



**COST Action E55**  
**Modelling the Performance of Timber Structures**  
**Final Conference, ETH Zurich, Switzerland - May 26-27, 2011**

# **NMR imaging study and multi-Fickian numerical simulation of moisture transfer in Norway spruce samples**

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## International collaboration

- Project: **Improved Moisture** (Improved glued wood composites – modeling and mitigation of moisture induced stresses), 2007-2010.

Coordination: VTT. Partners: VTT-Finland, KTH-Sweden, TUW-Austria, MPA-Germany, University of Harbin-China.



- International network: **COST action E55** (Modelling the performance of Timber Structures), 2006-2010.

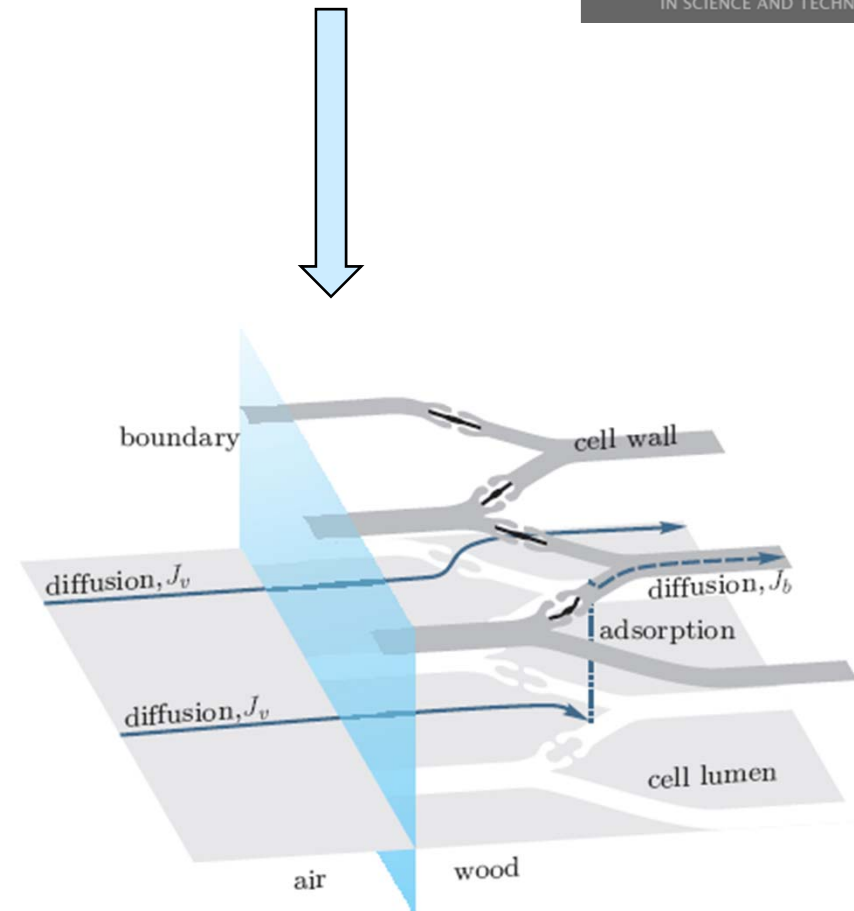
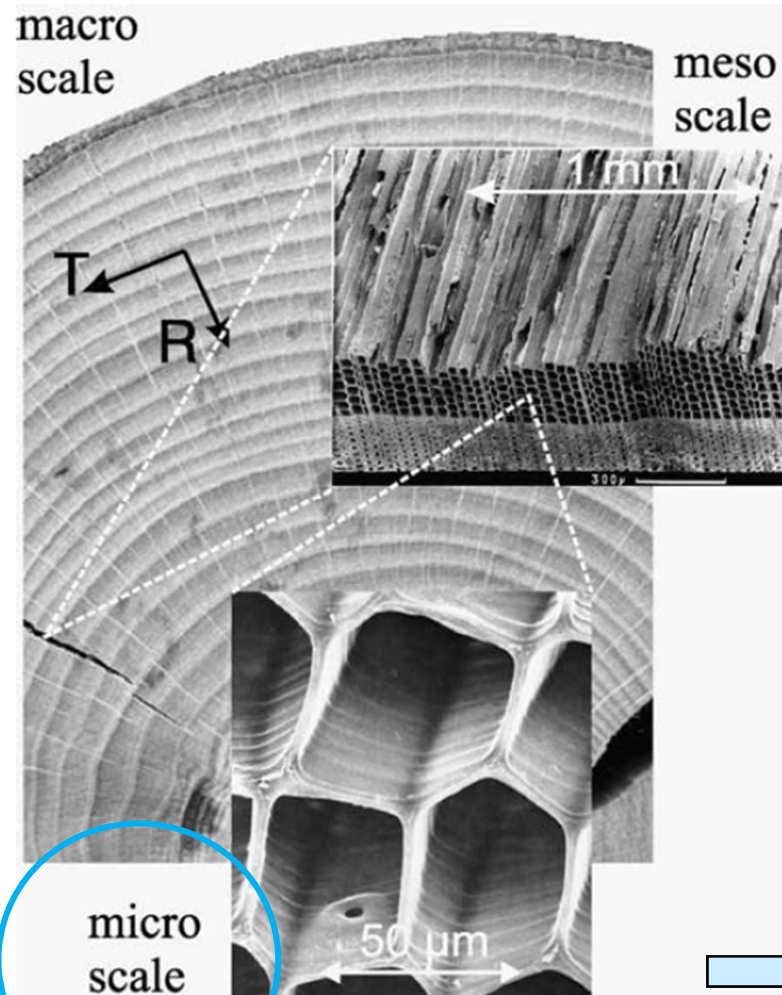


- Collaboration with University of Calabria, Italy.



# Multiscale

# Multiphysics



## Aims of the work

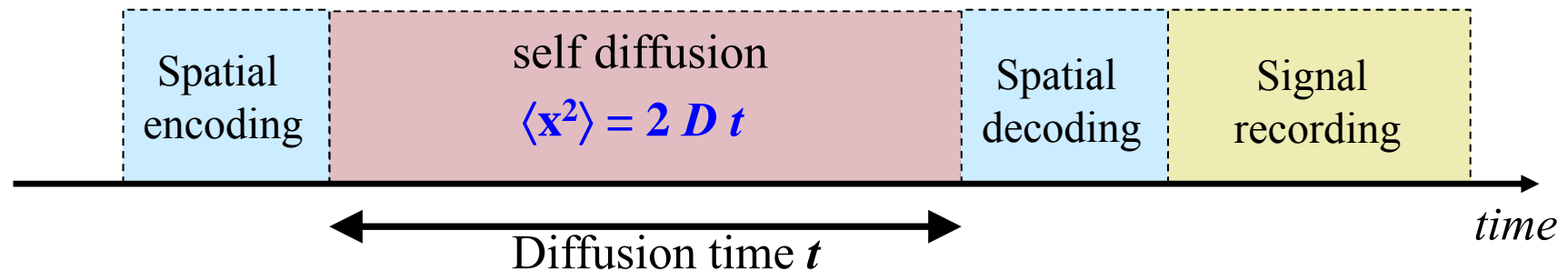
- To measure the moisture distribution in Norway spruce samples under relative humidity variations by using an advanced technique: **the nuclear magnetic resonance (NMR) imaging**.
- To experimentally test both uncoated and uncoated specimens.
- To numerically simulate the uncoated experiments by using a method able to realistically describe the complex phenomenon of moisture transfer in wood: **the multi-Fickian method**.

**Motivation:** The accurate measurement/prediction of moisture content and moisture gradients is fundamental for the accurate evaluation of Moisture Induced Stresses (MIS) in timber structures.



## Measurement of self-diffusion by NMR

- Spatial encoding and decoding are performed by using pulsed magnetic field gradients.
- Spatially label the nuclear spins using a gradient of the magnetic field (spatial encoding).
- The average displacement  $\langle x^2 \rangle^{1/2}$  of nuclei (**protons in the water**) is monitored by measuring their spatial positions at a later time (spatial decoding).



Monitoring of particle position is based on the linear relationship between the NMR frequency  $\omega$  and the applied external magnetic field  $B$

$$\omega = gB \quad (\text{Larmor equation})$$

The amplitude of the NMR signal is dependent on the applied magnetic field gradient according to exponential function (dependent on the diffusion coefficient  $D$ ).



## Considerations on NMR technique

- **Spatial resolution of the order of tens of micrometers can be achieved in one-dimensional image.**
- **NMR experiments have typically been performed in green wood** (high moisture content, results in favorable NMR parameters, such as long spin relaxation times).
- **Fewer studies have investigated wood dried to equilibrium with ambient air humidity.** This is part of the present study.



## NMR experiments - Materials

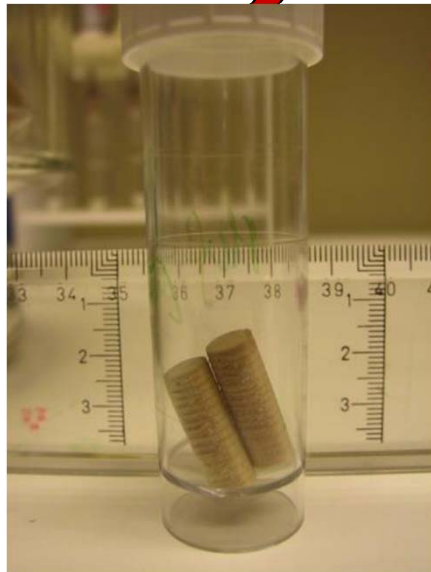
- A wood board of Norway spruce (*Picea abies*) grown in northern Sweden was equilibrated in a climate chamber to 65% relative humidity (RH) and 23°C from an original green state. Average density: 420 kg/m<sup>3</sup>.
- **Cylindrical pieces of ca 8 mm in diameter and 20 mm long** were drilled from the same wood board. Sample axes were parallel to the main tree axes, longitudinal (L), radial across the annual rings (R) and approximately tangential to the annual rings (T).
- **For the measurements the pieces were kept over different saturated salt solutions** of MgCl<sub>2</sub>, NaNO<sub>2</sub>, KNO<sub>3</sub> which provided relative humidities of 33%, 65% and 94 % respectively.

## Experimental setup - NMR

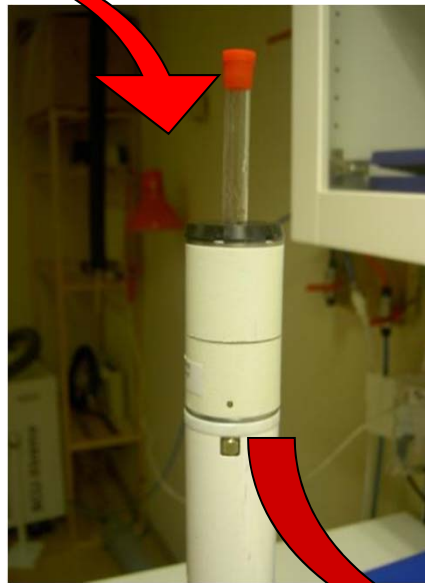
**Bruker Avance II spectrometer at resonance frequency of 300 MHz (superconducting magnet and consol with electronic devices).**

**The images covered a field of view of 20 mm with a spatial resolution of 78  $\mu\text{m}$ .**

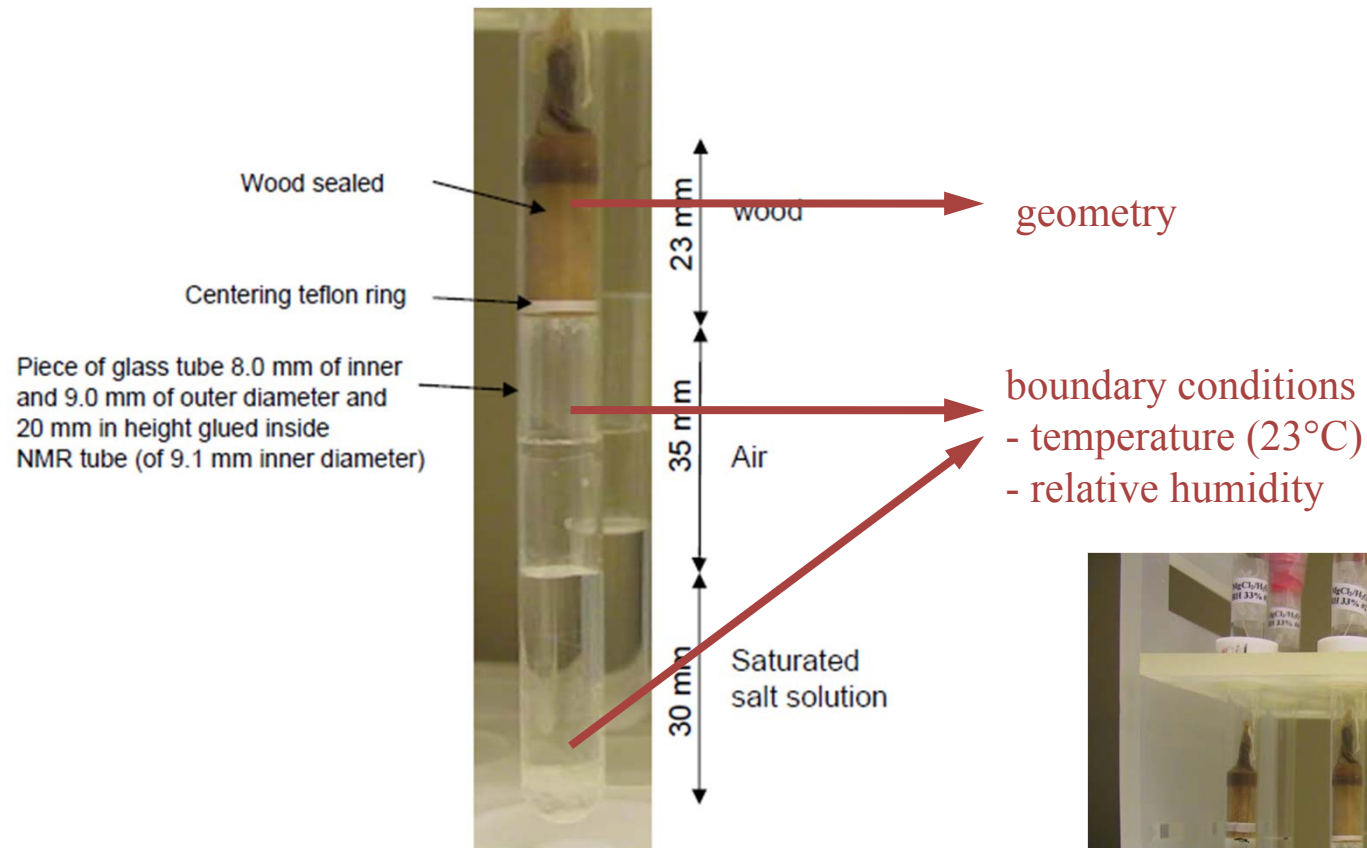
**cylindrical samples**



**mini dessiccators**



## Experimental setup – mini desiccators

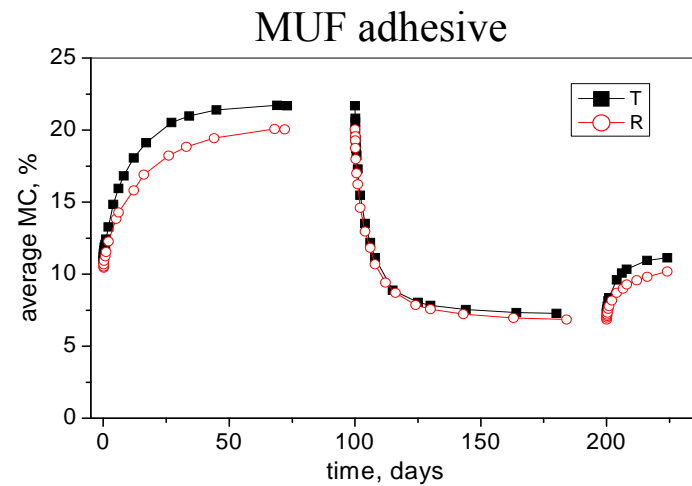
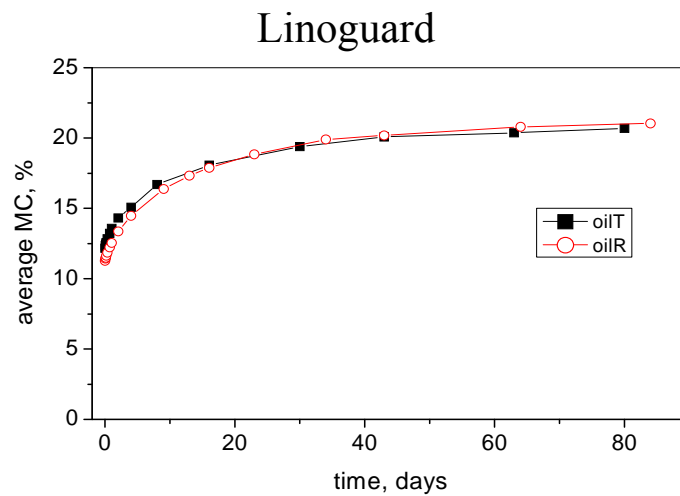
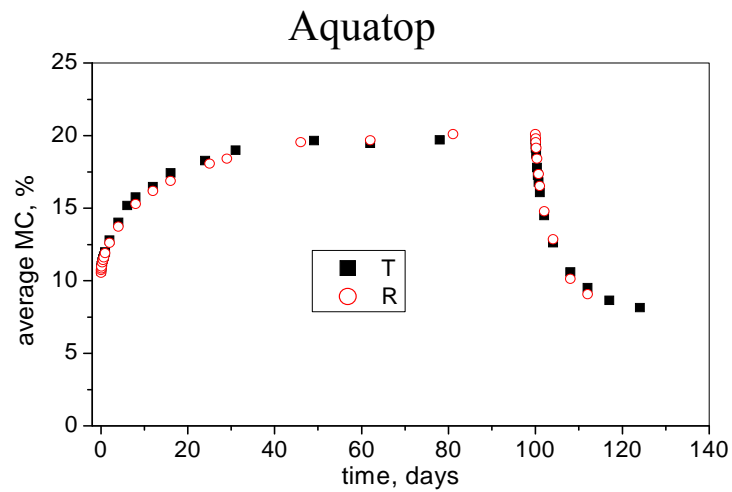
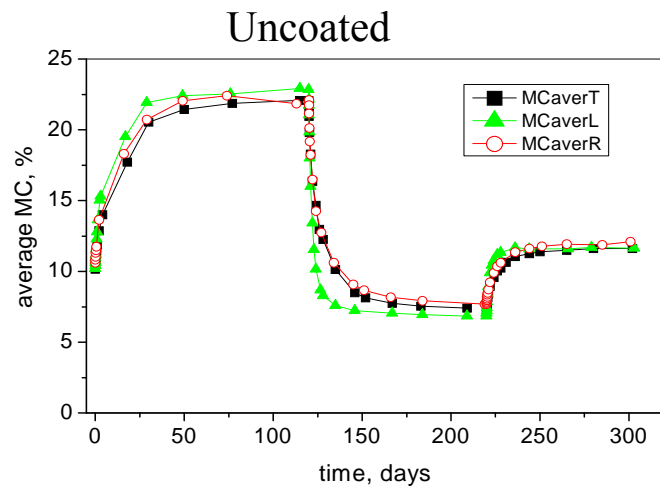




## Tests

- For three uncoated wood samples in L, T, and R orientations, the moisture profiles were obtained for three consecutive changes of RH:
  - (1) from 65% to 94 %;
  - (2) from 94% to 33 %;
  - (3) from 33% to 65 %.
- **Tested coatings:**
  - Aquatop** (a waterborne acrylic lacquer);
  - Linoguard** (linseed oil based);
  - Melamine-Urea-Formaledhyde (MUF)**, adhesive bond.
- For each step, the sample reached equilibrium moisture distribution during a 2-3 months.
- The obtained linear relationship between NMR signal intensity and gravimetric moisture content provided the needed calibration constants to convert NMR intensities into MC.

## Average MC during NMR measurements (over the axis of the specimen: 0-14 mm)



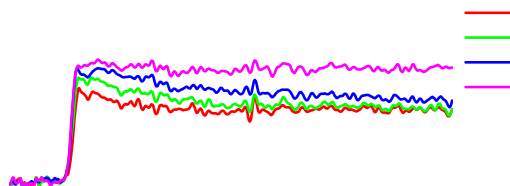
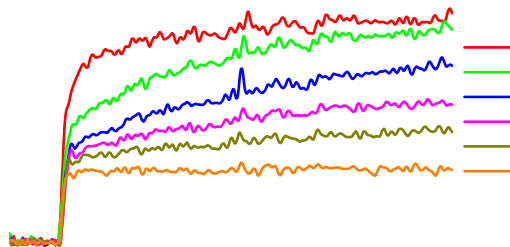
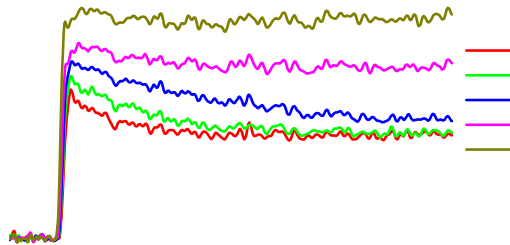
Results for three consecutive changes of relative humidity are shown:  
(a) from 65 to 94 %; (b) from 94 to 33 %; (c) from 33 to 65 %.

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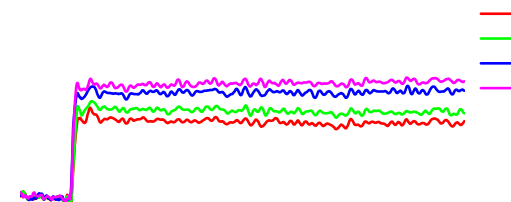
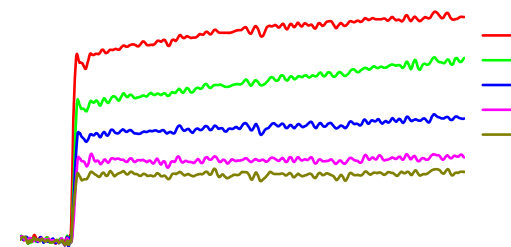
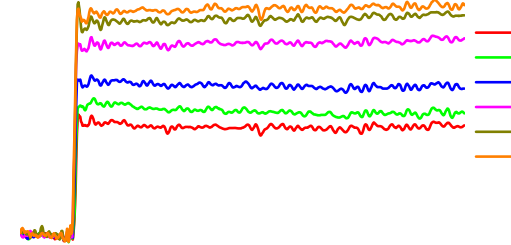
12



Tangential case



Longitudinal case



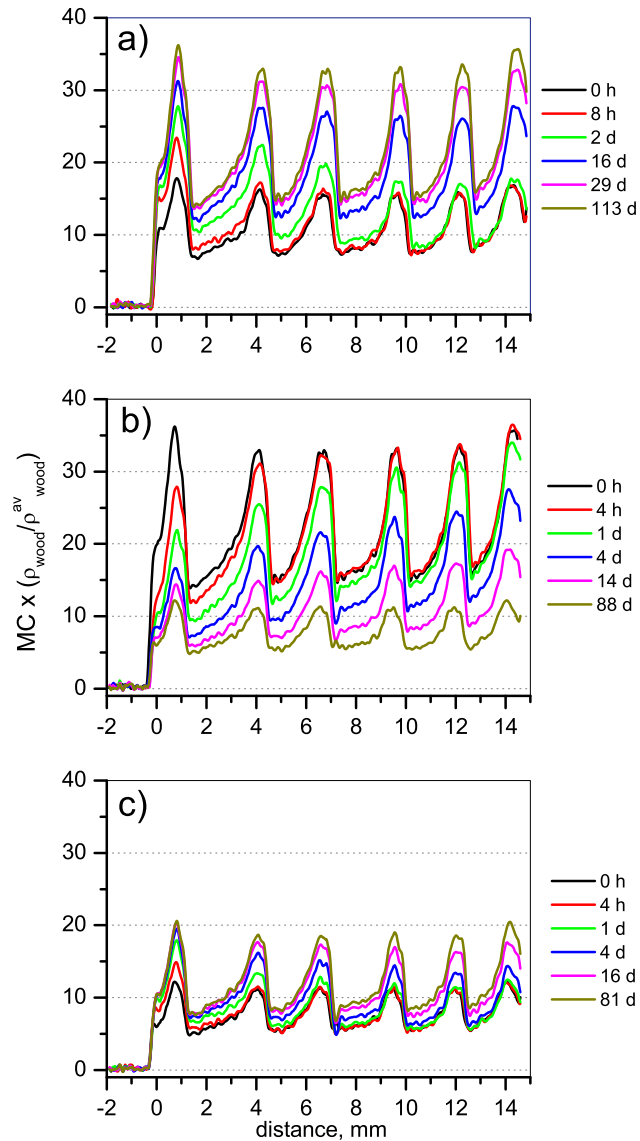
**Results for three consecutive changes of relative humidity are shown:  
(a) from 65 to 94 %; (b) from 94 to 33 %; (c) from 33 to 65 %.**

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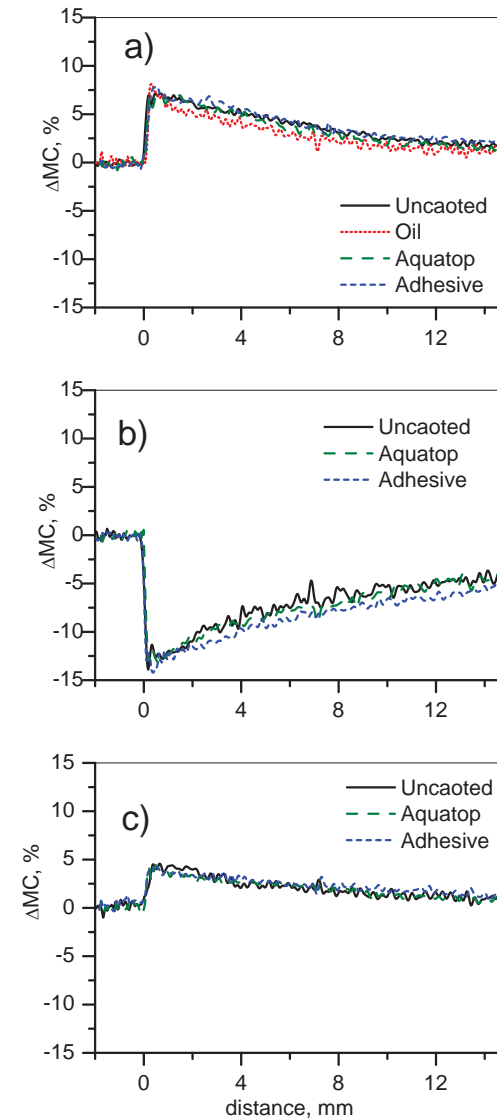
13



**Radial case**



**Coating or adhesive**



## Multi-Fickian model of moisture transfer in wood

- Multi-Fickian (or **multi-phase**) model (Frandsen et al., 2007)

- a) **water vapor diffusion in the cell lumens**

$c_v$  = water vapor concentration

- b) **sorption of bound-water** (coupling)

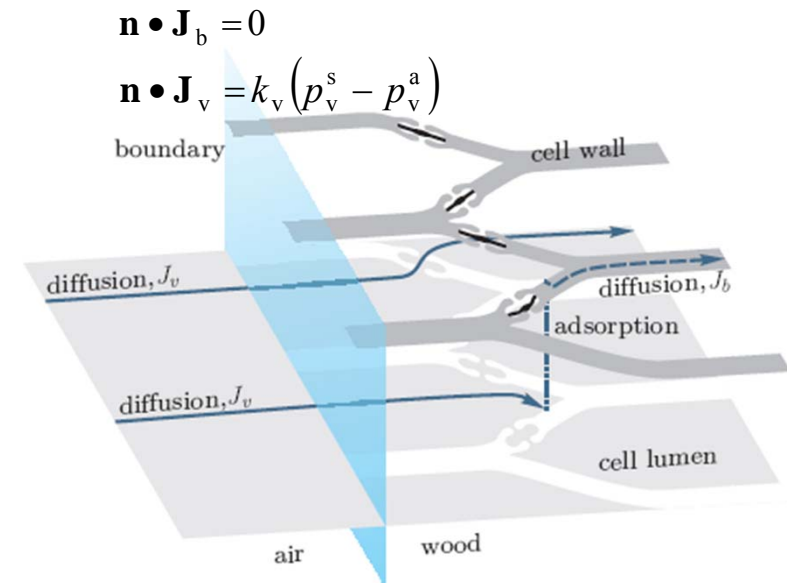
- c) **bound-water diffusion in the cell walls**

$c_b$  = bound water concentration

$$\frac{\partial c_v}{\partial t} = \nabla \cdot (\mathbf{D}_v \cdot \nabla c_v) - \dot{c}$$

Sorption rate

$$\frac{\partial c_b}{\partial t} = \nabla \cdot (\mathbf{D}_b \cdot \nabla c_b) + \dot{c}$$



Frandsen H.L., Damkilde L. and Svensson S. A revised multi-Fickian moisture transport model to describe non-Fickian effects in wood. *Holzforschung* 2007; 61(5), 563-572.

## Sorption rate and sorption isotherm

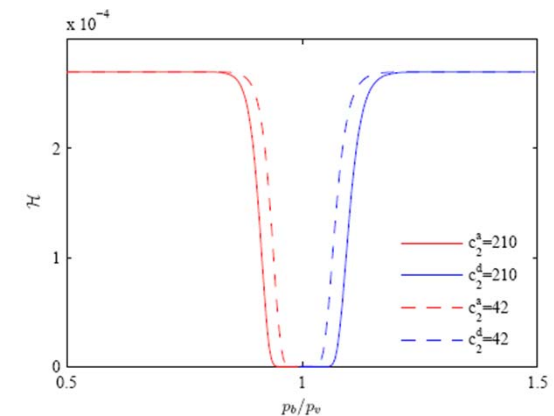
- $H_c$ : moisture-dependent reaction rate function
- $c_{bl}$ :  $\rho_0 m_{bl}$  ( $\rho_0$  density in the reference conditions ,  $m_{bl}$ : sorption curve)
- $h$ : relative humidity (%)

Sorption rate

$$\dot{c} = H_c(c_{bl} - c_b)$$

$$H_c = \begin{cases} C_1 \exp \left( -C_2 \left( \frac{c_b}{c_{bl}} \right)^{C_3} \right) + C_4 & c_b < c_{bl} \\ C_1 \exp \left( -C_2 \left( 2 - \frac{c_b}{c_{bl}} \right)^{C_3} \right) + C_4 & c_b > c_{bl} \end{cases}$$

$$C_2 = c_{21} \exp(c_{22}h) + c_{23} \exp(c_{24}h)$$



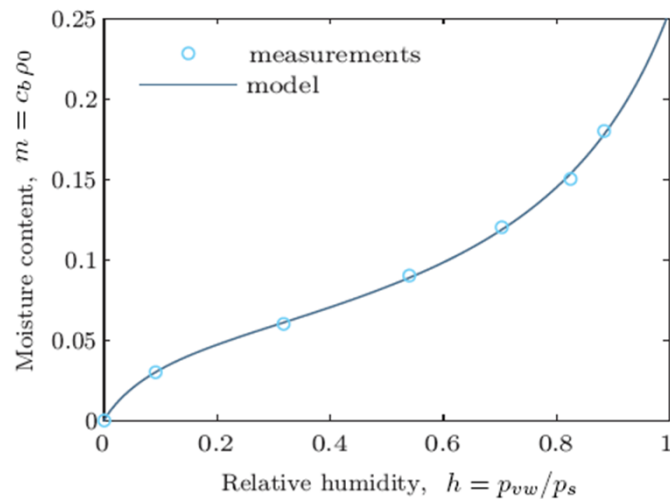
Variation of the  $H$  function with the relation between the equilibrium vapor pressure  $p_b$  and the actual vapor pressure  $p_v$ .

Sorption isotherm function

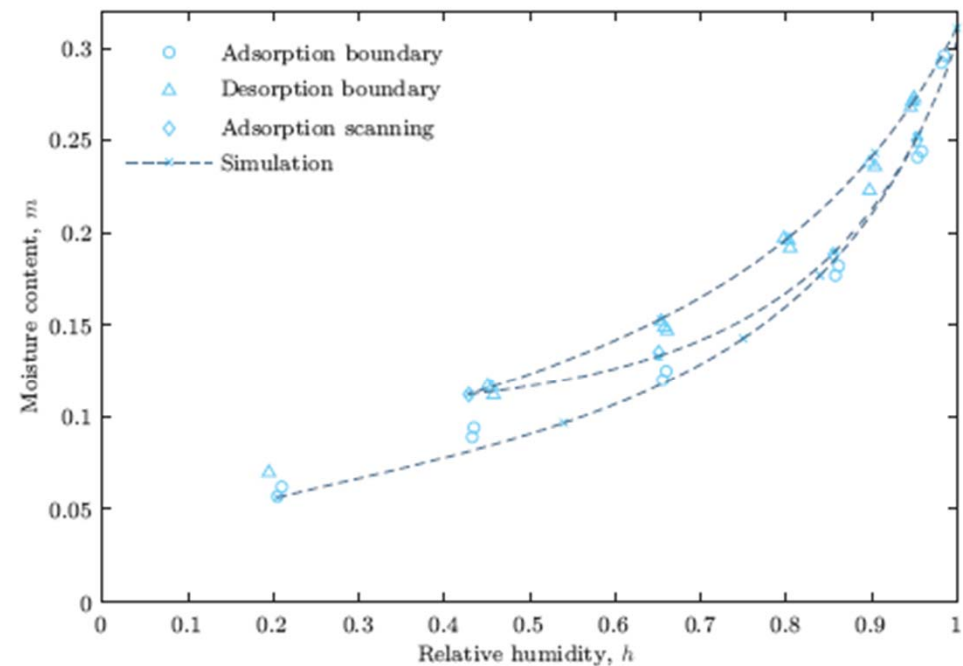
$$m_{bl} = \frac{h}{f_1 + f_2 h + f_3 h^2}$$

## Sorption isotherm and hysteresis

- Example of sorption isotherm and hysteresis curves used in (Frandsen et al., 2007)



Sorption isotherm for klinki pine at 40°C calibrated to the measurements by Christensen (1965).



Variation of MC due to the RH sequence in the text compared with the measurements by Ahlgren (1972).

Frandsen H. L. and Svensson S. Implementation of sorption hysteresis in multi-Fickian moisture transport.  
 Holzforschung 2007; 61(6): 693-701

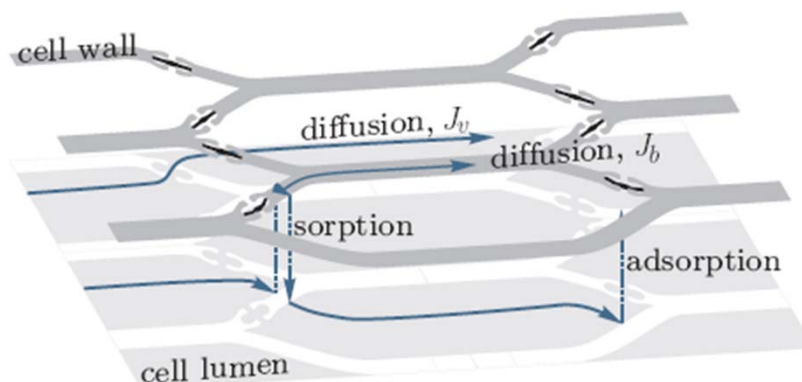
## Diffusion coefficient for water vapor (Schirmer, 1938)

$$D_v = \xi \left( 2.31 \times 10^{-5} \frac{p_{atm}}{p_{atm} + p_v} \left( \frac{T}{273K} \right)^{1.81} \right) m^2 s^{-1}$$

- $\xi$ : reduction factor ( $\xi_T$   $\xi_L$ )

## Diffusion coefficient for bound-water (Skaar and Siau, 1981)

$$D_b = D_b^0 \exp \left( \frac{-E_b}{RT} \right) m^2 s^{-1}$$



where  $E_b$  is the activation energy for bound-water diffusion ( $J mol^{-1}$ ). The diagonal terms of  $D_0$ , i.e.,  $D_T^0$  and  $D_L^0$  are equal to  $7 \times 10^{-6} m^2 s^{-1}$  and  $17.5 \times 10^{-6} m^2 s^{-1}$ , respectively, for a two-dimensional case (Siau 1984). The activation energy may be approximated by the linear expression  $E_b = (38.5 - 29m) \times 10^3 J mol^{-1}$  (Siau, 1995), where  $m = c_b/\rho_0$  is the moisture content and  $\rho_0$  is the dry density of wood. The decrease in activation energy with moisture content is due to the decrease in bonding energy at the sorption sites.

## Some Abaqus® operative details (analogy with heat transfer analysis)

Sequential analysis approach.

Solution of water vapor equation:

- **Moisture transfer analysis** (same differential equations as heat transfer analysis).
- **DFLUX user subroutine** for the evaluation of the surface moisture flow (→ surface heat flux in heat transfer analysis).
- **HETVAL user subroutine** for 1) the calculation of the sorption rate (→ internal heat flux generated in heat transfer analysis); 2) a first approximation of the bound water value:

$$c_b(t) = c_b(t - \Delta t) + \dot{c}(t) \Delta t$$

Solution of bound water equation:

- **Moisture transfer analysis.**
- **HETVAL user subroutine** for the calculation of the sorption rate.

## Material parameters used for the numerical model

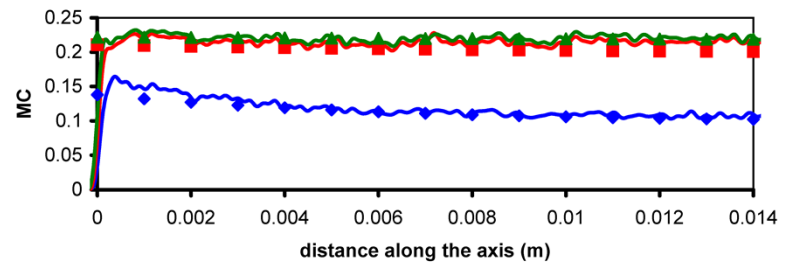
- **Dimensionless parameters for the sorption isotherm** (satisfying the results of NMR experiments for the equilibrium):

$$f_1 = 1.36, f_2 = 14.32, f_3 = -11.90$$

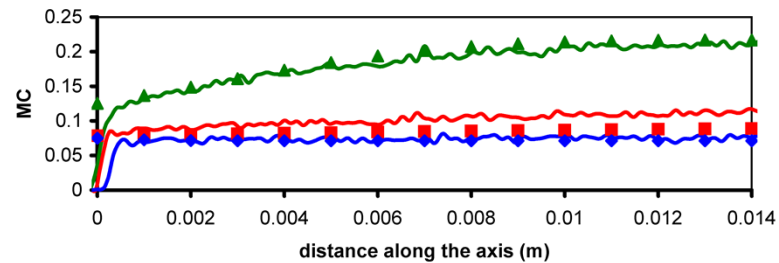
- **Reduction factors** ( $\xi_T = 0.11$ ,  $\xi_L = 0.9$ )
- **Shape parameters** of the function  $H_c$  ( $C_1, C_3, C_4$ ) and of function  $C_2$  ( $c_{12}$ ,  $c_{22}$ ,  $c_{23}$ ,  $c_{24}$ )

$C_1[s^{-1}]$	$C_3[-]$	$C_4[s^{-1}]$	$c_{12} [-]$	$c_{22} [-]$	$c_{23} [-]$	$c_{24} [-]$
3.8e-4	80	5.94e-7	3.58	2.21	1.59e-3	14.98

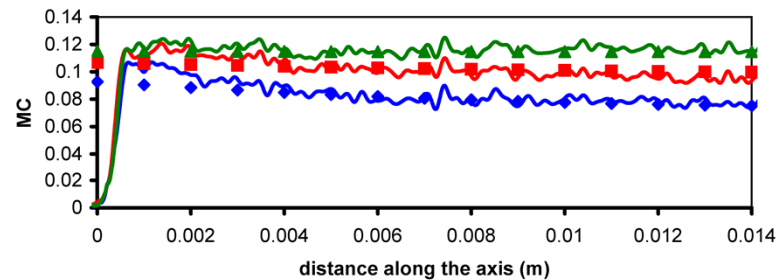
# Comparisons between experimental data and numerical results - Tangential uncoated case



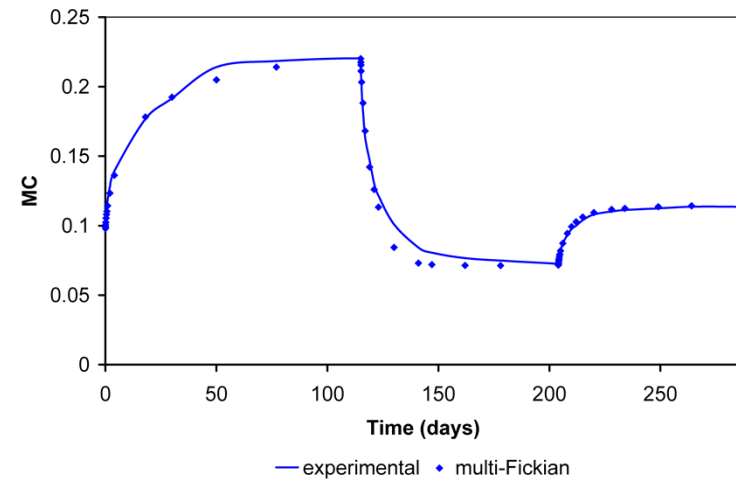
— experimental - 1 day    — experimental - 50 days    — experimental - 115 days  
 ◆ multi-Fickian - 1 day    ■ multi-Fickian - 50 days    ▲ multi-Fickian - 115 days



— experimental - 116 days    — experimental - 130 days    — experimental - 204 days  
 ▲ multi-Fickian - 116 days    ■ multi-Fickian - 130 days    ◆ multi-Fickian - 204 days



— experimental - 205 days    — experimental - 212 days    — experimental - 286 days  
 ◆ multi-Fickian - 205 days    ■ multi-Fickian - 212 days    ▲ multi-Fickian - 286 days

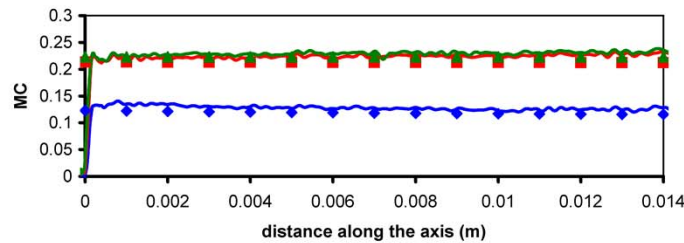


Average values over the axis of the specimen (0-14 mm).

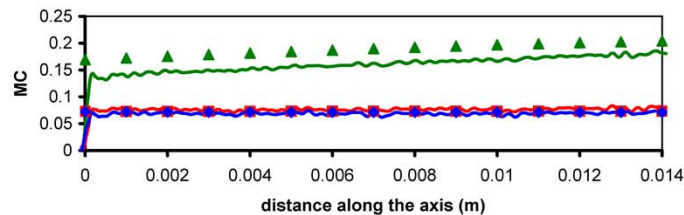
# Comparisons between experimental data and numerical results – Longitudinal uncoated case

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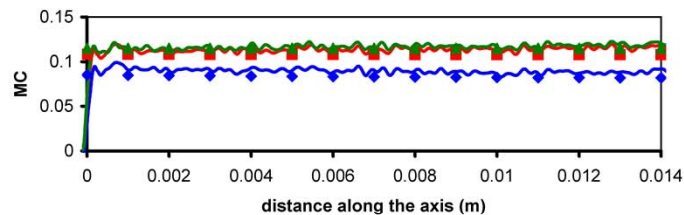
21



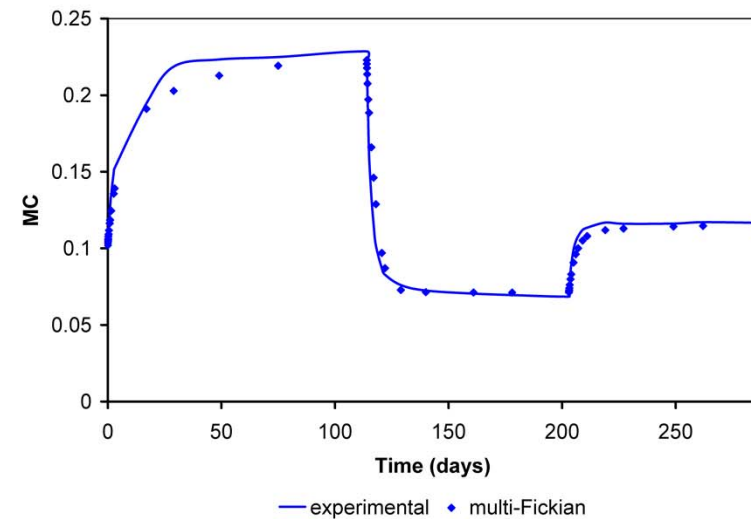
— experimental - 1 day    — experimental - 49 days    — experimental - 114 days  
◆ multi-Fickian - 1 day    ■ multi-Fickian - 49 days    ▲ multi-Fickian - 114 days



— experimental - 115 days    — experimental - 129 days  
— experimental - 203 days    ▲ multi-Fickian - 115 days  
■ multi-Fickian - 129 days    ◆ multi-Fickian - 203 days



— experimental - 204 days    — experimental - 211 days  
— experimental - 286 days    ◆ multi-Fickian - 204 days  
■ multi-Fickian - 211 days    ▲ multi-Fickian - 286 days



Average values over the axis of the specimen (0-14 mm).

## Scientific paper in *Engineering Structures*

Engineering Structures

Article in Press, Corrected Proof - Note to users



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### NMR imaging study and multi-Fickian numerical simulation of moisture transfer in Norway spruce samples

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Available online 17 May 2011.

#### Abstract

Wood has potential as a renewable material for a large variety of applications that often call for improved properties such as dimensional stability, moisture insensitivity, and durability. Moisture migration in wood is a particularly important factor in determining the cost-effective service life of wooden construction. Within the present research, proton NMR imaging was applied for recording the moisture spatial distribution of various samples of Norway Spruce. Moisture distribution along the radial, tangential and longitudinal directions in wood was monitored at different times upon three consecutive changes of relative humidity: (1) from 65% to 94%; (2) from 94% to 33%; (3) from 33% to 65%. Uncoated samples and specimens treated with different types of surface coatings were studied.

The experiments were numerically simulated by using the multi-Fickian model. The model describes the moisture transport process in wood which is characterized by three phenomena: (a) bound water diffusion, (b) water vapor diffusion and (c) coupling between the two phases through sorption. The model is implemented into the Abaqus FEM code. The numerical results are found to be in agreement with the experimental data.

**Keywords:** Bound water; Diffusion; FEM; Moisture transport in wood; Multi-Fickian models; Nuclear magnetic resonance (NMR) imaging



Thank You!

