

Measurement of moisture content profiles in coated and uncoated Scots Pine using Magnetic Resonance Imaging

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ABSTRACT

Non-invasive spectroscopic method of accessing information about moisture profile in wood is presented. The technique used is proton magnetic resonance imaging (¹H MRI) with a low-field Benchtop Imager Magnet and an effective Single-Point-Imaging sequence (SPI sequence) and with a high-resolution orthogonal permanent magnets system and multi-echo acquisition. Different examples of application of this method in wood science are presented. One example is the interaction between indoor air fluctuations and Scots pine. SPI images of bound-water distribution in Scots pine with a spatial resolution on a sub-millimeter scale were acquired when one of the orthotropic directions of the wood material was exposed to typical indoor day-to-day moisture fluctuations. Another example is cyclic sorption experiments with coated Scots pine exposed to day-to-day relative humidity step changes and water absorption and evaporation through the wood coating. Even other examples with higher resolution moisture profiles describing water interaction with wood, coatings and adhesives are presented.

INTRODUCTION

In a biogenetic and heterogeneous material like wood the action of water and moisture plays an important role in the performance of the material as such but even more where wood is in combination with other materials. These material combinations exhibits interfaces that sometimes are polar and high energy interfaces causing accumulation of water. The spatial distribution of water in wood has been studied by ¹H MRI (proton Magnetic Resonance Imaging), which is a widely used versatile non-invasive tool. The spatially resolved MRI of the moisture content (MC) in wood during drying using Hahn spin echo acquisition has been demonstrated by several authors (Araujo et al., 1992).

The present paper presents research where the action of water and moisture are studied. The research was part of doctoral thesis of Stéphane Hameury (2006) at KTH Stockholm and also part of an EU funded project MARWINGCA (*M*agnetic *R*esonance *I*maging of *W*ood at the *I*Nterface with *G*lues, *C*oatings and *A*ir) (Berglind et.al 2006).

Moisture buffering capacity

Wood is a porous and hygroscopic material constantly undergoing transient changes in its moisture content while trying to reach a state of hygrothermal equilibrium with its surrounding environment. The moisture gradients developed within wood in response to climate fluctuations are accompanied by e.g. shrinkage and swelling, moisture-induced stresses and mechano-sorptive effects, which may ultimately lead to cracking or loss of load-bearing capacity. This affinity of wood to liquid water and water vapour is usually perceived as a drawback by the building community, but it can also be an advantage regarding the moisture buffering capacity of the building materials facing the indoor climate. This capacity may be used as a passive way to regulate the relative humidity fluctuations together with an efficient ventilation system (Osanyintola & Simonson, 2006). An increase in the amount of wood facing the indoor environment will help to regulate the indoor relative humidity.

Measuring of moisture buffering capacity requires accurate method which follows moisture changes of the exposed surface in the very shallow depth. With resistive sensor it is not possible. Another way to study the moisture buffering effect of wood might be to use a Computed Tomography scanner (CT scanner) (Wiberg, 2001, Rosenkilde and Arfvidsson, 1997, Lindgren, 1992;) which show capacity of

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measuring moisture profiles in wood with a resolution of ca. 240 μm . In the present paper the moisture profiles of uncoated and coated wood in response to dynamic moisture cycles obtained with MRI are presented (Hameury and Sterley 2006, Hameury 2006).

Measurement of wood surface layer drying (MARVINGCA project)

Wood drying is an important industrial process in the sawmilling industry since it has a great impact on both the product quality and the production economy manufacturing costs. Hence, several scientists have aimed to improve the industrial drying process by developing theoretical models with the aim to analyse the drying process. These models need relevant experimental measurements in order to verifying their results. A weak point in some of those models has been the description of the moisture transport above the fibre saturation point, FSP, and the behaviour at the surface interface. Therefore, the moisture transport behaviour has been studied near the surface interface between wood and air based on high resolution measurements during drying using MRI (Rosenkilde et al 2004).

Water accumulation at the coating/wood interface (MARVINGCA project)

The use of surfactants is essential for controlling the colloidal stability of the dispersion during synthesis, storage, application and film formation of surface coatings on wood, especially water-borne coatings. During film formation, phase separation occurs and these surfactants in the water phase may be mobilised and transported into the wood substrate. The surfactants can also be released by rain after film formation and, either washed away, or transported into the wood substrate. The chemical nature of surfactants, with a hydrophilic and a hydrophobic part in the molecule, causes them to accumulate at interfaces, where high concentrations may occur. In order to study the moisture dynamics at the wood/coating interface and especially the influence of surface-active substances, which under some circumstances accumulate at interfaces measurements has been performed with the MRI technique.

Water evacuation from a curing glue line (MARVINGCA project)

Wood gluing can be split into three phases: glue spreading, assembly and pressing. Knowing the rate of water evacuation from the glue line through evaporation to the air and absorption into the wood during the different process steps with high accuracy is essential for modelling of the curing behaviour. In order to quantify the velocity of these individual processes, evaporation and diffusion, high spatially resolved MRI has been used to follow glue line curing for various substrates.

MATERIALS AND METHODS

Moisture buffering capacity

Images were obtained from the samples of Scots pine (*Pinus sylvestris* L.) shaped as small cylinders with a diameter of 12 mm and a length of 18 mm to 22 mm and placed in *NMR* glass tubes. Details are presented in Hameury and Sterley (2006) and Hameury (2006). The samples sealed with a wax varnish on all sides except on the surface to be exposed to the climate fluctuations and were exposed to periodical step changes in relative humidity typical of the daily indoor. The samples were placed for 8 hours a relative humidity of 79.5 % and thereafter the samples were then exposed for 16 hours at 24 % RH and 22°C. The cyclic procedure was repeated for 4 days to reach an acceptable quasi-steady-state and moisture profiles were then recorded every hour during the 5th day.

The measurements were made using a low-field MARAN Benchtop Imager Magnet operating at 24.5 MHz from Oxford Instruments Ltd. The 1-dimensional gradient set provided a maximum gradient strength in the magnetic field of approximately 3 T.m⁻¹. The *RF* probe used was an 18 mm diameter solenoid probe driven by a 300 W RF output amplifier. The measurement sequence was the Single-Point-Imaging (SPI) also known as Constant-Time Imaging (CTI). The SPI sequence was chosen because of its efficiency to measure bound water in wood under fibre saturation point. The spacial resolution was 127 μm .

MARVINGCA project

The MRI measurements were performed at University of Surrey, School of Physics, Guildford, U. K. with a high-resolution orthogonal permanent magnets system. The measurement sequences were multi-echo acquisition similar in form that of Carr and Purcell. Profiles of the samples were measured using the thin film coil using the standard pulse sequence $(\alpha_x-\tau(\alpha_y-\tau\text{-echo}-\tau)_n)$. The magnetic field strength on the sample was 0,7 T with a magnetic gradient strength of 17 Tm^{-1} . The spatial resolution for the measurements is in the order of 15-20 μm .

RESULTS AND DISCUSSION

Moisture buffering capacity

Figure 1 and 2 present the one-dimensional MRI profiles of bound water in Scots pine measured in the two orthotropic directions along the moisture flow, i.e. the tangential and radial. These profiles are presented as moisture concentration profiles. The profiles shown in Figure 1 for the tangential direction indicate that only a narrow region interacts with the surrounding environment. The periodic penetration depth is approximately 2 mm and after 9 mm no moisture changes occur with time. We call this depth the Active Buffering Depth (ABD) to differentiate it from the penetration depth. In Figure 2, where the axis of the sample is along the radial direction, the profiles match the annual growth rings shown in the superimposed digital picture. In this case, it is not possible to define a periodic penetration depth as was possible in the tangential direction because of the presence of fluctuations induced by the annual growth rings. However, the ABD is still measurable and is approximately 9–10 mm. Secondly, the exposed surface of the samples does not reach equilibrium immediately. This feature may be explained by a considerable moisture surface resistance but it is certainly not the only cause and we suspect that a retarded sorption process occurs in the wood. The MRI technology and the sequence used in this paper could be of help to further analyse this phenomenon. Moreover the Moisture Buffer Value calculated from the moisture profiles was almost exactly the same as that determined by the gravimetric method (Hameury and Sterley 2006).

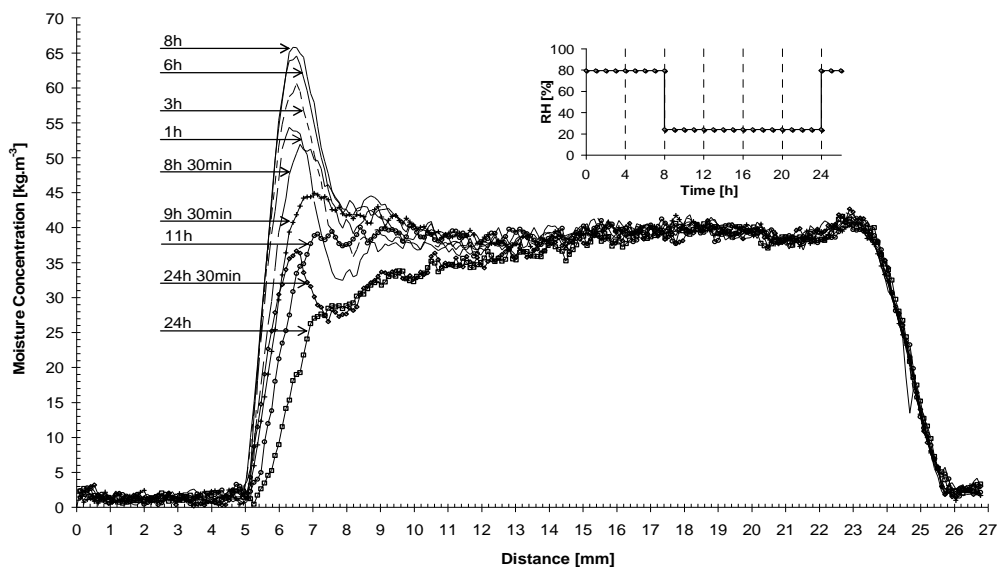


Figure 1. 1D SPI magnitude profiles in the tangential direction of Scots pine at different times. The surface exposed to cyclic humidity changes is to the left. The resolution is ca. 127 μm .

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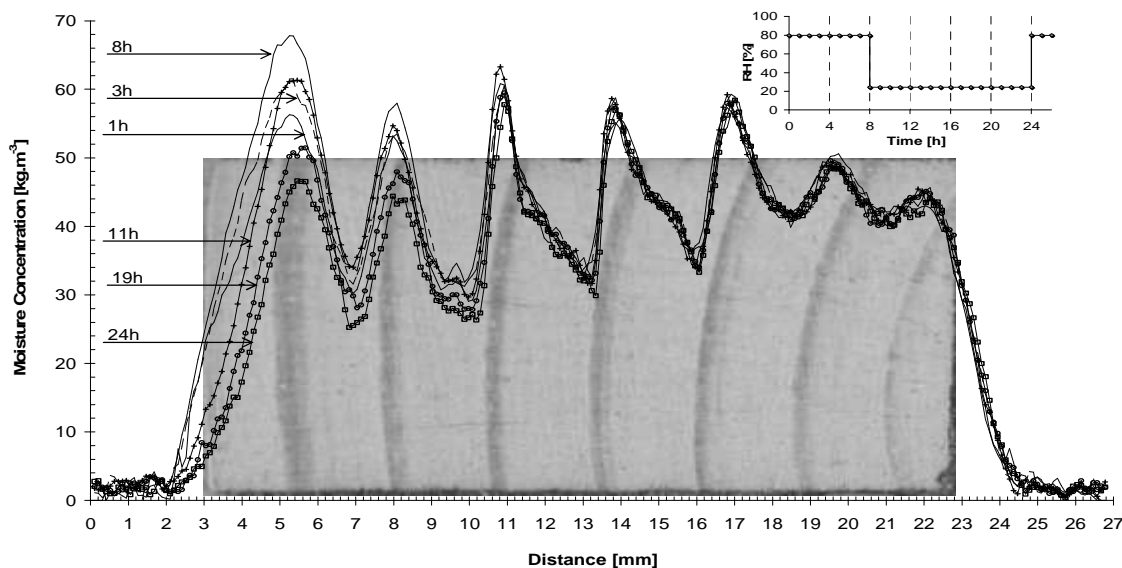


Figure 2. 1D SPI magnitude profiles in the radial direction of Scots pine at different times. The surface exposed to cyclic humidity changes is to the left. The resolution is ca. 127 μm . The profiles are superimposed on a digital picture along the axis of the cylinder of the wood sample.

Wood drying

Measurements were made from fresh condition and down to end use moisture content with Scots pine. The measurements in the surface layer showed the very early development of a dry zone close to the surface interface, Figure 3. In that zone or shell the moisture content was below the fibre saturation point, FSP, even though the bulk moisture content was far above the FSP.

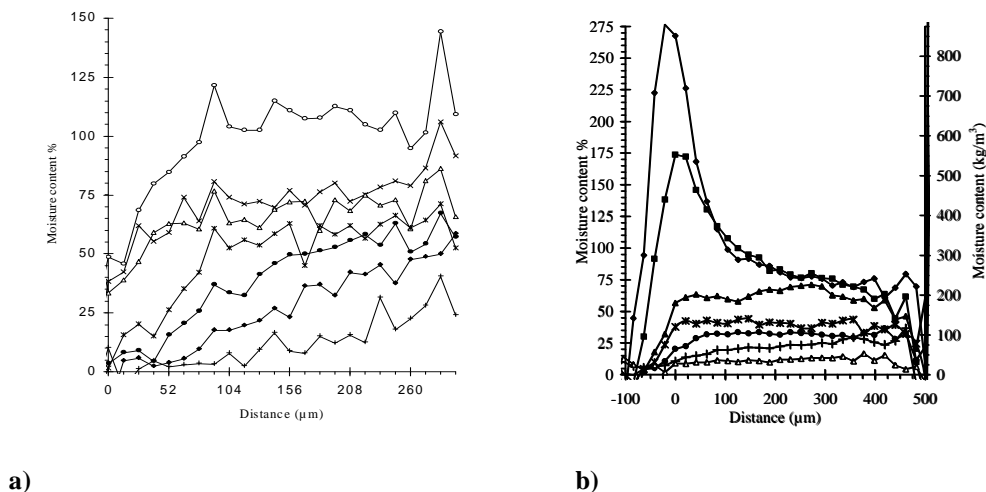


Figure 3. Moisture content profiles for Scots pine during drying:

- a) —○— 0.5 h, —x— 1 h, —△— 2 h, —*— 4 h, —●— 7 h, —◆— 19 h, —+— 24 h,
 b) The actual surface is at 0 μm on the distance axis, —◆— 0 min, —■— 5 min, —▲— 9 min, —*— 16 min, —●— 41 min, —+— 56 min, —△— 1254 min.

Figure 3 b) show that after 9 minutes of drying the moisture profile is almost flat. The moisture profiles remain approximately flat down to a moisture content slightly above 30% at 41 minutes. At this point, a gradient starts to develop at the surface. The profiles recorded after 41 minutes all show a gradient from the surface interface towards the bulk. This behaviour showing almost flat profiles above a moisture content of approximately 30% and a gradient developing from the surface at lower moisture contents has been reported before, Tremblay *et al.* (2000); Wiberg (2001); Rosenkilde and Glover (2002) and Salin (2002). Wiberg (2001) reported about what he called the “dry shell formation” which means a surface layer where the moisture content is below the fibre saturation point,

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of approximately 30%. This indicates that present measurements of moisture content are obtained within that dry shell. Furthermore it is possible to see the very early development of that shell with a much higher spatial resolution than reported in Tremblay *et al.* (2000) and Wiberg (2001).

Wood coating

Figure 4 shows the spatial water distribution in at the coating/wood interface. The water proton density profiles (green start profile before water ingress and red profile after 120 hours) has been superimposed on an ESEM image of coating (thickness approx. 350 μm) applied to wood. The water proton density is proportional to the moisture content. It is clearly seen that accumulation of moisture occurs at the interface between coating and wood, at “distance 750”. This behaviour presented with MRI technique can be of grate importance for durability of this coating and wood.

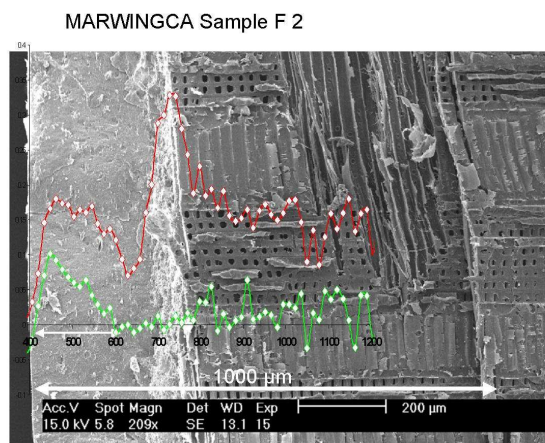


Figure 4. Proton density profiles through an acrylic coating on wood during water ingress

Curing of the glue line

In figure 5 a, glue has been placed on a glass cover slip without any upper substrate. The glue signal is reduced due to chemical hardening and drying through evaporation. The evaporation velocity, defined as the change of glue line thickness with time, was proportional towards time with 1.6 $\mu\text{m}/\text{minute}$ during the first 70 minutes. In figure 5 b, glue has been placed between a wood substrate and a glass cover slip. The diffusion velocity into the wood, defined as the change of test piece thickness with time, was proportional towards time with 0.6 $\mu\text{m}/\text{minute}$ during the first 85 minutes.

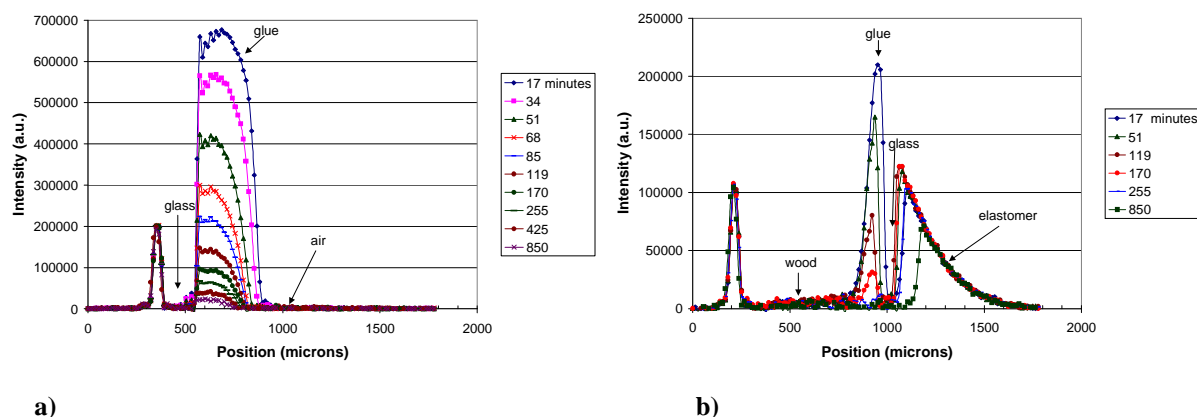


Figure 5 a) Glue curing profiles for glue spread on a glass substrate. The signal to the left is a marker tape. b) Glue curing profiles for glue spread between a wood and a glass substrate. The elastomer facilitates a precise determination of the thickness of the glue line.

Combining these separate measurements yields the glue-spreading situation, where the water evacuation should be around 2.2 $\mu\text{m}/\text{minute}$. The second measurement indicates that during the assembly, the water evacuation into each wood substrate would be 0.6 $\mu\text{m}/\text{minute}$.

CONCLUSIONS

The MRI method presented in this article based on the ^1H MRI technique and a standard SPI sequence is suitable to probe moisture profiles in wood materials at a low moisture content with high resolution. It was confirmed that the moisture exchange between the indoor environment and wooden material is confined to a few millimetres below the wood surface during typical daily moisture fluctuations and a Moisture Buffer Value can be calculated from the NMR profiles with good accuracy. The advantage of the method is however the spatial information obtained, something that the gravimetric method cannot provide.

Moisture content profiles with high spatial pixel resolution, 21 μm , have been measured in Scots pine heartwood while drying from wet conditions to near equilibrium in the surface layer, (0 – 300 μm).

It was shown that when the mean moisture content in the surface layer decreases to the fibre saturation level, gradients starts to build up from the surface towards the bulk of the sample, although the bulk moisture content is far above the fibre saturation. Furthermore, the results presented in this study imply that there is a dry shell forming in the surface layer shortly after drying has begun.

The MRI technique applied to wood coating and wood adhesive research show possibility to analyze water transport phenomena through the interface which can explain performance of different products and can be useful for development of new products.

REFERENCES

1. Araujo, C. D., MacKay, A. L., Hailey, J. R. T., Whithall, K. P., and H. Le. (1992). Proton magnetic resonance techniques for characterization of water in wood: Application to white spruce. *Wood Science and Technology*, 26(2), 1001–113.
2. Berglind H., Ekstedt J., Rosenkilde A., Salin J-G., McDonald P., Bennett G., Keddie J.L., Brands G. and P. Jokinen (2003) Magnetic Resonance Imaging of wood at its interface with glue coating and air. Final report to EC MARWINGCA. Träteknik P0305014. ISSN 1102-1071.
3. Hameury, S. (2006). The hygrothermal inertia of Massive Timber Constructions. Doctoral Thesis. KTH Architecture and the Built Environment, Stockholm Sweden. ISSN 0349-5752.
4. Hameury, S. and M. Sterley. (2006): Magnetic resonance imaging of moisture distribution in *Pinus sylvestris* L. exposed to daily indoor relative humidity fluctuations. *Wood Material Science and Engineering*, 2006,1, pp:116-126.
5. Lindgren, O. (1992). Medical CT-scanners for non-destructive wood density and moisture content measurement. Doctoral Dissertation, Tech. Rep. No. 1992:111, Luleå University of Technology.
6. Osanyintola, O. F. and C. J. Simonson. (2006). Moisture buffering capacity of hygroscopic building materials: Experimental facilities and energy impact. *Energy and Buildings* (In Press).
7. Rosenkilde, A., Gorce J-P. and A. Barry. (2004) Measurement of moisture content profiles during drying of Scots pine using magnetic resonance imaging. *Holzforschung*, 58, 138-142.
8. Rosenkilde, A., and P. Glover. (2002). High resolution measurement of the surface layer moisture content during drying of wood using a novel magnetic resonance imaging technique. *Holzforschung*, 56, 312–317.
9. Rosenkilde, A. and J. Arfvidsson. 1997. Measurement and evaluation of moisture transport coefficients during drying of wood. *Holzforschung* 51, 372-380.
10. Salin, J-G. 2002. Theoretical analysis of mass transfer from wooden surfaces. 13th International Drying Symposium, Aug. 27-30, Beijing, China
11. Tremblay, C., A. Cloutier and Y. Fortin. 2000. Experimental determination of the convective heat and mass transfer coefficients for wood drying. *Wood Sci. Technol.* 34:253-276.
12. Wiberg, P. (2001). X-ray CT-scanning of wood during drying. Doctoral Dissertation, Tech. Rep. No. 2001:10, Luleå University of Technology.