comparative experiment should be carried out with a specimen with the same dimensions and of the same tree species (black alder) and according to the same drying schedule by the individual calibration method of pin electrodes.

Combining the prediction method with the individual calibration method of insulated pin electrodes enables to receive individual calibration of electrodes also for the average moisture content of the specimen in moisture contents above FSP.

In conclusion it may be suggested that both hypotheses (H1 and H2) proposed for this research have been confirmed (validated) by results and discussion.

Conclusions

In addition to ordinary wood electrical resistance measurements, specific electrical



Teoreetilised kvanitiilid / Theoretical Quantiles

Figure 14. Q-Q plot of residuals for regression model 2 (Figure 12).

Joonis 14. Tõenäosuspaber regressioonmudeli (mudel 2, tabelis 2 ja joonisel 12) prognoosi jääkide normaaljaotuse hindamiseks.

measurements were carried out, as well. Real-time polarization resistance, polarization current, depolarization voltage and depolarization current were measured. The specific electrical measurements were performed at a temperature of 20 °C.

This examined several side effects involved in using resistance type wood moisture sensors in wood moisture contents above FSP, such as wood charging and discharging (polarization and depolarization and residual polarization) in the process of measurements, phenomena related to repeated measurements in both a single electrode insertion spot and different insertion spots in the specimen. The experiments made use of specimens made of pine sapwood and black alder wood. The research presented approaching equations separately for the voltage and current of the polarization and depolarization process for the first 20 seconds from the starting moment. Empirical equations were found for wood electrical resistance and electrical capacitance in the polarization process for describing the time dependence of these quantities in the initial phase of the process (within 20 seconds). The research also lists a few technical notes that need to be considered when repeating the experiments conducted in the research.

For black alder, possibilities for calibration of a resistance meter and resistance type electrodes into a wood moisture meter at 50 °C were explored. It was found that to improve the calibration accuracy of resistance type measuring electrodes, an additional individual calibration with regard to the average moisture content of the specimen may be carried out with the electrodes above FSP. Research results can be implemented in monitoring the wood drying process using resistance type wood moisture sensors in moisture contents above FSP, but also in determining the electrical resistance and electrical capacitance in growing trees.

Acknowledgements. The work was supported by the Environmental Investment Centre of Estonia (grant no. 906).

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Takistus-tüüpi puidu niiskuse andurite eksperimentaalne uurimine puidu kuivatamise monitooringul niiskussisaldustel üle kiu küllastuspunkti

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Kokkuvõte

Käesolevas töös uuriti takistus-tüüpi puidu niiskuse andurite kasutamisega puidu niiskussisaldustel üle kiu küllastuspunkti kaasnevaid mõjusid, nagu puidu elektriline laadumine (s.o. puidu polarisatsioon) ja tühjaks laadumine (s.o. puidu depolarisatsioon koos jääkpolarisatsiooniga) mõõtmiste protsessis. Veel uuriti selles töös praktika seisukohalt olulisi korduvate mõõtmistega seotud nähtusi nii elektroodide ühes konkreetses paigalduskohas kui ka elektroodide erinevates paigalduskohtades katsekehal. Katsetes kasutati männi (Pinus sylvestris L.) maltspuidust ja musta lepa (Alnus glutinosa (L.) Gaertn.) puidust valmistatud katsekehasid. Töös esitati lähendusvalemid polarisatsiooni ja depolarisatsiooni protsessi pinge ja voolu jaoks esimese 20 sekundi jooksul alates stardimomendist. Polarisatsiooni protsessi jaoks leiti puidu elektritakistuse ja puidu elektri-

mahtuvuse empiirilised valemid nende suuruste ajalise sõltuvuse kirjeldamiseks protsessi algfaasis 20 sekundi jooksul. Töös on esitatud ka mõned tehnilised kaalutlused, mille arvesse võtmine on vajalik selles töös teostatud katsete kordamisel. Musta lepa jaoks uuriti temperatuuril 50 °C takistusmõõtja ja takistus-tüüpi elektroodide puidu niiskusmõõtjaks kalibreerimise võimalusi. Leiti, et takistus-tüüpi mõõteelektroodide kalibreerimise täpsuse parandamiseks on puidu niiskussisaldustel üle kiu küllastuspunkti võimalik teha mõõteelektroodidele täiendav individuaalne kalibreerimine katsekeha keskmise niiskussisalduse suhtes. Töös saadud tulemused on rakendatavad puidu kuivatuse protsessi monitooringul takistus-tüüpi puidu niiskuse andurite abil niiskussisaldustel üle kiu küllastuspunkti, samuti kasvavate puude elektriliste parameetrite määramisel.

Received March 25, 2014, revised April 25, 2014, accepted May 6, 2014

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VIIS VIIMAST KAITSMIST

ELSA PUTKU

PREDICTION MODELS OF SOIL ORGANIC CARBON AND BULK DENSITY OF ARABLE MINERAL SOILS MINERAALSETE PÕLLUMULDADE ORGAANILISE SÜSINIKU JA LASUVUSTIHEDUSE STATISTILISED PROGNOOSIMUDELID Professor **Alar Astover**, dotsent **Christian Ritz** (Kopenhaageni ülikool) 14. juuni 2016

ALICE AAV

PHENOTYPIC CHARACTERISATION OF POTATO LATE BLIGHT PATHOGEN *PHYTOPHTHORA INFESTANS* IN BALTIC COUNTRIES KARTULI-LEHEMÄDANIKU TEKITAJA *PHYTOPHTHARAINFESTANS* BALTIKUMI POPULATSIOONIDE FENOTÜÜBILINE ISELOOMUSTAMINE Dotsent **Eve Runno-Paurson**, professor **Marika Mänd**

20. juuni 2016

REELIKA RÄTSEP

EFFECT OF ORGANIC CULTIVATION TECHNIQUES ON GRAPEVINE (VTTIS SP.) AND STRAWBERRY (FRAGARIA ×ANANASSA DUCH.) FRUIT QUALITY MAHETEHNOLOOGIATE MÕJU VIINAPUU (VTTIS SP.) JA AEDMAASIKA (FRAGARIA × ANANASSA DUCH.) VILJADE KVALITEEDILE Dotsent **Ele Vool**, professor **Kadri Karp** 20. juuni 2016

KALLE TOOMEMAA

FACTORS INFLUENCING THE OVERWINTERING AND POSSIBILITIES TO REDUCE WINTER MORTALITY OF HONEY BEE (*Apis mellifera* L.) COLONIES MESILASPEREDE TALVITUMIST MÕJUTAVAD TEGURID JA VÕIMALUSED NENDE TALVISE HUKKUMISE VÄHENDAMISEKS Professor **Marika Mänd**

28. juuni 2016

HARDO BECKER

THE EFFECT OF FOREST MANAGEMENT ON THE CARBON AND NITROGEN FLUXES AND STORAGES IN DIFFERENT FOREST ECOSYSTEMS METSAMAJANDAMISE MÕJU SÜSINIKU- JA LÄMMASTIKUVOOGUDELE NING VARUDELE ERINEVATES METSAÖKOSÜSTEEMIDES Professor **Veiko Uri**

25. august 2016

ISSN 2382-7076 ISBN 978-9949-569-38-0 (trükis) ISBN 978-9949-569-39-7 (pdf)

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