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Real time monitoring of the through-thickness moisture profile of thin sheets using NMR

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ABSTRACT

The through-thickness distribution of liquids and liquid migration over time in thin, porous media is highly relevant for the production and product properties of materials such as textiles, membranes, paper, barrier materials, and thin films.

We present a 1D NMR profiling method for the analysis of through-thickness liquid distribution during liquid absorption, migration, and drying of thin sheets [1].

A key aspect of our method is the simultaneous scanning of a liquid saturated reference sheet. Using a reference, local liquid saturation and absolute amount of liquid present over time can be evaluated.

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INTRODUCTION

- The z -directional distribution of liquids in thin porous media and liquid migration over time is of interest in many fields such as textiles, membranes, production and printing of paper, barrier materials, coatings, and thin films.
- Temporal changes in moisture distribution are relevant when studying processes like liquid absorption, migration, diffusion, and drying. These substrate-liquid interactions are important both in the production of thin porous media as well as for the resulting product properties.
- In the production and printing of paper, the z -directional moisture profile during drying has a strong influence on the out-of-plane material deformation observed as paper curl.
- As liquid absorption, migration, diffusion and drying processes in thin porous media are fast and substrate thickness is in the range of tens of micrometers to a few millimeters, a spatially and temporally resolved method for measuring moisture distribution with both high temporal and spatial resolutions is required.

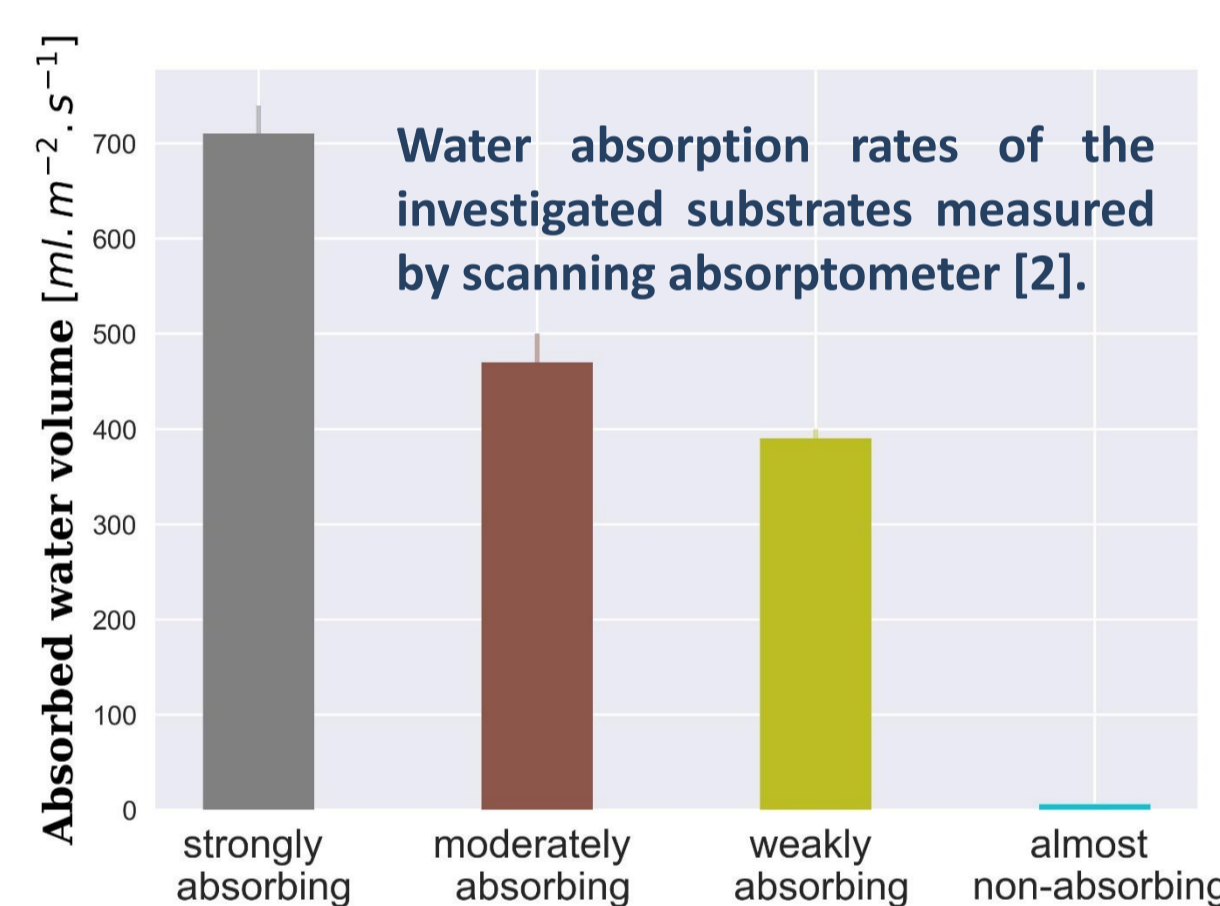
In the present study:

- We introduce a NMR-based method for observation of the moisture profile over the thickness of thin sheets.
- The method uses a custom-built surface NMR coil integrated in a device that allows the uniform application of a controlled quantity of liquid to the surface of the sheet.
- We perform an application study observing the drying of inkjet ink on different papers.

MATERIALS

Properties of the paper used in this study	Strongly absorbing	Moderately absorbing	Weakly absorbing	Almost non-absorbing
Grammage [g/m ²]	97.20	78.50	79.89	77.20
Thickness [mm]	129	89	92	105
Thickness (wet) [mm]	154	124	123	125
Porosity [%]	40.3	38.8	23.6	20.6
Water absorption rate [ml.m ⁻² .s ⁻¹]	710	470	390	6

The ink employed in this study was an ink-jet ink. These types of ink are mainly composed of water as solvent, together with diethylene-glycol and glycerol as co-solvents, and pigments.



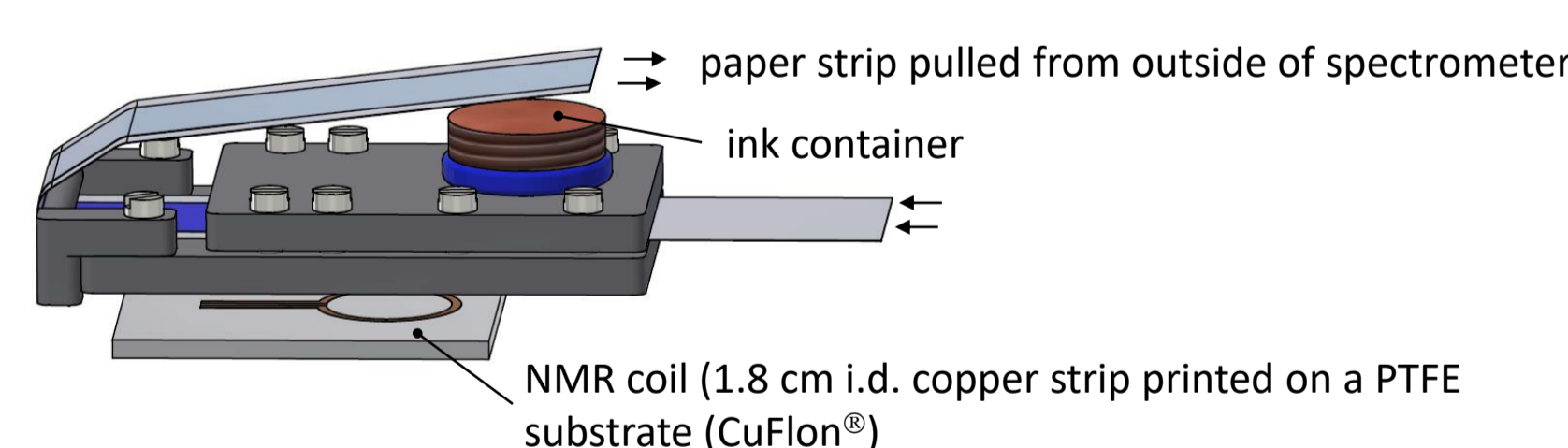
METHODS

a - NMR setup



Bruker Biospec 24/40
2,34 Tesla

2D probe and measuring cell



Specifications of the Bruker Biospec 24/40 (100 MHz)

opening	max. magnetic field gradient
∅ (cm)	g_{max} (G/cm)
20	20 (x, y, z)
6 (BGA6S insert)	100 (x, y, z)

b - experimental procedure

The paper strip is pulled from outside the spectrometer: it first passes under the marker tip which acts as an application sponge, and then reaches the NMR probe immediately after application. The strip is then stopped and the 1D moisture profiles are recorded as a function of time.

The 1D NMR imaging sequence is composed of a pair of r.f. pulses ($\pi/2, \pi$) separated in time by $TE/2$ produces a "spin echo" NMR signal at time TE . During the acquisition of the signal, a magnetic field gradient of amplitude g is turned ON for spatial encoding. The 1D profile is obtained by Fourier transform of the temporal signal. The echo time (TE) was set to 6.8 ms and the repetition time (TR) to 155 ms. $N = 8$ scans were accumulated to increase the SNR, giving a time resolution between consecutive 1D liquid concentration profiles of 1.24 s.

Each profile contains 256 points in a field of view of 2.5 mm, corresponding to a nominal spatial resolution of 9.77 $\mu\text{m}/\text{point}$. The sensitive surface of the coil is about 2.5 cm², so that the sample volume probed by each point of the profile is about 0.79 mm³.

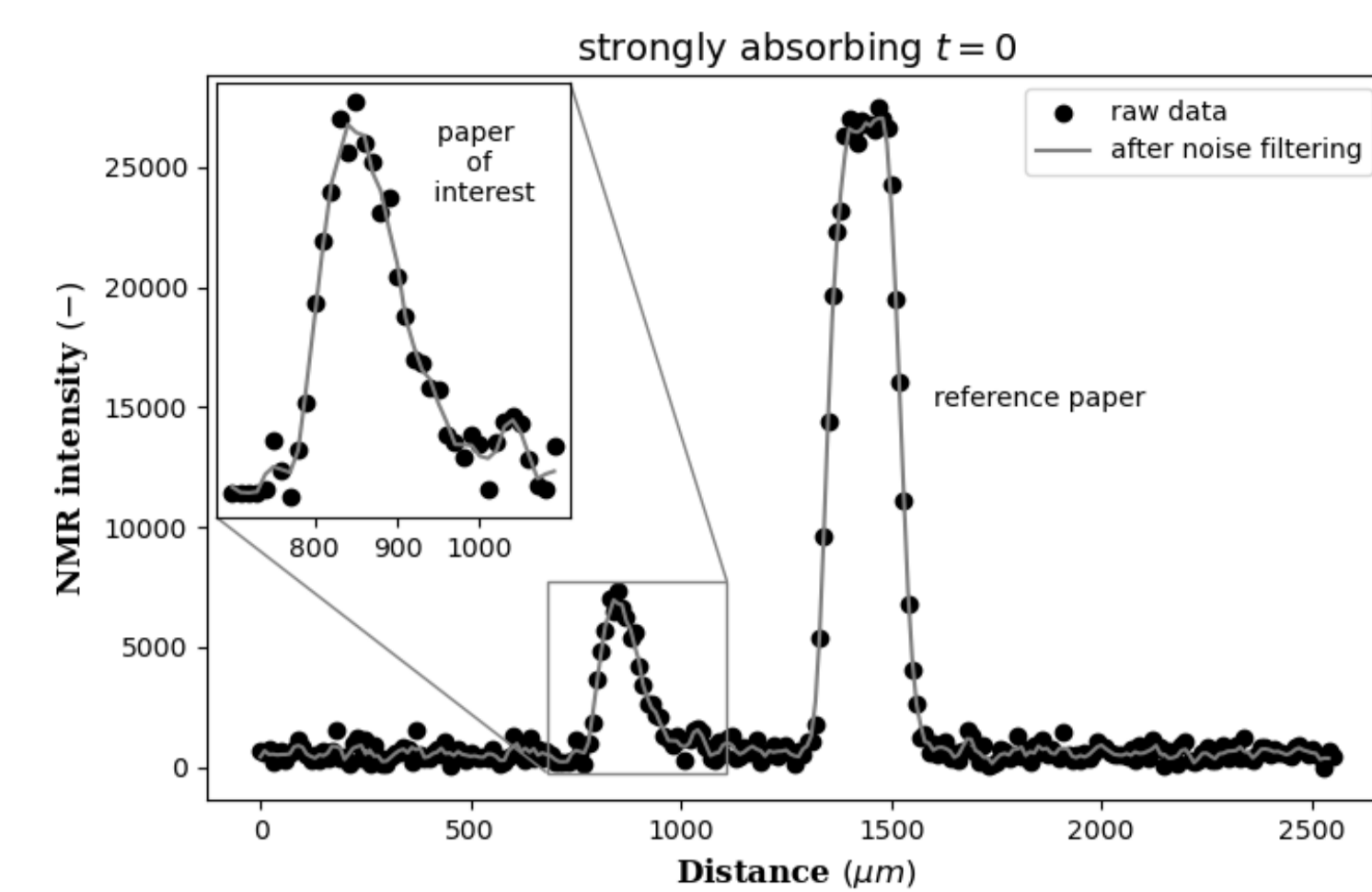
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[1] This study : *Chemical Engineering Science* 251 (2022) 117464.

[2] Krainer, S., Hirn, U., 2018. Short timescale wetting and penetration on porous sheets measured with ultrasound, direct absorption and contact angle. *RSC Adv.* 8, 12861–12869.

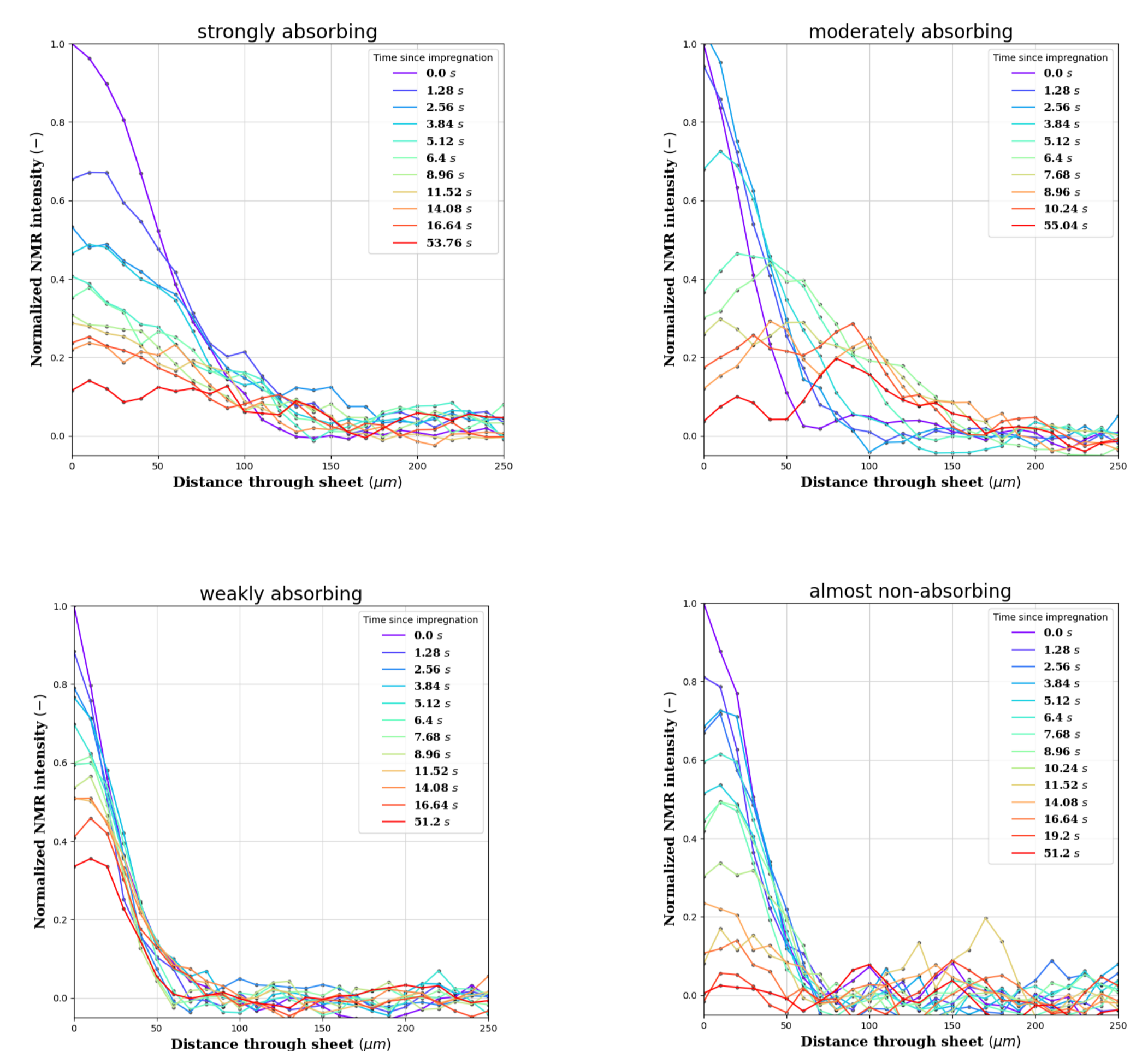
RESULTS

a - Example of water profile in a strongly absorbing paper just after ink application (t = 0)



The raw data of the profiles were first filtered using a Wiener function. A baseline correction was then performed by subtracting the average intensity of the first 50 points. The signal-to-noise (SNR) ratio of this measurement is > 40.

b - Through-thickness moisture profiles after different drying times for paper samples

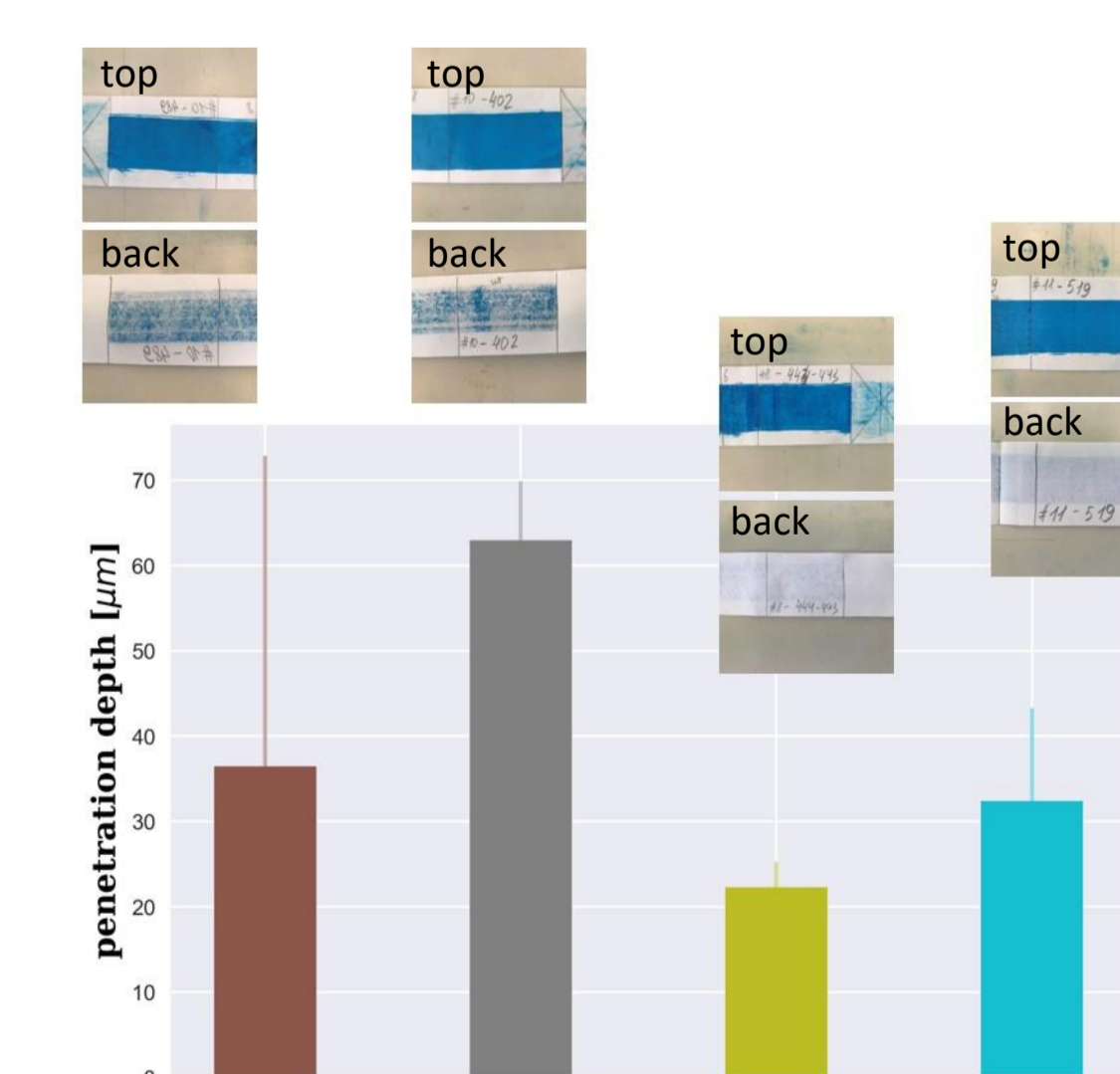
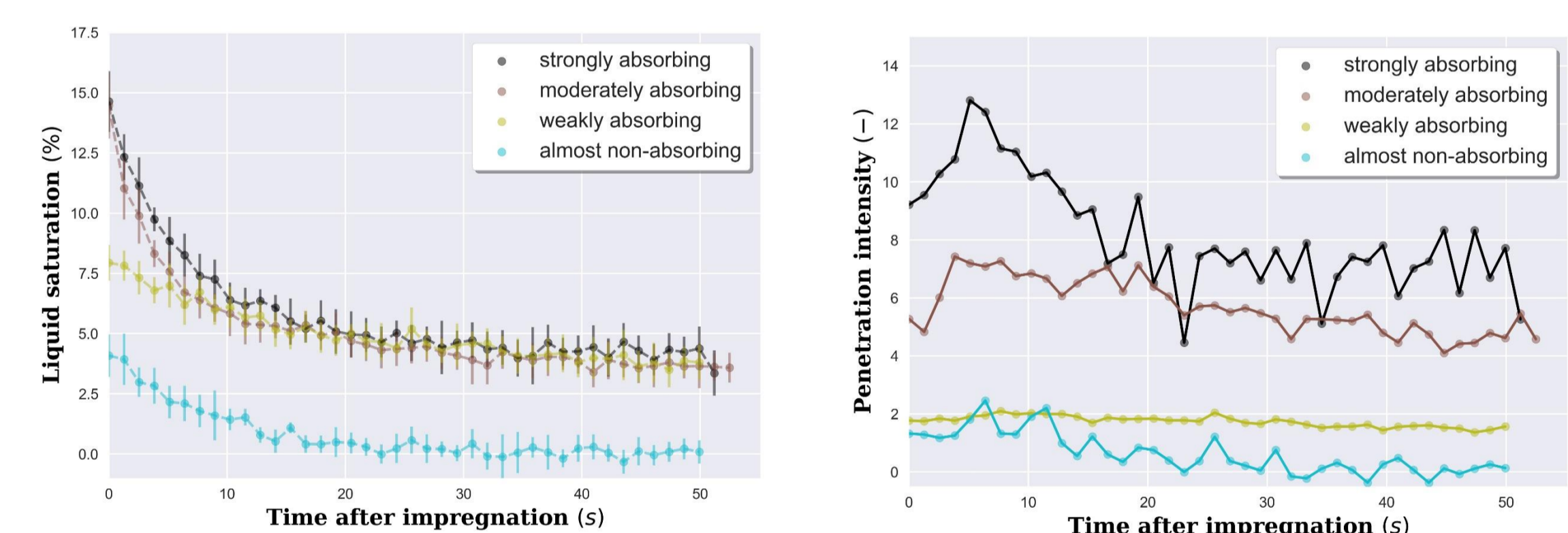


Through-thickness moisture profiles after different drying times for paper samples

- Moderately absorbing and strongly absorbing paper:** the water was redistributed quickly throughout the sheet leading to rather flat profiles after a short amount of time. Also, the NMR intensity decreased fast, indicating quick drying.
- Almost nonabsorbing paper:** showed hardly any redistribution, as the z -directional moisture profile is not flattening or covering the whole thickness at any time. Can essentially be regarded as completely dry after only about 10s.

c – penetration intensity

Penetration intensity is calculated as penetration depth \times relative liquid saturation



While the moderately absorbing and strongly absorbing paper exhibited only minor differences in drying kinetics and liquid saturation, they differed significantly more in terms of penetration intensity. The strongly absorbing paper thereby clearly displayed the highest penetration intensity. This is in line with the strongly absorbing paper absorbing much greater water amounts than the moderately absorbing paper in the ASA measurements. In addition, the almost non-absorbing paper exhibiting the lowest penetration intensity is consistent with the ASA measurements as well.

CONCLUSIONS

- We have introduced an NMR based setup for comprehensive z -directional analysis of liquid distribution and migration in thin sheets.
- We believe that this method is a suitable tool to quantify absorption, migration and drying processes, and thus supports a better understanding of moisture distribution and transport processes in thin sheets.