7.2 Publikation II: Measurement system for long-term recording of wood moisture content with internal conductively glued electrodes.

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Abstract

Moisture as an important influence factor on fungal growth needs to be considered for service life prediction of wood and wood-based products. Therefore a long-term moisture measuring and data logging method for wood in weathered conditions was developed. The method is based on measuring the electrical resistance with glued electrodes for sustainable connection. The measuring point at the tip of the electrodes was glued conductively into the wood, the remaining outer part of the electrodes was glued with an isolating glue. For this purpose special conductive and isolating glues and electrodes were developed and comparatively evaluated in laboratory tests. As most suitable proved a system consisting of a 2k-epoxy resin, serving for the isolating glue and also as conductive glue (when mixed with graphite powder and ethanol) in combination with a partly isolated stainless steel cable, acting as both, electrode and cable. This system was further tested in combination with mobile mini data logger at 29 different exposure sites in Europe and the United States. After 4-6 years of natural weathering with many extreme climatic and moisture changes no loosening or other detectable abnormality in 541 pairs of electrodes was observed. The data logging systems were working without any problems for 5 years with the first and only battery, and without any additional

maintenance. For the calibration of the measuring system resistance characteristics were determined for different provenances of Scots pine (*Pinus sylvestris* L.), Norway spruce (*Picea abies* Karst.), and Douglas fir (*Pseudotsuga menziesii* Franco).

Keywords: Decay factor, in-service performance, long term adhesion, moisture content, polarization, service life prediction.

7.2.1 Introduction

Service life prediction of wood is required by more and more building codes worldwide [1] and thus methods for the quantification of decay influencing factors are sought. The wood moisture content, the wood temperature, and their dynamics can be expressed as the "material climate" of wood [1], which directly determines the service life of wooden components. Moisture has an essential influence on fungal growth and is for this reason an important factor for the prediction of service life of wooden constructions [1, 2]. Against this background it is advisable to measure and record the electrical resistance in the inner part of wood exposed to weather over a long period of time to determine moisture [3, 4]. The moisture content can be measured by metal electrodes which are incited in the wood. A problem is the inability of metal to produce a sustainable mechanical adhesion to the wood cells. Consequently the electrodes do not maintain electrical contact due to the swelling and shrinking of wood [5, 6].

- 1. A lower resistance is measured because of the entry of water in the capillary interstices along the electrode.
- 2. A higher resistance is measured because of the decreasing contact pressure between electrode and wood resulting in its loosening.

The capillary water uptake can be avoided by gluing the upper part of the electrode in the wood [8]. However, this does not prevent the most important measuring point at the tip of the electrode from declining contact. As a consequence a sustainable and isolating connection of the upper part and a

sustainable and conductive connection of the measuring point to the wood is needed.

The tool to be developed should provide the detection of critical moisture conditions, which allow fungal growth and thus the onset of decay. This paper on long-term moisture monitoring, describes a method based on internal conductively glued electrodes in combination with a data recording system to be applied for field studies as well as for in-situ measurements to monitor objects in service.

7.2.2 Materials and methods

Specimens

The test specimens were made exemplarily from the following wood species: non-durable and permeable Scots pine sapwood (*Pinus sylvestris* L.), nondurable and refractory Norway spruce (*Picea abies* Karst.), and moderately durable and refractory Douglas fir heartwood (*Pseudotsuga menziesii* Franco). The wood species were chosen with respect to their different decay and moisture susceptibility as well as their use in different studies on decay influencing factors (e.g. [9, 10]). In addition, pine sapwood is the commonly used reference material in various European standards (e.g. EN 252 [11]). For calibration experiments wood from three different provenances of each wood species was used (Table 7.2-1).

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Wood species	Provenance 1	Provenance 2	Provenance 3
Norway spruce	North Rhine- Westphalia, Germany	Lower Saxony, Germany	Scandinavia
Scots pine sapwood	Mecklenburg-Western Pomerania, Germany	Scandinavia	Rhineland-Palatinate, Germany
Douglas fir heartwood	Rhineland-Palatinate, Germany	Baden-Wuerttemberg, Germany	Oregon, U.S.

Table 7.2-1. Origin of timber used for specimens in calibration experiments.

For the field tests and the polarization tests specimens of 500x50x25 mm³ were used, for the calibration of the measurement system specimens were

75x50x25 mm³ in dimension. A slight deviation of the grain angle up to 10° was allowed to simulate practice conditions.

Glue formulations

To formulate a suitable conductive glue, experiments with the following agents were made:

- 1. 1k-polyurethan solvent based lacquer (product name: Zweihorn SDF 9) and acetone
- 2. 2k-epoxy resin (Epoxydharz L, Conrad Deutschland, Artikel-Nr.: 236349-62 and Härter L, Conrad Deutschland, Artikel-Nr.: 236357-62) and ethanol

As conductive agents were used:

- 1. copper powder (granulation < 63 μ m)
- 2. graphite powder (granulation < 40 μ m)

The glue/electrode combinations were tested regarding the optimal combination for later field applications. The isolating glue was formulated from the 2k-epoxy resin with wheat flour (type 405) as filler. For microscopic and macroscopic examinations, the isolating glue was stained with Sudan IV, which assured the exclusive coloration of the glue.

Examination of glue properties

The viscosity of the glues was evaluated by extruding the mixture with a 1 mm injection needle, the homogeneity of the glues by their visual appearance. For the examination of conductivity bands of glue with a length of 100 mm and a diameter of 5 mm were dried for 24 hours in a climate chamber at $20 \,^{\circ}\text{C}/65\%$ RH. The electrical resistance of the band was measured with a moisture meter type GANN Hydromette RTU 600.

The different mixtures were dried for two days at 20 °C/65%RH and then evaluated with respect to their physical condition into the categories soft, gummy hard and hard. Abnormalities like cracking and splintering were documented.

Electrodes

The electrodes were made of either polyamide coated stainless steel cables with a core diameter of 1.2 mm or a commercial PVC coated copper cable with a diameter of 1.5 mm. The copper electrodes were used in combination with copper containing glue mixtures, the stainless steel electrodes were used in combination with the graphite containing electrodes.

The electrodes were glued in predrilled holes of 4 mm diameter in different depths depending on the position, where the moisture content should be measured. The distance between the centers of the holes was 30 mm parallel and 6 mm orthogonal to the grain (Figure 7.2-1) in order to minimize the risk of crack formation. 0.1 ml of the conductive glue was injected to the bottom of the holes without contaminating its walls. At the tip of the electrode the first 5 mm of the plastic coating of the electrodes were removed and the electrode was put into the glue. After 24 h hardening, the isolating glue was filled into the holes (Figure 7.2-2) and the electrodes were attached to the data logger.



Figure 7.2-1. Specimen according to EN 252 [11] and position of holes for installation of electrodes (all dimensions in mm).



Figure 7.2-2. Schematic cross cut through a specimen and installed electrodes.

Data logger

The data logger (product name: Materialfox Mini) were developed by Scanntronik Mugrauer GmbH, Zorneding, Germany, in co-operation with the Federal Research Center for Forestry and Forest Products (BFH). The memory capacity was 16 000. The data logger were equipped with three ports. The measuring ranged from $2*10^4 \Omega$ to $5*10^8 \Omega$. A sampling interval of 24 h was chosen for the field installation.

The measuring principle was based on the discharge – time – measurement method. First a capacitor was charged through a very small ohmic resistance and then discharged through the material to be measured. Based on the time needed for discharging, the resistance of the material can be calculated.

For the field work lithium/thionylchloride batteries (type SL-770, Sonnenschein Lithium GmbH, Buedingen, Germany) were chosen, which could be used in a temperature range between -55 to +85 °C. The battery voltage was 3.6 V, the nominal capacity 7.2 Ah. Stored in the shelf at 20 °C they loose less than 1% of their capacity per year. Connected to three logger the batteries should last five years in the field.

Polarisation test

A polarisation test was performed with electrodes installed in Scots pine sapwood and Douglas fir heartwood specimens at a moisture content of 12%. 16 measurements of resistance were performed with the data logger Materialfox Mini in 16 min. Afterwards the electrical poles were changed and the resistance was measured again.

Calibration of the measurement system

To determine the relationship between the electrical resistance and the wood moisture content (MC) gravimetric and electric MC measurements were carried out in comparison at three target MCs (MC = 15, 25 and 50%) and three target temperatures (T = 4, 20, and 36 °C). The target yields were selected with respect to their relevance in practice, where MCs below 15% can not be expected in weathered outdoor exposure and temperatures below the freezing point as well as MCs above 50% lead to inaccurate and implausible resistance values [4, 12]. Specimens were prepared from Norway spruce, Scots pine sapwood, and Douglas fir heartwood, each from three different origins (Table 7.2-1). For each MC/wood species combination eight replicates were used.

The conditioning regime for moistening of the specimens to the target MC is described in Table 7.2-2. The specimens were tightly packed in polyethylene bags as soon as the target MC was achieved. The steel cables were lead through the bag to allow connection to the data logger. The moisture measurements were conducted successively at the three temperatures with two Materialfox Mini data logger, the mean values of both logger were used for plotting the electrical resistance against the gravimetrically measured MC.

Target moisture content	Conditioning procedure
15%	Storing above a saturated solution of sodium chloride at 20 °C for 9 weeks
25%	Storing above a saturated solution of potassium sulfate at 20 $^{\circ}$ for 9 weeks
50%	Water pressure impregnation, storing in polyethylene (PE) foil for six days at 4 °C, afterwards drying down to 50% moisture content at room temperature, subsequent storage in PE foil for another six days at 4 °C

 Table 7.2-2. Conditioning procedure for calibration specimens.

Exposure sites

After screening, the optimal combination of electrodes and glues was tested in the field. In total 541 pairs of conductively glued electrodes in ten European wood species were located at 29 different sites in Europe and in the United States for natural weathering and moisture recording.

7.2.3 Results and discussion – laboratory tests

Screening of the glues

Glue mixtures containing copper showed:

- 1. The copper sedimented in the mixture
- After hardening and weathering a green discoloration in the wood was observed, indicating the formation of copper acetate ions in the wood. Consequently copper should not be used as a conductive medium in the glue.

Glue mixtures containing graphite powder showed:

- 1. Different quality in applicability depending on the proportion graphite powder : resin.
- 2. Different quality in hardness, splintering and cracking after drying.
- 3. Different electrical resistance.
- 4. Glues containing 2k-epoxy resin with a proportion of 1.4 for graphite powder : resin performed best after drying (Table 7.2-5).

Details show Table 7.2-3 and Table 7.2-4.

1k-PUR-solvent based lacquer: Zweihorn SDF-9	Acetone	Graphite powder, granulation < 50 μm	Copper powder, granulation < 63 μm	Viscosity, suitability for extrusion	Electrical resistance a)	Evaluation of the elasticity ^{a)}	Electrical resistance	Evaluation of the elasticity ^{b)}
[g]	[g]	[g]	[g]		[kΩ]		[kΩ]	
50	-	-	50	applicable ^{c)}	1	hard	1	hard ^{d)}
30	15	-	55	applicable ^{c)}	1	hard	1	hard
70	-	30	-	applicable	1	soft	1	hard
33	33	33	-	applicable	1	hard	1	hard

Table 7.2-3. Formulations and screening results of copper containing glues.

^{a)} after 24 h drying in climate chamber at 20 °C/65%RH

^{b)} after 1h at 105℃ and 24 h climatisation in climate chamber at 20℃/65%RH

c) copper sediments

^{d)} green discolouration

			-	-	-			
2k-epoxy resin: Conrad Epoxydharz L	Ethanol	Graphite powder granulation < 50 μm	Ratio graphite : resin	Viscosity, suitability for extrusion	Electrical resistance ^{a)}	Evaluation of the elasticity ^{a)}	Electrical resistance ^{b)}	Evaluation of the elasticity ^{b)}
[g]	[g]	[g]			[kΩ]		[kΩ]	
50	-	50	1.00	not applicable	-		-	
60	-	60	1.00	not applicable	24	hard	24	hard
65	-	35	0.54	not applicable	320	hard	320	hard
70	-	30	0.43	applicable	5000	hard	5000	hard
35	30	35	1.00	applicable	27	hard	18	hard
40	30	30	0.75	applicable	90	hard	600	hard
25	35	40	1.60	not applicable ^{c)}	1	hard	1	hard
25	40	35	1.40	applicable	1	hard	1	hard
30	40	30	1.00	applicable	18	hard	38	hard
18	50	32	1.78	applicable	1	hard	1	hard ^{d)}
18	57	25	1.39	applicable	1	hard	1	hard ^{d)}
9	78	13	1.45	applicable	1.5	hard	1.5	hard

Table 7.2-4. Formulations and screeni	ng results of graphite containing glues.
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a) after 24 h drying in climate chamber at 20 °C/65%RH
 b) after 1h at 105 °C and 24 h climatisation in climate chamber at 20 °C/65%RH

c) no adhesion to the electrodes

d) little cracking in the dried glue

Polarisation test

In the preliminary polarisation tests no polarisation or electrostatic charging occurred. Furthermore a potentially occurring increase in electrical resistance with the measurement duration as described by Welsh [13], seemed to be negligible, because amperages and measuring times applied in this study were too small to have a significant effect.

Properties of the most suitable system after screening

The screening tests revealed the following system, specified in Table 7.2-5, as most suitable: A 2k-epoxy resin serving for the isolating glue and conductive glue (when mixed with graphite powder) in combination with an isolated stainless steel cable, acting as both electrode and cable (Figure 7.2-2 and Figure 7.2-3).



Figure 7.2-3. Cross cut through a glued electrode. The polyamide coated stainless steel cable \oplus is glued in predrilled holes. On the bottom of the hole the uncoated cable is glued with the conductive glue @, the upper part of the coated cable is glued with the isolating glue @. The glue has penetrated into the lumina of the tracheids and produces a very durable connection @.

The screening showed that the ratio between 2k-epoxy binder and graphite powder was crucial for conductivity and elasticity of the glue, whereby ethanol had only a minor effect. The glues described in Table 7.2-5 revealed a strong adhesion to the wood by penetrating about one to two tracheids as visible in Figure 7.2-3, where stained glue was applied.

Glue type	Ingredient	Ratio of ingredients
Conductive glue	Epoxy resin: Epoxidharz L, Conrad Germany, Artikel-Nr.: 236349 - 62	19.2 g / 100 g
	Hardener: Härter L, Conrad Germany, Artikel-Nr.: 236357 - 62	5.8 g / 100 g
	Ethanol	40.0 g / 100 g
	Graphite powder (granulation <50 μm)	35.0 g / 100 g
Isolating glue	Epoxy resin: Epoxidharz L, Conrad Germany, Artikel-Nr.: 236349 - 62	58.8 g / 100 g
	Hardener: Härter L, Conrad Germany, Artikel-Nr.: 236357 - 62	17.7 g / 100 g
	Wheat flour type 405	23.5 g / 100 g

Resistance characteristic curves

Norway spruce, Scots pine sapwood and Douglas fir heartwood revealed differences in electrical resistance at same moisture content (Figure 7.2-4 to Figure 7.2-6) between the different wood species as well as between different temperatures. The dependency of the electrical resistance on the wood species and on temperature was described by many authors [4, 6, 14, 15] and points out the need for wood-species specific characteristic curves.



Figure 7.2-4. Resistance characteristic for spruce (O = provenance 1, $\Box = provenance 2$, $\diamondsuit = provenance 3$, black filled marks = 36 °C, grey filled marks = 20 °C, non-filled marks = 4 °C).



Figure 7.2-5. Resistance characteristic for Scots pine sapwood (\bigcirc = provenance 1, \square = provenance 2, \diamondsuit = provenance 3, black filled marks = 36 °C, grey filled marks = 20 °C, non-filled marks = 4 °C).



Figure 7.2-6. Resistance characteristic for Douglas fir (\bigcirc = provenance 1, \square = provenance 2, \diamondsuit = provenance 3, black filled marks = 36 °C, grey filled marks = 20 °C, non-filled marks = 4 °C).

From Figure 7.2-4 to Figure 7.2-6 can be seen that the course of the resistance characteristics differs not only between the wood species, but also between the three different provenances. Therefore the MC was calculated separately for the different provenances and additionally for all provenances of a species pooled together. A two-step approach was applied in this study: Firstly, the regression

equations in Table 7.2-6, which were obtained by the method of least squares, were considered for the calculation of the moisture content at a temperature of 20 °C ($MC_{20 °C}$). Secondly, the influence of the temperature on the electrical resistance (R) was considered in terms of correcting the MC_T by a temperature compensation factor C_T as the following:

$$MC_T = MC_{20^{\circ}C} + C_T$$

Table 7.2-6. Regression equations of resistance characteristics at 20° C for Norway spruce, Scots pine sap and Douglas fir heart based on the different provenances (1-3) and on all provenances pooled together.

(1)

Species	Provenance	MC _{20°C} [%]
Norway spruce	1	512042.751*(10lgR) ^{-2.090164} - 1453.202*(10lgR) ^{-0.390780} + 224.139
	2	512042.767*(10lgR) ^{-2.081273} – 1465.255*(10lgR) ^{-0.337258} + 293.831
	3	512042.919*(10lgR) ^{-2.029077} – 1583.831*(10lgR) ^{-0.272779} + 424.797
	all	$512042.767^{*}(10 \log R)^{-2.064137} - 1465.248^{*}(10 \log R)^{-0.334548} + 293.847$
Scots pine sap	1	205374.406*(10lgR) ^{-1.974893} – 968.075*(10lgR) ^{-0.494454} + 90.923
	2	205374.569*(10lgR) ^{-1.914759} – 1023.206*(10lgR) ^{-0.402548} + 144.403
	3	205374.625*(10lgR) ^{-1.896288} – 1041.797*(10lgR) ^{-0.327135} + 213.664
	all	205374.625*(10lgR) ^{-1.899652} – 1041.795*(10lgR) ^{-0.327715} + 213.668
Douglas fir	1	1390587.879*(10lgR) ^{-2.434878} – 827.785*(10lgR) ^{-0.335629} + 172.505
	2	1390587.870*(10lgR) ^{-2.456182} – 797.873*(10lgR) ^{-0.493026} + 78.485
	3	1390587.875*(10lgR) ^{-2.433124} – 815.704*(10lgR) ^{-0.377869} + 136.909
	all	1390587.875*(10lgR) ^{-2.432829} – 815.620*(10lgR) ^{-0.369578} + 143.472

A linear dependency between IgR and temperature, as described by Skaar (1964), was assumed and the temperature compensation factor C_T was determined as follows:

$$C_{T} = \frac{\Delta MC}{\Delta T} = \frac{MC_{36^{\circ}C} - MC_{4^{\circ}C}}{36 - 4} [\%/^{\circ}C]$$
(2)

in which ΔT is the temperature difference between 4 °C and 36 °C and ΔMC is the difference in electrically measured MC between 4 °C and 36 °C.

In addition, the influence of temperature on the measurement differs between the wood species and depends on the MC. Therefore the compensation factor C_T needs to be determined separately for each wood species, provenance, and MC. The regression equations in Table 7.2-7 were used for the calculation of the temperature compensation factor C_T .

Table 7.2-7. Regression equations of the temperature compensation factor C_T in relation to the electrical resistance of the wood for Norway spruce, Scots pine sap and Douglas fir heart based on the different provenances (1-3) and on all provenances pooled together.

Species	Provenance	C _T [%/°C]
Norway	1	-0.0000108*(10lgR) ³ + 0.0025533*(10lgR) ² - 0.2118686*(10lgR) + 6,2304876
spruce	2	$-0.0000129^{*}(10 \text{lgR})^{3} + 0.0030618^{*}(10 \text{lgR})^{2} - 0.2564196^{*}(10 \text{lgR}) + 7.5635011$
	3	-0.0000133*(10lgR) ³ + 0.0031598*(10lgR) ² - 0.2674947*(10lgR) + 8.0747234
	all	$-0.0000114^{*}(10 \text{lgR})^{3} + 0.0027042^{*}(10 \text{lgR})^{2} - 0.2278660^{*}(10 \text{lgR}) + 6.8569967$
Scots	1	-0.0000013*(10lgR) ³ + 0.0003590*(10lgR) ² - 0.0420241*(10lgR) + 1.8204137
pine	2	-0.0000015*(10lgR) ³ + 0.0004120*(10lgR) ² - 0.0501462*(10lgR) + 2.2482478
sap	3	-0.0000011*(10lgR) ³ + 0.0003297*(10lgR) ² – 0.0437547*(10lgR) + 2.0633359
	all	$-0.0000019^{*}(10 \text{lgR})^{3} + 0.0005006^{*}(10 \text{lgR})^{2} - 0.0569046^{*}(10 \text{lgR}) + 2.3903511$
Douglas	1	-0.0000018*(10lgR) ³ + 0.0004961*(10lgR) ² - 0.0565622*(10lgR) + 2.3822573
fir	2	0.0000032*(10lgR) ³ -0.0006707*(10lgR) ² +0.0333667*(10lgR)+0.1099423
	3	-0.0000059*(10lgR) ³ + 0.0014579*(10lgR) ² – 0.1315450*(10lgR) + 4.3150158
	all	$-0.0000031^{*}(10 \text{lgR})^{3} + 0.0007999^{*}(10 \text{lgR})^{2} - 0.0791982^{*}(10 \text{lgR}) + 2.9270513$

The calculated MC is plotted against the gravimetrically determined MC in Figure 7.2-7 and Figure 7.2-8. The correspondence between calculated and determined values was much better when different provenances were considered separately. Although a linear regression could be assumed, the confidence interval of the single values was not constant. In contrast, with higher MC the maximum error linearly increased. Therefore, the upper and lower limitation curves were determined graphically indicating the maximum error at a confidence of 99%.



Gravimetrically measured MC [%]

Figure 7.2-7. Calculated MC compared with gravimetrically measured MC. Underlying regressions were determined for all provenances pooled together (\triangle = Scots pine sap, \Box = Douglas fir, \diamondsuit = Norway spruce).

Gravimetrically measured MC [%]



Figure 7.2-8. Calculated MC compared with gravimetrically measured MC. Underlying regressions were determined separately for the different provenances (\triangle = Scots pine sap, \Box = Douglas fir, \diamondsuit = Norway spruce).

Variations in electrical resistance between different specimens of the same wood species and provenance maybe obtained due to differences in extractive content, density and anatomical structure [6, 15]. However, the overall result of the calibration experiments was a sufficiently high accuracy of the measurement system. The main objective – to detect critical moisture condition for fungal activity – proved to be achievable.

7.2.4 Results and discussion – field tests

The system was tested at 29 different exposure sites in Europe and the United States for 4-6 years of natural weathering with extreme changes in moisture content and temperature of the wood from −21 to +55 °C. No loosening or detectable abnormality in 541 pairs of electrodes was observed, and even after severe decay the connection between wood and conductive glue was still stable (Figure 7.2-9). All daily recorded values of resistance were plausible and showed little differences between replicates. The data logger operated with lithium/thionylchloride batteries worked for 5 years without any error.



Figure 7.2-9. Glued electrodes in Scots pine specimens. Left: After six years in the field. Right: Cross cut through a glued electrode in wood decayed by severe white rot after four years of exposure.

7.2.5 Conclusions

The overall objective of this study was to find a suitable long-term moisture measuring and data recording system to be used in weathered conditions. Therefore different electrodes, conductive glues, and isolating glues in combination with mini data logger were investigated. Initially the use of standard copper cables as electrodes in combination with a copper based glue was examined. However, the sedimentation of the copper as well as the green discoloration indicating the production of copper ions through the reaction of acids in the wood and the copper discarded the copper based system for further applications. Consequently, graphite based glue and stainless steel cables were applied for field tests instead of copper containing glue and copper electrodes.

Therefore the best performing mixture of the graphite glue was sought. Some mixtures of 1k solvent lacquer and 2k-epoxy resin based glues performed well. To get the same glue components for the isolating and the conductive glue a 2k-epoxy resin was chosen. With this combination a good resistance against the degradation of blue stain fungi and mould was achieved and problems occurring in long term use between different glue types were minimized.

The resistance characteristics for three different wood species revealed a highly satisfactory accuracy of the measurement system. The need for wood-species specific resistance characteristics and temperature compensation became obvious, to consider also different provenances of the timber is recommended, if possible.

The system was working steadily with no loosening of the electrodes and no obvious failure in measuring or data logging. Different applications of the system for moisture monitoring in field test studies and on buildings in service will be presented in a follow-up publication.

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