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*Tapio Fabritius*

# OPTICAL METHOD FOR LIQUID SORPTION MEASUREMENTS IN PAPER

FACULTY OF TECHNOLOGY,  
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*TAPIO FABRITIUS*

**OPTICAL METHOD FOR LIQUID  
SORPTION MEASUREMENTS  
IN PAPER**

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## **Fabritius, Tapio, Optical method for liquid sorption measurements in paper**

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### ***Abstract***

This thesis presents an effective optical method for measuring liquid sorption into paper. From the two tested methods, based on a streak-camera and optical coherence tomography (OCT), the last-mentioned proved very promising for investigating dynamical paper-liquid interactions as spatially and temporally dependent processes.

The streak-camera measurements were performed to explore the relationship between paper properties and light migration in dry and refractive index matched paper in general. Based on streak-camera measurements, a novel procedure for determining the average refractive index of cellulose fibre tissue was also presented here. In addition, the streak camera method lent itself to paper porosity determination.

Results of the performed OCT measurements proved that liquids cannot penetrate into paper before filling the pores and pits of the paper surface. As a liquid penetrated into paper, the border between the wetted and dry area could be investigated in the depth direction. The liquid penetration velocity seemed to be slower at the beginning and end of the process. Liquid absorption into paper fibres could be investigated concurrently. For the first time, the location and moment of structural changes in paper could be determined during wetting, and the effect of three different coexistent subprocesses related to paper wetting could be detected. OCT only fell short of detecting the effect of liquid migration along fibres.

Despite the limitations of the utilized method (resolution, probing depth and depth scanning rate), the obtained OCT measurement results are very promising for the development of an effective paper wetting measurement device for industrial applications. Even if this thesis focused on paper wetting, it is reasonable to assert that the presented ideas and obtained results have more general value in terms of explaining liquid penetration into porous structures and offer an alternative method of evaluating that process.

*Keywords:* optical coherence tomography, paper testing, streak-camera, swelling, wetting



## Acknowledgements

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I would like to acknowledge the assistance and support that so many people have provided during this work. Firstly, I want to express my sincere gratitude to my supervisor, Professor Risto Myllylä, for providing me with the opportunity to write this thesis and for his support and advice throughout the entire course of this work.

I also wish to thank the entire personnel in the Optoelectronics and Measurement Technology Laboratory at the University of Oulu. And I owe special thanks to the co-authors of the publications included in this thesis: Jukka Hast, Erkki Alarousu, Juha Saarela and Tuukka Prykäri. Furthermore, I'm grateful for the comments and suggestions of Professor Alexander Priezhev from Moscow State University and Professor Valery Tuchin from Saratov State University.

Thanks go to Mr. Rauno Varonen for checking the English of the separate publications and of this thesis.

While conducting this work, I attended the Graduate School of Modern Optics and Photonics and the Graduate School in Electronics, Telecommunications and Automatics. The work was also supported by the M.H. Paloheimon erikoisrahasto foundation.

Last, but most important, I wish to express my deepest gratitude to my family, parents, brothers and sister, who have supported and encouraged me throughout my research.

Oulu, October 2006

Tapio Fabritius



## Abbreviations and symbols

2D	Two dimensional
3D	Three dimensional
CCD	Charge-coupled device
CLSM	Confocal laser scanning microscopy
FD-OCT	Fourier domain optical coherence tomography
FF-OCT	Full field optical coherence tomography
OCT	Optical coherence tomography
SLD	Super luminescent diode
TAPPI	Technical Association of Pulp and Paper Industry
TOF	Time of flight
$g$	optical anisotropy parameter
$r$	radius
$n_1$	refractive index of surrounding
$n_2$	refractive index of ground material
$m$	ratio of refractive indices
$l_R$	optical path length in reference arm
$E_r$	electromagnetic field in reference arm
$E_s$	electromagnetic field in sample arm
$l_s$	optical path length in sample arm
$I_d$	detected interference signal
$I_s$	detected light intensity backscattered by the sample
$I_R$	detected light intensity reflected by the reference mirror
$C$	interferometric response in ideal case
$R$	reflectance
$t_d$	propagation delay
$n_c$	refraction index of cellulose
$t$	time
$B$	grammage
$L$	physical thickness
$K_t$	specific light transmission constant

$K_d$	specific light propagation delay constant
$T$	light transmission coefficient
$\mu_a$	absorption coefficient
$\mu_s$	scattering coefficient
$\mu_s'$	reduced scattering coefficient
$\Delta l$	optical path length difference
$\Delta n$	refractive index difference
$\Delta\mu_a$	absorption coefficient difference
$\Delta\mu_s$	scattering coefficient difference
$\phi$	porosity
$\rho_f$	density of cellulose

## List of original papers

This thesis is a summary of the following papers published in international referee journals (Supplements III, IV and VI) and conference proceedings (Supplements I, II and V):

- I Fabritius T, Saarela J & Myllylä R (2006) Photon migration in highly scattering and low-absorbing media during compression measured with streak-camera, Proceedings of SPIE, Saratov Fall Meeting Conference, Saratov, Russia 26-30 Sept. 2005. 6164 0E 1-8.
- II Fabritius T, Saarela J & Myllylä R (2006) Determination of the refractive index of paper with clearing agents, Proceedings of SPIE, International Conference on Lasers, Applications, and Technologies, St. Petersburg, Russia 11-15 May 2005. 6053 0X 1-8.
- III Fabritius T, Alarousu E, Prykäri T, Hast J & Myllylä R (2006) Characterization of optically cleared paper by optical coherence tomography, Quantum Electronics 36(2), 181-187. ([www.turpion.org/php/paper.phtml?journal\\_id=qe&paper\\_id=13121](http://www.turpion.org/php/paper.phtml?journal_id=qe&paper_id=13121))
- IV Fabritius T & Myllylä R (2006) Investigation of swelling behaviour in strongly scattering porous media using optical coherence tomography, Journal of Physics D: Applied Physics 39: 2609-2612. ([www.iop.org/journals/jphysd](http://www.iop.org/journals/jphysd))
- V Fabritius T & Myllylä R (2006) Dynamic optical coherence tomography for paper wetting measurements, Proceedings of SPIE, International conference of Optics and Photonics 2006, San Diego, USA 13-18 Aug. 2006. 6293 07 1-6.
- VI Fabritius T & Myllylä R (2006) Liquid sorption investigation of porous media by optical coherence tomography, Journal of Physics D: Applied Physics 39: 4668-4672. ([www.iop.org/journals/jphysd](http://www.iop.org/journals/jphysd))

Author's contribution to publications I-VI:

- I – II Together with J. Saarela, one of the co-authors, the author defined the research plan and conducted the measurements. He analyzed the research results, carried out the literature review, wrote a first version of the manuscript and, after discussions with the co-authors, produced the final paper.

- III With E. Alarousu, one of the co-authors, the author defined the research plan. He then carried out the light transmission measurements together with Alarousu and Prykäri. Optical coherence measurements and data modification were made by Alarousu. The author analyzed the obtained measurement results, carried out the literature review and wrote a first version of the manuscript, except the section on the theoretical background of the optical coherence tomography method, which was written by Alarousu. After discussions with the co-authors, the author wrote the final paper.
- IV-VI The author defined the research plan, carried out the experiments and analyzed the research results. He also conducted the literature review, produced a first version of the manuscript and, after discussions with the co-author, wrote the final paper.

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# 1 Introduction

Testing the end products of a manufacturing process is a common feature for all industrial processes. As industrial manufacturing processes become more sophisticated and requirements for each product's end-use quality properties increase, the importance of reliable and relevant testing grows. In this way, testing has come to play a vital role in all industrial activities, including paper manufacturing [1].

Within the paper industry, wetting of paper and fibres is an important phenomenon in a number of processes, including sizing [2,3,4,5], coating [6,7,8,9] and printing [10,11,12]. The significance of liquid interactions is well recognized by the industry, and it has been the subject of numerous theoretical and experimental studies. Liquid interactions in idealized solids are quite well understood in equilibrium conditions. However, complications are introduced by rough, heterogeneous and porous solids such as paper. When non-inert liquids like water interact with paper, its chemical and physical properties are temporally altered by a number of different coexistent subprocesses. Consequently, it is necessary to clarify all details related to dynamic interactions between paper sheets and liquids.

Among the most important largely unexplored subjects in paper testing are liquid transport properties and dynamic interactions with liquids [13]. The main ambition of this thesis is to develop an optical method for paper wetting measurements in all three directions, with particular attention placed on investigating wetting phenomena in the depth direction. For a better general understanding of the dynamic paper wetting process, this thesis seeks answers to the following questions: Why is liquid penetration into paper delayed? Can the borderline between a wetted and dry area be separated in the depth direction? How does penetration velocity behave temporally? How are paper properties affected by interaction with a liquid during wetting?

This thesis is a compendium of six separate publications where the effect of refractive index matching on the physical and optical properties of paper is investigated experimentally using two different optical methods. The results are presented and discussed taking into consideration the main ambition of the thesis, although the obtained results are also considered in a larger perspective.

## 1.1 Related work on paper wetting investigation

Techniques for measuring wetting and the penetration of liquids into paper have traditionally not distinguished aspects related to surface chemistry, morphology and swelling. Neither have they attempted to replicate the highly dynamic conditions that pertain in processes where wetting and penetration are important [14]. Despite their limitations, many of the conventional methods used in measuring paper wetting, such as the Cobb test, drop penetration test or contact angle measurement, have a TAPPI standard and play an important role in industrial testing [15,16,17]. To cut a long story short, although liquid spreading on the paper surface can be measured very effectively, there is still no method for exploring liquid sorption in the depth direction.

Many research papers have presented theoretical models and simulations of wetting and liquid penetration into porous structures such as paper [18]. Even if the reported findings are relevant and important for understanding liquid penetration and wetting properties, this chapter only reviews the latest findings concerning paper wetting measurements in pure liquids.

The applicability of the contact angle method to studying paper-liquid interaction is still being analyzed. At the beginning of the millennium, Shen *et al.* investigated the effect of paper sizing on contact angles. For that purpose, they used a confocal laser scanning microscopy (CLSM) device for the first time and concluded that, if calculations are based on apparent contact angles, the heterogeneity of paper produces erroneous liquid penetration values [19]. Modaresi and Garnier investigated the effects of chemical and physical heterogeneity on the mechanism of wetting and water absorption in sized paper [20]. They utilized a CCD video camera to measure dynamic contact angle behaviour during wetting and found that wetting depends on physical properties, but is independent of the chemical heterogeneity of the paper's surface structure. Kannangara *et al.* studied the influence of liquid drop impact and recoil on liquid-paper interaction [21]. Also they used a CCD camera for dynamic contact angle measurements and concluded that the impact of a droplet on paper surface is highly dependent on the droplet's kinetic energy and on liquid-substrate interactions.

Paper wetting behaviour can also be investigated using ultrasonic devices. Stor-Pellinen *et al.* have performed ultrasonic transmission experiments on paper. In their first publication, they suggested that capillary wetting within pores and diffusion wetting into fibres could be distinguished by their method [22]. In a second paper, they studied the effect of high-power ultrasound on wetting and concluded that its application resulted in faster sorption processes [23]. In a third publication, they investigated changes in surface roughness during paper wetting and demonstrated that dynamical roughness changes can be determined [24].

One alternative method to measuring the transport rate of liquids into paper has been presented by Miyauchi and Nakanishi [25]. They measured electrostatic capacitance behaviour during liquid penetration and showed that the method can be used to measure liquid penetration in the depth direction of a thin paper sheet.

Moreover, Karppinen *et al.* have presented an optical method for paper wetting measurements. Measuring the transmitted light intensity behaviour of monochromatic light during wetting, they showed that pure capillary and diffusion wetting can be

distinguished from each other [26]. The same author has also published research where liquid penetration in the thickness direction was investigated by measuring reflected light [27].

The major disadvantage of the methods presented above is that they observe the wetting phenomena as a macro-scale process. In practise, this means that temporally and spatially dependent sorption processes cannot be measured directly. To overcome this limitation, these measurement methods require further development.

## 1.2 Organization of the thesis

This thesis is organized as follows:

- Chapter 2 addresses issues affecting light behaviour in refractive index matched paper. First, the structural properties of paper are presented, followed by a brief discussion on light migration in porous highly scattering media. Finally, the physical background of refractive index matching and its relation to paper wetting are presented.
- Chapter 3 discusses the used materials and methods. The first part of this chapter concentrates on streak-camera measurements and the second part on optical coherence tomography (OCT) measurements.
- Chapter 4 presents the main results of this thesis, highlighting such results as have a particular significance for paper sorption measurements.
- Chapter 5 contains a discussion on the obtained results with a focus on the contribution of this thesis on paper wetting measurements. Also discussed are the limitations of the used OCT method and some suggestions are made to avoid those restrictions.
- Chapter 6 provides a summary of the results.

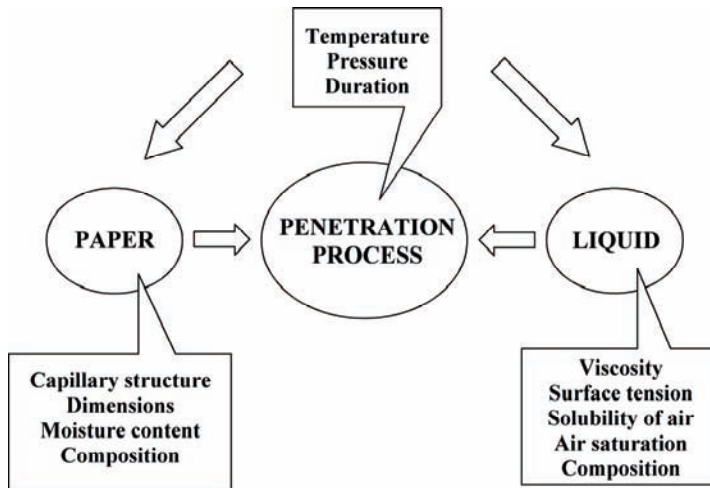
## **2 Paper wetting**

At the beginning of this chapter, the most important features affecting liquid sorption in paper will be presented, followed by a discussion on the structural properties of paper that have an influence on photon migration. From the optical point of view, liquid penetration into paper is a dynamical process where scattering properties change due to refractive index matching. The physical background to this phenomenon will also be discussed.

### **2.1 Sorption properties of paper**

Liquid sorption into paper is a very complicated phenomenon. At least four different coexistent subprocesses occur during this process: liquid filling of the pores and pits of paper surface, capillary penetration through pores and cavities, migration along the fibres and absorption and diffusion inside ground material [27]. Factors that influence those subprocesses can be categorized into three groups (Figure 1), of which the first includes factors related to the paper sheet (capillary structure, dimensions, etc.), while the second group includes factors related to the liquid (viscosity, surface tension, etc.). The third group contains process conditions (temperature, pressure, etc.). Also circumstance factors affect the paper sheet and the liquid. Conclusions concerning the most important factors and the relationships between them are presented in Figure 1 [28].

Sorption has a significant effect on paper structure formation during manufacture. In the paper coating process, for example, liquid sorption from the coating pasta to ground paper plays a major role [6,7]. On the other hand, sorption properties are also important in terms of the paper's end-use quality. For packing papers, liquid sorption must be minimized, while the sorption characteristics of filtering paper have to be fairly high. Liquid sorption also plays an important role in paper printing [14]. An effective method of investigating sorption properties is necessary, because a better understanding of liquid sorption enables improved control of manufacturing processes which, in turn, ensures better end-use properties.



**Fig. 1.** Effect of different factors on liquid penetration into paper. The factors relate to liquid properties, the paper sheet and process conditions. Factors in the third group also affect the other factors.

## 2.2 Photon migration in paper

### 2.2.1 Paper structure

The most important ingredients of paper products are cellulosic fibres, made from wood or other similar natural origins and the pore space formed by and in between the fibres. Inhomogeneity in paper arises from the sheet forming process. Thus, the stochastic nature of the drainage process results in a planar network structure where the positions, orientations and shapes of the fibres are partially random [13,29,30].

Fiber shapes can be altered depending on the origin of the pulp and the used treatment. The length of hardwood fibres is about 0.8-1.3 mm, while their width is typically a hundred times smaller. On the other hand, the length of softwood fibres is 1mm, and their width is 50 times smaller. In high quality papers, such as printing paper, different fillers and coatings, measuring a few microns in size, are used to improve the product's appearance. The chemical bindings between the cellulose fibres, filler or coating particles and the used sizing agents construct the three dimensional structure of paper, and determine its porosity and surface roughness [31,32].

### 2.2.2 *Light and random media*

As light strikes a paper surface, it scatters. In this process, the numerical uniformities of paper, known as scatterers, influence the light waves' propagation path. The optical properties of highly scattering materials like paper can be described by the absorption coefficient ( $\mu_a$ ), scattering coefficient ( $\mu_s$ ) and anisotropy factor ( $g$ ). If the refractive index difference between the environment ( $n_1$ ) and the scatterers ( $n_2$ ) is sufficiently large and the size of the scatterers is large enough ( $r \gg \lambda$ ), as in paper, the scattering anisotropy is rather high, the propagation direction changes considerably and photon paths rapidly become chaotic.

The refractive index of cellulosic fibres is about 1.55, while the corresponding value for typical filler and coating particles is 1.5-2.7, depending on the type of additives used [33]. Thus, light scatters strongly in paper, and the scattering coefficient ( $\mu_s$ ) is also large [34,35]. However, the absorption coefficient of paper is very small [36]. Moreover, the anisotropy factor ( $g$ ) of dry paper is typically about 0.8-0.9, which means that paper is a strongly forward scattering material [37].

Influential early studies and theories on light migration in paper include those by Kubelka & Munk and Scallan & Borch [38,39]. Since then, other researchers have presented more sophisticated models [36,37,40]. One of the biggest problems afflicting models describing the optical properties of paper is that some influential factors are not known exactly. Most models also fail to account for the physical and chemical changes that occur during measurements. As a result, theoretical models only provide general information about optical phenomena in paper which has been wetted with a liquid.

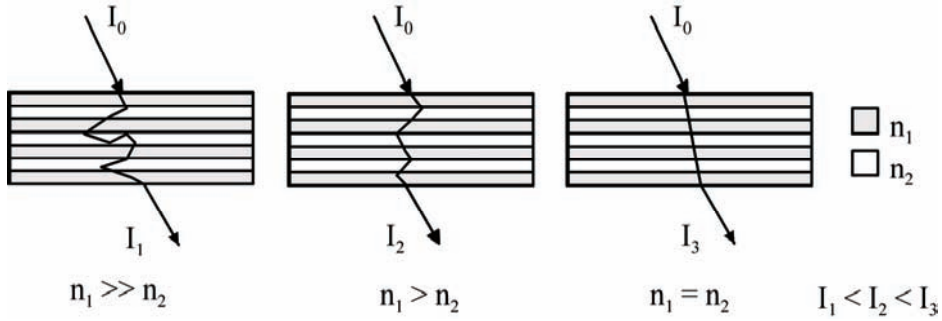
### 2.2.3 *Refractive index matching of paper*

The refractive index matching method is usually referred to as either the optical clearing or the immersion method. In paper research, the term optical brightening is used when fluorescent additives are used to improve the appearance of high quality paper products [41]. Being more specific, the term refractive index matching will be used in this paper to avoid confusion.

In a dry paper sheet, light scatters at the air-solid ( $n_1$  and  $n_2$ ) interfaces. Assuming that all the components of paper have similar refractive indices  $n_2 \sim 1.55$ , total light scattering is a function of the air-solid interfaces. When a refractive index matching liquid is used, paper pores are filled with a solute whose refraction index is larger than that of air. This serves to reduce the refraction mismatch, thereby decreasing the light scattering coefficient. The ratio  $m = n_1/n_2$  determines the reduced scattering coefficient so that as  $m = 1$ , the reduced scattering coefficient  $\mu'_s = (1-g)\mu_s \rightarrow 0$  [42]. However, a systematic investigation on how refractive index matching affects the optical properties of paper will not be performed here.

Fig. 2 shows the effect of a decreased refractive index mismatch on photon migration. A higher refractive index difference between the pores and fibres ( $n_1 \gg n_2$ ) results in a longer photon trajectory. Many scattering events occur before photons get through the

paper tissue. Conversely, as the refractive index difference is reduced, the transmitted intensity of light increases [42].



**Fig. 2. Effect of refractive index matching on light migration in a layered structure. When the refractive index difference between the layers decreases, the propagation path of photons also decreases, while the transmitted light intensity increases.**

In medical applications, refractive index matching of liquids such as glucose and glycerol is used to improve the light penetration properties of the tissues under study [42]. A wide range of studies on the water content of paper and its influence on light migration have demonstrated that the refractive index matching method is also applicable to paper research [43].

## **3 Materials and methods**

Two promising optical methods are used in this thesis. Supplements I and II use the streak-camera method and Supplements III-VI optical coherence tomography (OCT). This chapter presents the operation principles of both methods and a description of the measurement procedures and the materials used.

### **3.1 Used materials**

Two different types of paper sample were used in this thesis. Supplement III measured industrially manufactured copy papers (Optitext) with a grammage of  $80 \text{ g/m}^2$  and a physical thickness of  $102 \text{ }\mu\text{m}$ . Detailed information about the raw material components was not available. Supplements I, II and IV-VI investigated laboratory manufactured sheets, made from fully bleached, unbeaten, soft kraft wood pulp.

In paper wetting measurements, glycerol was used as test liquid (Supplements V and VI), while the other supplements used several other refractive index matching liquids with refractive indexes between 1.33- 1.741. A more detailed description of them can be found in Supplements II-IV.

### **3.2 Streak-camera method**

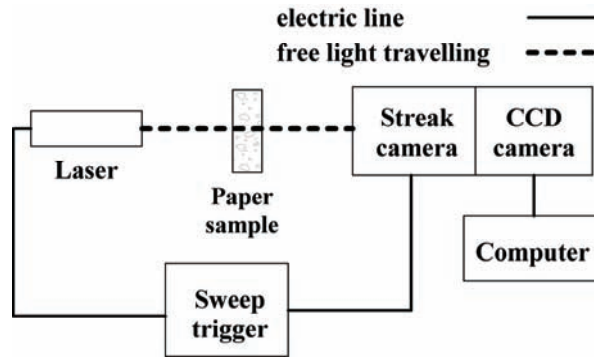
To measure the light transmission and propagation delay properties of paper, a streak-camera system was used. A streak-camera measures ultra-fast light phenomena and delivers information about light intensity as a function of time and position.

When the streak-camera device is used, the light beam travelling through the sample is focused on the photocathode of a streak cube. Light hitting the photocathode is converted into a number of electrons, which then pass a pair of sweep electrodes, directing them towards slightly differing locations on a microchannel plate (MCP). As the electrons pass the MCP, they are multiplied and impacted against the phosphor screen, where they are

converted back into light. More detailed information about the operation principle of the streak-camera can be found in [44].

In this work, short laser pulses were focused on a paper sample and photons passing through it were detected by a streak-camera. Coordination of the incident light pulses and camera operations were performed by the triggering section. A sensitive CCD camera was set to take pictures from the streak-camera's phosphor plate, and the pictures were subsequently processed using a computer.

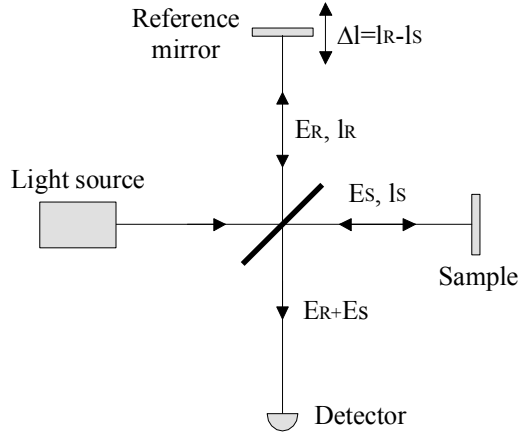
Fig. 3 shows a basic configuration of the used streak-camera system. In Supplement I, the paper sample was set between two hardened glass plates that were compressed by a special presser. Moreover, Supplements I and II employed a gallium arsenide laser with a central wavelength of 905 nm at room temperature. The laser's nominal pulse power was 100 W, and the pulse's full width at the half maximum was 40 ps. The temporal resolution of the used streak-camera was 2 ps.



**Fig. 3. Schematic of the measurement system used in the streak-camera measurements. A short laser pulse is shot to the sample and the photons travelling through it are detected by the streak-camera. A sweep trigger synchronizes the laser and the streak-camera, and the obtained data are processed by a computer.**

### 3.3 Optical coherence tomography method

Optical coherence tomography (OCT) is a cross-sectional imaging technology, which uses a low coherence optical source to perform reflectometry measurements on the microstructures of materials. OCT utilizes an interferometer to measure light reflected from scattering structures with a high spatial resolution ( $<10 \mu\text{m}$ ) and sensitivity ( $>100 \text{ dB}$ ). The method was originally developed for imaging such biological tissues as the eye and skin as well as various types of skin tumours [45,46]. Later, the same technique has also been applied to the study of other materials, such as paper [35,47].



**Fig. 4. Simplified schematic of the OCT system based on an open-space Michelson interferometer. Light from the source (SLD) is divided into two parts by a beamsplitter. Of these, the first part goes to a reference mirror ( $E_r$ ), while the second one is directed to the sample ( $E_s$ ). Moving the reference mirror allows scanning in the depth direction. The modulated interference signal is detected by a detector.**

The optical coherence tomography method is based on low coherence interferometry. Fig. 4 presents a simplified version of an open-space Michelson interferometer. Since OCT is based on coherence measurements, the signal is obtained if the optical path lengths of the reference and measurement arms are within the source's coherence length. Interference modulation of the detected signal can be described by the following equation:

$$I_d(l_s, l_R) = 2\sqrt{I_S I_R} \left( \sqrt{R(l_s)} \otimes C(l_s, l_R) \right) \quad (1)$$

where  $l_s$  and  $l_R$  are the optical path lengths in the sample and the reference arm, respectively;  $I_s$  is the detected light intensity backscattered by the sample; and  $I_R$  is the detected light intensity reflected by the reference mirror. In Eq. (1),  $(R(l_s))^{1/2}$  acts as normalized path-length-resolved reflectance or a normalized derivative from the light intensity depth distribution of the measuring wave, representing a fraction of the power reflected from the layer located at position  $l_s$  within the object. The symbol  $\otimes$  denotes convolution operation and  $C(l_s, l_R)$  represents the interferometer's response in the ideal case with a mirror in both arms.

In short, OCT traces out local relative variations in path-length resolved reflectance induced by local inhomogeneities in an object's optical properties, including the refractive index  $\Delta n$ , scattering coefficient  $\Delta\mu_s$ , anisotropy  $\Delta g$  and absorption coefficient  $\Delta\mu_a$  [46].

In this thesis, two different OCT devices were used. Thus, Supplement III utilized an open-space OCT laboratory device, whereas Supplements IV-VI made use of a commercially manufactured device (Institute of Applied Physics, Nizhny Novgorod, Russia). In both cases, data from the detector was saved and processed on a computer. A detailed description of the measurement setups and data handling methods can be found in Supplements III-VI.

## 4 Results

The results of this thesis are divided into two parts, of which the first focuses on the effects of paper and liquid properties on light propagation in a wetted sample. This part serves as an introduction to the second part, which concentrates on characterizing the liquid sorption properties of paper.

### 4.1 Streak-camera studies of light propagation

This chapter aims to clarify the influence of refractive index matching on the optical properties of paper and seeks answers to the following questions: What is the optimum refractive index of a liquid for paper studies? How do porosity and thickness affect light scattering? How do optical properties change as liquids with different refractive indexes interact with paper?

The results reported in this part were obtained by a streak-camera device using laboratory manufactured cellulose sheets as test samples. A more detailed description of the measurement setup and the used paper samples are found in Supplements I and II.

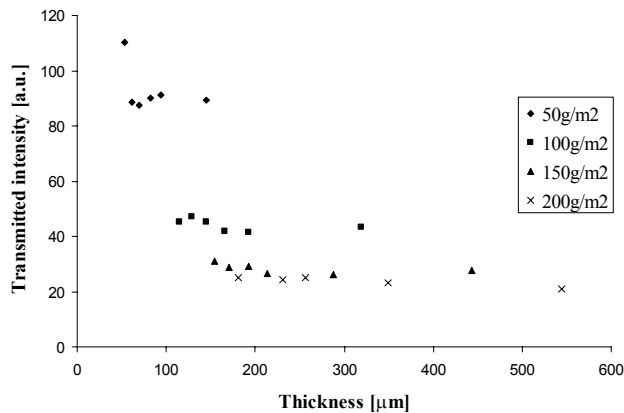
#### *4.1.1 Effect of basis weight and thickness changes of paper on photon migration*

Photon migration in a highly scattering medium during compression was evaluated by a streak-camera, using as test samples cellulose fiber based tissues with a grammage range of 50-200 g/m<sup>2</sup>. The samples were compressed and laser pulses were shot through them during pressing to evaluate the effect of light scattering on transmittance and propagation delay behaviour.

#### 4.1.1.1 Light transmission

The influence of grammage on transmission was investigated by measuring the pulse peak amplitude of light pulses that travelled through a paper sample. The grammage of the samples was between 50-200 g/m<sup>2</sup>, and their thicknesses were modified by compressing them between hardened glass plates. Fig. 5 displays the obtained results.

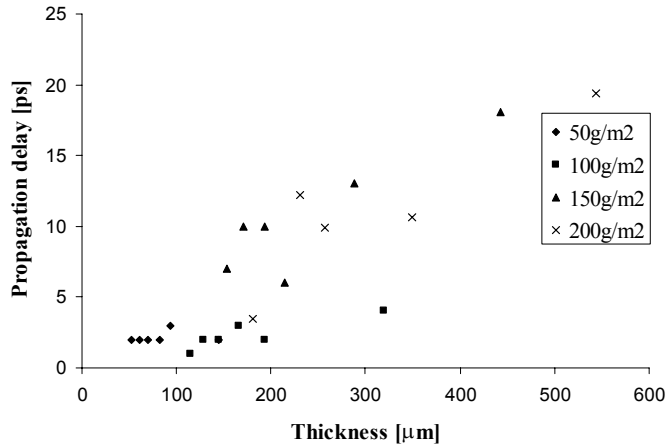
The results show that transmittance is independent of a sample's thickness. This indicates that the grammage of cellulose tissue can be obtained by measuring the intensity of light pulses passing through the it. Further, transmittance is inversely proportional to the sample's grammage, suggesting that it is inversely proportional to the number of scattering surfaces. The amount of photons passing through does not depend on the sample's thickness, which proves that the absorption coefficient of cellulose tissues is very low. In addition, the results suggest that transmittance variance increases as grammage increases. As a result, optical grammage determination based on transmitted light intensity measurements is limited to about 200 g/m<sup>2</sup>.



**Fig. 5. Pulse peak amplitudes of light pulses that have passed through a sample as a function of the sample's thickness. Light transmittance of cellulose fibre tissues is slightly dependent on their thickness.**

#### 4.1.1.2 Delay of transmitted photons

Measuring the pulse peak time of a light pulse that has passed through a sample and subtracting from it the pulse peak time measured without a sample, enables determining the average delay of the transmitted photons. Fig. 6 shows propagation behaviour as a function of thickness.



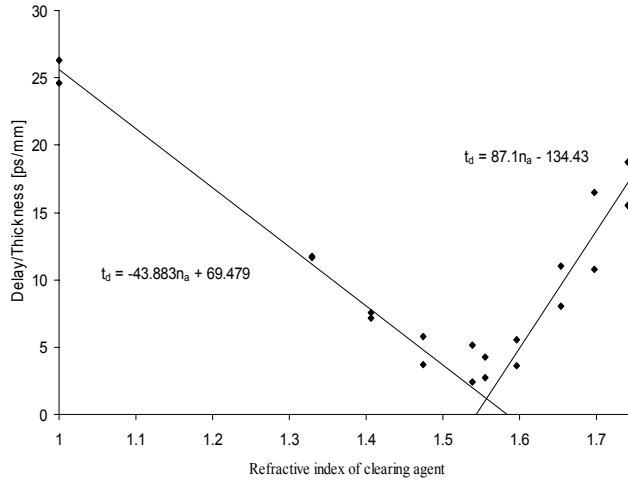
**Fig. 6. Propagation delay of photons that have passed through a sample as a function of the sample's thickness. The effects of thickness and grammage changes on propagation delay cannot be distinguished from each other.**

During compression, delay in the transmission of photons decreases. Since transmittance depends on the number of scattering surfaces and remains constant during compression, the delay decrement during compression is caused by the distance decrement of the scattering surfaces. The effects of grammage and sample thickness on propagation delay cannot be distinguished reliably.

#### ***4.1.2 Average refractive index of paper raw material***

An alternative method to measuring the average refractive index of paper is based on the following principle: The propagation delay of photons decreases when the refractive index mismatch between the raw material and the pores decreases. A minimum value for propagation delay is achieved when the refractive index of the paper and the clearing agent are equal.

In this study, the presented method was tested with a bleached, filler-free cellulose sheet. Figure 7 shows how propagation delay, obtained by a streak camera, changes as a function of liquid's refractive index.



**Fig. 7. Propagation delay of a laser pulse ( $\lambda=905$  nm) as a function of the liquids' refractive index. The delay was measured with a streak-camera.**

Assuming that a partially linear approximation can be used to estimate propagation delay ( $t_d$ ) in a paper board allows the average refractive index to be calculated. The propagation delay achieves its minimum value when the refractive index of the paper and the clearing agent are equal. According to the obtained measurement results, the refractive index of paper board is 1.56.

## 4.2 Optical coherence tomography measurements

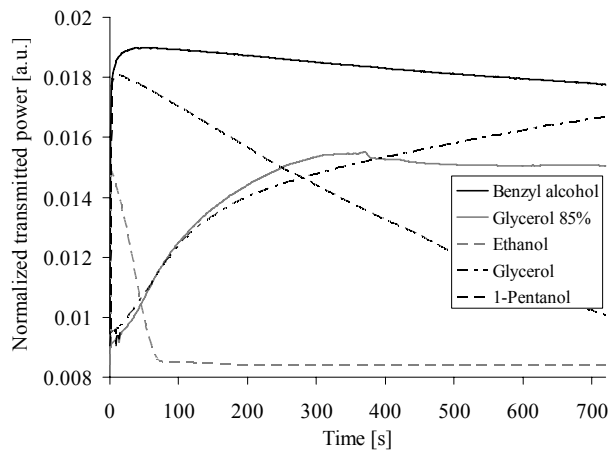
In the research reported in Supplements III-VI, optical coherence tomography (OCT) was used as measurement method. While Supplement III provides a general discussion on the effect of a refractive index matching liquid on the scattering properties of paper, it also explores its effects on the imaging depth of OCT. Swelling behaviour is investigated in Supplement IV, while Supplements V and VI focus on liquid sorption behaviour. All measurements were conducted on laboratory-manufactured cellulose sheets, except in Supplement III, which used copy paper. A more detailed description of the measurement setup and the used samples and refractive index matching liquids can be found in Supplements II-VI.

### 4.2.1 Effect of refractive index matching on light transmission

Time domain OCT imaging is a time-consuming process, because OCT images are constructed from point to point. Investigating the time-dependence of light transmittance

is necessary, because OCT measurements require a sufficient amount of a stable clearing agent. Moreover, for a successful measurement, the clearing agent's evaporation rate has to be relatively slow. Needless to say, processes that change the structure of paper are not desirable. One possible way of investigating the suitability of liquids to OCT measurements in paper is light transmission experiments.

To evaluate other important properties of a suitable refractive index matching liquid for optical measurements, Supplement III studied light transmittance as a function of time. To that end, liquids with a refractive index smaller than that of cellulose sheets ( $n_c \approx 1.55$ ) were tested and the results are collected in Figure 8.



**Fig. 8. Time-dependence of light transmittance. At zero time, the clearing agent was applied. The solid black line represents benzyl alcohol, the solid grey line 85% glycerol with water, the grey dashed line ethanol, the black dash-dot line pure glycerol and the black dashed line 1-pentanol.**

Light transmittance measurements on different refractive index matching liquids provided a wealth of information concerning the penetration of light into paper. Among the most important findings was that benzyl alcohol has the largest influence on transmission. Thus, light penetrates about 2 times better into paper wetted with benzyl alcohol than into dry paper. The maximum transmittance enhancement factor of benzyl alcohol was 2.01, while the corresponding value for 1-pentanol was 1.91, for glycerol (85%) 1.76 and for ethanol 1.77. A sample wetted with pure glycerol, however, failed to achieve its maximum transmittance enhancement factor during the 12-minute measurement.

These paper measurements further demonstrated that, in terms of stability, the most salient characteristics are liquid sorption and evaporation. The sorption process for pure glycerol is really slow, and the maximum transmittance value could not be achieved in 12 minutes. Nevertheless, when glycerol was diluted by water sorption, this velocity increased. Consequently, glycerol (85%) wetted paper achieved its maximum light

transmittance value in 6 minutes, which suggests that some of the applied refractive index matching liquid was sorpted. Transmittance measurements, combined with a visual examination of glycerol (85%) wetted paper, proved that certain structural changes take place in about 6 minutes from the beginning of the measurement. Finally, as shown by Figure 8., ethanol, 1-pentanol and benzyl alcohol all seem to wet pores and cavities at a similar velocity.

The figure also indicates that the transmittance of glycerol (85%) wetted paper does not change after 9 minutes, although some kind of saturation value can be observed in the case of pure glycerol. However, this saturation time is much longer than 12 minutes. Ethanol, on the other hand, evaporated from the sample in 3 minutes and 1-pentanol in 15 min. Light transmittance in paper wetted with benzyl alcohol decreased only by 8 % during 12 minutes, indicating that the evaporated volume of the refractive index matching liquid was very small. Linear extrapolation suggests that the evaporation of (3  $\mu$ l) benzyl alcohol takes over 1 hour and 30 minutes. In practise, the evaporation time is even longer than that.

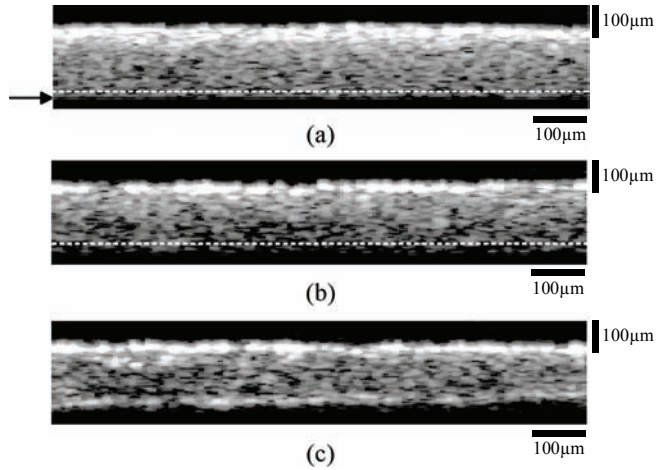
Some of the requirements for a suitable clearing agent depend on the used measurement system. It is evident from the light transmittance measurements that the most suitable clearing agent for OCT measurements is benzyl alcohol. The sample that was wetted with benzyl alcohol had a good light penetration depth and exhibited no unwanted changes in paper structure.

#### ***4.2.2 Imaging depth enhancement with clearing agents***

In dry paper, the imaging depth of OCT is not adequate and therefore the bottom surface of paper remains unclear. Probing depth can be improved by the use of clearing agents, however, because they serve to reduce light scattering. Figure 9 shows constructed cross-section profiles demonstrating this improvement in imaging depth in optically cleared copy paper.

As the constructed cross section profiles for dry and 1-pentanol wetted copy papers reveal, the imaging depth of OCT is inadequate, because the samples' rear borders cannot be detected. Due to limited scanning depth, the profiles for dry and 1-pentanol wetted samples seem cut. In reality, the intensity of the obtained OCT signals should fade away gradually. Although paper wetted with 1-pentanol has a smaller scattering coefficient than dry paper, the imaging depth of OCT is still insufficient.

Using benzyl alcohol as the refractive index matching liquid allowed both surfaces of the copy paper sample to be detected. The results show that the optical thickness of the benzyl alcohol wetted sample is about 160  $\mu$ m. Assuming that the average refractive index of wetted copy paper is  $\sim 1.55$ , the physical thickness is 103  $\mu$ m, which correlates well with the measured thickness. These results suggest that OCT can be used for the structural characterization of paper in all three dimensions, and that it enables studying dynamical processes such as paper wetting.



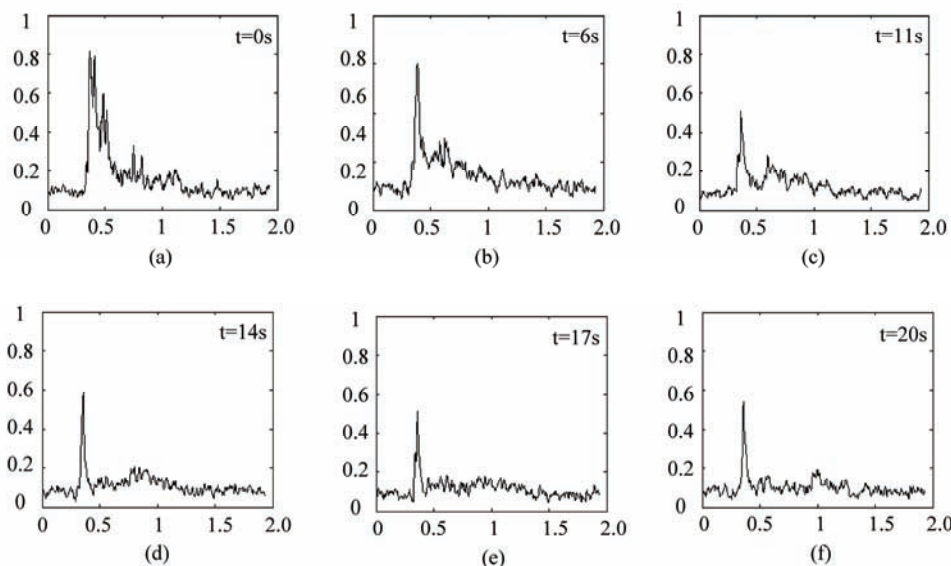
**Fig. 9. Constructed 2D images of the used paper samples: (a) dry paper, paper wetted with (b) 1-pentanol, (c) benzyl alcohol. In the lateral and depth direction, the pixel size of the images is  $15\ \mu\text{m}$  and  $0.39\ \mu\text{m}$ , respectively. The black arrow indicates the scanning depth limit of the used measurement system, while the white dashed line represents an estimation of the paper sheet's rear border.**

### ***4.2.3 Liquid penetration into paper***

The main ambition of this thesis was to develop a new optical method for measuring liquid penetration into paper. From the optical point of view, liquid penetration into dry paper is a dynamical process involving a change in refractive index matching. It is known that as refractive index matching improves, scattering decreases. In practical terms, this means that the scattering properties between dry and wetted areas of paper differ significantly.

#### ***4.2.3.1 Investigation of separate OCT A-scans***

Measuring separate OCT A-scans from the same location enables determining dynamical changes in scattering properties in the depth direction. The glycerol penetration process into paper was measured by OCT, and the separate A-scans obtained from the same spot at different time moments are shown in Figure 10. Of these, scan (a) marks the beginning of the liquid penetration phase, before the sample was actually wetted. In all scans (a)-(f), the first peak indicates the paper's front surface. In scans (b)-(e), the second peak is caused by the reflection coefficient difference between the dry and the wetted area. In scan (f), glycerol has already reached the sample's rear surface and the intensity peak of obtained OCT signal represents the sample-air interface.



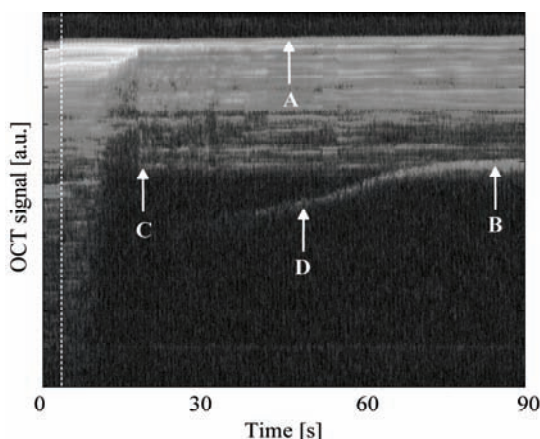
**Fig. 10. Separate A-scans at different time moments. The y-axis represents the normalized OCT signal power in arbitrary units, while the x-axis represents optical depth in millimetres. Scan (a) was measured at the initial stage of the liquid penetration process.**

The magnitude of first peak, caused by back scattering from the front surface of the paper sheet, is about 0.8 at the beginning of the wetting process, (a) and (b). When the liquid penetrates deeper into the paper, the normalized intensity peak of OCT signal decreases to approximately 0.5-0.6, (c)-(f). This observation suggests that, as the liquid is applied to the paper, some air filled gaps exist between the liquid and the fiber surface. Due to the high viscosity of glycerol, it takes over 6 s for these gaps to disappear.

When A-scans are analyzed separately, it is difficult to determine the border between the wetted and dry region, especially at the initial stage of penetration. When the liquid reaches deeper into the sample, scattering and absorption serve to decrease the obtained peak intensity OCT signal. Thus, the depth of the second peak, occurring at end of penetration (e), is also difficult to ascertain. A simpler way of studying liquid penetration involves constructing a two-dimensional image from separate A-scans.

#### 4.2.3.2 Backside OCT measurements

Measurement direction seems to have a significant effect on the measured back-scattered signal. To demonstrate this, paper wetting measurements were made both from the upside and backside. Figure 11 shows OCT measurements results obtained from the backside of a paper sheet. When a droplet of liquid is applied on the surface of the sheet, it descends a few microns and the amount of detected backscattered photons starts immediately to decrease (Supplements V and VI).



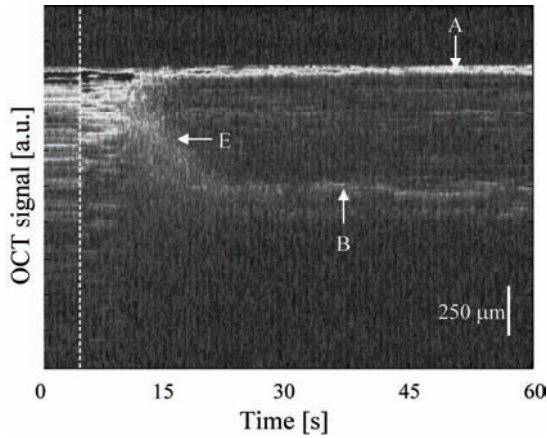
**Fig. 11. Repeated OCT A-scans as a function of time measured from the backside of a non-pressed sample. The dotted vertical line stands for the beginning of the wetting process. A is the paper's front surface, B is the rear surface and C the border between the dry and wetted area. D is the surface of the droplet.**

When the liquid penetrates into the sheet, the intensity of the obtained OCT signal first drops very rapidly. However, as the liquid penetrates deeper into the sample, the decrement becomes slower. After about 15 s from the beginning of the wetting process, the obtained signal changes dramatically indicating that liquid reaches the rear border of sample. This observation further affirms the fact that the penetration depth of single or slightly scattered photons into a cellulose sheet is very short. Consequently, the rear border of a sheet can only be detected, if the sample is totally wetted.

Making measurements from the backside of the sample allows dynamical changes in scattering properties to be detected during the penetration process. As a result, the beginning and end of the capillary penetration phase can be observed. However, the border between dry and wetted areas cannot be determined. To reduce the harmful effect of multiple scattering on the OCT signal in dry paper, these measurements have to be made from the upside of the sample.

#### 4.2.3.3 Upside OCT measurements

The liquid penetration process was investigated by measuring OCT A-scans recorded repeatedly from the same place during wetting. Figure 12 shows dynamical changes in OCT signals as a function of time. Similar to backside measurements, the obtained intensity of the backscattered signal starts to decrease immediately after the application of a liquid droplet. Due to multiple scattering from the totally wetted sample, some signals were obtained behind the paper's rear surface.

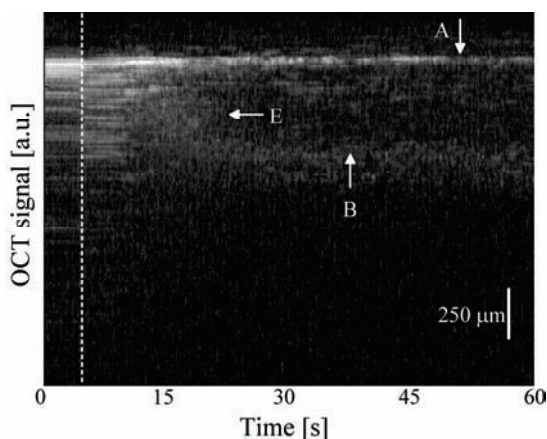


**Fig. 12.** Repeated OCT A-scans as a function of time measured from the upside of a non-pressed sample. The dotted vertical line stands for the beginning of the wetting process. A is the paper's front surface, B the rear surface and E the border between the dry and wetted area.

The results prove that the border between the dry and wetted area can be separated during liquid penetration. There seems to be some kind of delay before the capillary penetration phase starts. Although the border between the dry and wetted area moves almost linearly in the middle of the penetration process, penetration velocity decreases at the beginning and the end of the process. As seen, there is a 6 s delay before glycerol begins to penetrate into the sample, while the penetration process itself takes about 12 s.

As the border between the dry and wetted area can be discerned directly from the image, penetration velocity also can be determined. In these measurements, the average penetration velocity of glycerol was about 23 μm/s (Supplement VI). Further, the results show that penetration velocity decreases at the beginning and the end of the penetration process.

Supplement VI explores the effect of paper porosity on wetting behaviour, and the achieved results are shown in Figure 13. The glycerol penetration process was characterized for pressed samples and steps similar to those for the non-pressed samples were determined. As seen, the delay preceding capillary penetration is about 6 s and the capillary steps take 12 s. However, in pressed samples, the border between the dry and wetted area is harder to determine accurately than in non-pressed samples.



**Fig. 13.** Repeated OCT A-scans as a function of time measured from the upside of a pressed sample. The dotted vertical line stands for the beginning of wetting process. A is the paper's front surface, B the rear surface and E the border between the dry and wetted areas.

#### 4.2.4 Paper swelling

Paper wetting contains at least four coexistent processes, including liquid absorption and diffusion inside ground material. One of the most important phenomena related to the interaction between paper and liquid is swelling. When swelling occurs, internal stress breaks intermolecular bonds within fibres, thereby changing the paper's physical and chemical properties.

Supplement IV investigated the effect of swelling on the optical properties of paper using OCT, while Supplements V and VI explored swelling behaviour during the wetting process using the same method.

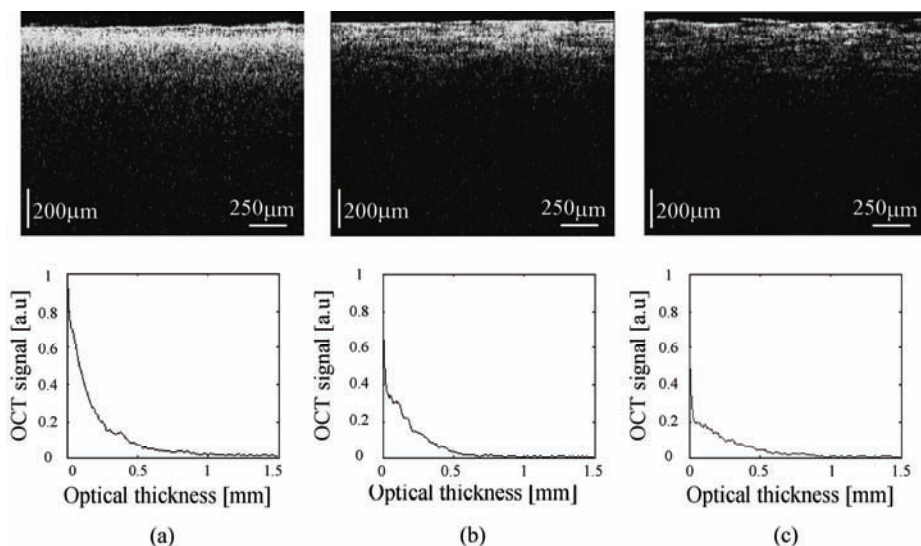
##### 4.2.4.1 Effect of swelling on the optical properties of paper

When two different liquids with an equal refractive index are used, changes in scattering properties can be detected by OCT. This idea was tested experimentally using isopropanol and a solution made up of 70% of water and 30% of glycerol. The isopropanol soaked sample was used as a reference, because isopropanol does not cause swelling.

Figure 14 shows that there is a difference in the measured heterodyne signal power between a sample wetted with isopropanol and a corresponding sample wetted with a mixture, although the refractive index of the two liquids is equal (Supplement IV). This difference proves that swelling does have an effect on light scattering in paper.

The slopes of OCT signals, calculated from four different measurements, were  $-2.56 \pm 0.13 \text{ mm}^{-1}$ ,  $0.79 \pm 0.17 \text{ mm}^{-1}$ ,  $-0.41 \pm 0.04 \text{ mm}^{-1}$  and - for dry, isopropanol and mixture wetted samples, respectively. Since the tail of the mixture-wetted sample is longer than

that of the isopropanol sample, it indicates that a swelled sample scatters more than a non-swelled sample when the optical depth exceeds 0.5 mm.



**Fig. 14. OCT images and averaged A-scans as a function of optical thickness: (a) dry, (b) isopropanol and (c) mixture. Scattering is much higher in the dry (a) than in the wetted samples (b) and (c). A decrease in the refractive index mismatch in the wetted samples decreases scattering. The difference between a non-swelled sample (b) and a swelled one can be distinguished (c).**

#### 4.2.4.2 Swelling during paper wetting

As reported in Supplements V and VI, the obtained measurement results suggest that also swelling behaviour can be detected during the paper wetting process. Glycerol that has penetrated into a paper sheet interacts with its cellulose fiber structure and changes its physical structure. In Figure 12, the detectable intensity peak of OCT signal has arisen at about 250 μm above the paper surface 50 s into the measurement. This intensity peak exists for about 5 s, before disappearing again. Due to the insufficient resolution of the used OCT system, pore size determination is irrelevant. Similar behaviour can be seen from the other positions as well. The cumulative effect of these separate additional gaps serves to increase the paper's physical thickness. Sufficient refractive index matching enables the sample's optical thickness to be determined. In our initial measurement, the sample's optical thickness was 610 μm, growing to 626 μm at the end of the measurement. Assuming that the average refractive index of glycerol wetted paper is about 1.50, the thickness increment in the non-pressed sample was about 11 μm.

The pressed sample interacted with glycerol in the same way as the non-pressed sample. In the case of the pressed sample, the first measurable thickness was about 400

$\mu\text{m}$ . At the end of measurement, the optical thickness was  $410 \mu\text{m}$ . By assuming that the average refractive index of a wetted sample is not affected by smaller porosity, the thickness increment can be calculated to be  $7 \mu\text{m}$ .

## 5 Discussion

Since the most important aim of this thesis was to develop an effective optical measurement device to measure the liquid penetration properties of paper, this perspective dominates the following discussion on results. This involves providing plausible explanations for the obtained results and discussing the limitations of the applied methods.

### 5.1 Streak-camera measurements

The results reported in the first part of this thesis were obtained by a streak-camera. This device was used to measure the average propagation delay and transmission behaviour of short light pulses migrating through paper. These results are relevant for understanding the relationship between paper properties and light migration into paper. Unfortunately, they are not promising for the study of the liquid penetration phenomenon itself.

Supplement I explored transmission and photon propagation delay behaviour in dry compressed paper. As the results show, transmitted light intensity ( $T$ ) is independent of a cellulose sheet's thickness. This means that the absorption coefficient ( $\mu_a$ ) of paper is very small, which correlates well with previously published results on models and experiments [35,36,37]. Further, the transmission of photons seems to be inversely proportional to the sample's grammage. However, the method is not relevant for measuring sheets with a high grammage [48,49].

Contrary to the conclusion drawn in a previous study [50], average photon delay ( $t_d$ ) seems to decrease systematically also in samples with a grammage less than  $280 \text{ g/m}^2$ . Due to the inadequate resolution of the used streak-camera (Supplement II), the effect of grammage changes on delay could not be distinguished from other results (Fig. 6). Nonetheless, time-of-flight (TOF) measurements have shown that the average propagation delay is linearly proportional to the sample's physical thickness ( $L$ ) and grammage ( $B$ ). Despite the fact that the measured propagation delay did not behave exactly as the equation suggests, there was a clear correlation.

If porosity is assumed to be the volume of pores or air and if transmission and propagation delay behave as described earlier, porosity can be calculated by the following equation:

$$\phi = 1 - \frac{B}{\rho_f L} = 1 - \frac{K_t^2 K_d}{\rho_f t_d T^2} \quad (2)$$

where  $\phi$  stands for porosity and  $\rho_f$  for fibre density ( $1500 \text{ kg/m}^3$  for pure cellulose).  $K_t$  and  $K_d$  are specific constants. The other terms have been determined above. This observation suggests that the porosity of a paper sample, one of the most important properties affecting liquid penetration into paper, can be measured optically by a streak-camera.

Supplement II focused on propagation delay and absorption behaviour in optically cleared paper. As mentioned in Chapter 3.3, the scattering coefficient of paper decreased significantly. Thus, the transmitted light intensity increased and the average propagation delay exhibited a significant decrease. It is easy to understand that, when a sample is optimally refractive index matched, transmission achieves a maximum value and propagation delay a minimum value. Hence, a sheet is optimally refractive index matched when the refractive index of the paper raw materials equals the refractive index of the liquid. What this means in practise is that the average refractive index of a porous and rough cellulose sheet can be determined. This was demonstrated by the fact that the obtained results showed good agreement with earlier experiments. Conventional measurement methods, such as reflectometry or ellipsometry, only allow determining the refractive index of the paper surface [51], while the streak-camera method also enables measuring the refractive index beneath the surface.

When the OCT method is used to investigate the liquid penetration properties of paper, the refractive index of the used liquid has a strong effect on measurement results. Supplement II demonstrated that, with the OCT method, a liquid with a refractive index of 1.56 gives the best results. A more detailed discussion on suitable liquids for paper investigation by OCT will be discussed later.

## 5.2 Optical coherence tomography measurements

### 5.2.1 Probing depth of OCT

Due to the highly scattering nature of paper, the imaging depth of OCT is limited. In the OCT method, information is derived mostly from single backscattered photons. Their amount, however, decreases as the imaging depth increases. As a result, the applicability of OCT is unsatisfactory in the depth direction. Thus, a typical imaging depth of OCT in highly scattering media is  $\sim 1 \text{ mm}$  [52]. However, using refractive index matching liquids offers a significant improvement in imaging depth. Supplement III concentrated on the effects of refractive index matching on the imaging depth of an OCT device in paper measurements. Probing depth is also an important factor affecting the applicability of OCT to liquid penetration measurements.

The five different organic matching liquids with a refractive index between 1.359-1.538 were used. It was assumed that imaging depth increases as transmittance increases. A comparison between streak-camera transmission measurement results obtained by a streak-camera (Fig. 5) and a photo diode (Fig. 8) shows a clear correlation in transmission behaviour in refractive index matched papers. Transmission does indeed increase as the refractive index difference between the paper raw components and a matching liquid is reduced. The obtained results also suggest that, as in medical applications, the imaging depth of OCT in paper can be improved [42]. In the case of dry paper, the applicability of OCT in depth direction measurements is maximally some tens of microns [53].

Shown in Figure 9, the measured cross-section profiles of copy paper demonstrate the practical effect of refractive index matching on the imaging depth of OCT. Although the obtained backscattered signal does not fade away as in reality, due to the limited scanning depth of the used OCT device, the results nonetheless show that a sufficiently good probing depth can be achieved in copy paper if it is wetted with benzyl alcohol. Because the physical thickness of the copy paper was about 102  $\mu\text{m}$ , probing depth is smaller than this in glycerol-wetted copy paper, if the effect of swelling is assumed to be negligible. On the other hand, OCT measurement made in pure cellulose sheets that were refractive index matched with glycerol prove that the imaging depth of OCT is higher than 440  $\mu\text{m}$  (Fig. 12). This observation indicates that, in the OCT method, the structure and raw components of paper strongly influence imaging depth.

Figure 9 also affirms that the imaging depth of OCT is much smaller in dry than in wetted paper. This observation is important for liquid penetration measurements. Due to the very poor imaging depth of OCT in dry paper, the border between the dry and wetted area during the liquid penetration process can be discerned only if the measurements are made from the front surface.

## ***5.2.2 Applicability of OCT to paper sorption measurements***

### *5.2.2.1 Sorption as a physical process*

Actual dynamical measurements on paper wetting were conducted during the research leading to Supplements V and VI. As far as the present author is aware, this is the first time that the borderline between wetted and dry area has been distinguished during paper wetting. Consequently, this observation is absolutely the most important contribution of this thesis. In addition, the ability to distinguish between three coexisting subprocesses which take place during wetting makes penetration measurements particularly noteworthy.

Shown in Figure 12, the obtained results suggest that, during the wetting process, there are systematic variations in the magnitude of the first intensity peak of OCT signal. After a certain time period elapses, the intensity peak of OCT signal decreases. The likely explanation for this phenomenon is that, at the beginning of wetting, there are small, air-filled gaps between the paper surface and the droplet. When those gaps are filled with the

liquid, the surface becomes refractive index matched, reducing the magnitude of the first intensity peak of OCT signal. This demonstrates that the method allows distinguishing liquid filling of pores and pits from other subprocesses.

According to the basic theory, the subprocesses are coexistent. However, the present OCT measurement results indicate that the other subprocesses do not start before the pores and pits of paper surface are filled with liquid [26]. Consequently, there seems to be a contradiction between the theory and the measurement results. This contradiction can be explained by the fact that the effective measurement area in these measurements was very small due to the collimated probe beam. In other locations, however, liquid penetration into paper may well have started. As to penetration velocity, it seems to be slower at the beginning and end of the penetration process.

This liquid penetration delay for polar liquids has been observed earlier by several other researchers [14]. However, the actual mechanism has never been elucidated unequivocally. The present measurement results indicate that the duration of the delay is relative to the time it takes the liquid to fill the pores and pits of the paper surface. This result correlates well with previously published contact angle measurements [20]. A more detailed investigation of this subprocess would be needed to clarify the background of the wetting delay phenomenon.

The fact that the borderline between the wetted and dry area can be detected means that the also the second subprocess, known as capillary penetration, can be distinguished. The results correlate well with previously made paper wetting measurements. Moreover, they are in good agreement with the simple, conventionally used Lucas-Washburn model, except for the beginning of the wetting process [54].

As the liquid penetrates into paper, it interacts with the paper sheet's raw components affecting its physical and mechanical properties. One of the most important phenomena is swelling. When swelling occurs, intermolecular bonds within fibres are broken by internal stress [55,56]. As a result, the paper's physical thickness increases and its optical properties change. The effect of swelling on the optical properties of paper is investigated in Supplement IV.

Among the most significant findings is the fact that swelling has a strong effect on the optical properties of paper. Because the slope of the OCT signal's linear part in a swelled sample (Supplement IV) was smaller than in a non-pressed sample, swelling appears to reduce the scattering coefficient. One likely explanation for this observation is that swelling enlarges the distance between scattering surfaces.

When liquid penetration is evaluated as a dynamical process, also the dynamics of swelling can be investigated. In a wetted sample, additional intensity peaks, caused by a deformation in paper structure, can be observed in the OCT signal during penetration. Internal stress breaks bonds within fibres, generating additional air filled gaps [55]. After a short delay, the intensity peaks of OCT signal disappear, as the gaps are filled with liquid. Steady state swelling measurements suggest that the phenomenon is dominated by increasing distances among the scattering particles. On the other hand, dynamical wetting measurements show that additional scattering surfaces arise. For a better understanding of swelling behaviour in paper, we need further research.

Yet another coexistent subprocess during wetting is absorption and diffusion inside ground material. Since this phenomenon causes swelling, its effects are detectable by the

method described here. A particularly useful property of OCT measurements is that it enables us to study time-dependent swelling locally.

### 5.2.2.2 *Industrial viewpoint*

As mentioned in the introduction, liquid sorption behaviour plays an important role in paper manufacturing processes, particularly as regards the usability of paper products. This chapter discusses the contribution of this work from the industrial point of view.

One prominent end-use property of paper is resistance to penetration by such liquids as water, ink and oils. This penetration is known as degree of sizing, size resistance or size test. Paper products intended for printing, wrapping, bags and insulating boards are sized, whereas papers designed for absorbing liquids readily, such as towels and blotters, are unsized. Sizing degree is measured by liquid penetration measurements such as the Cobb test or the contact angle method [57,58].

Determining liquid penetration properties from contact angle data is in many cases not a straightforward undertaking and the results may be misleading [19]. OCT, however, has the advantage that it enables measuring sorption behaviour as a 2D or even a 3D process. This means that, for the first time, liquid penetration in the depth direction and dynamic contact angle measurements can be performed concurrently. Due to limited resources, a detailed investigation was not carried out during this thesis. Given the established state of conventionally used sizing test methods in the industry, the OCT method seems most suitable for laboratory research.

In many industrial processes, it is necessary to coat a porous substrate with a thin film of coating material to achieve the required end-use quality. In the paper industry, coating is widely used to enhance the appearance and printability of paper. In addition, coating also gives paper the required quality for further applications. Applied on a paper surface, a typical coating pasta contains water, pigments, adhesive and processing aids. While coating the paper's external surface, the pasta also penetrates into its pore structure. To produce good quality coating, it is particularly important that the coating fluid penetrates somewhat into the porous structure of ground paper. But if the fluid penetrates too deeply, it may cause severe problems for the coating process and have an adverse effect on the quality of the final product [9,59].

As a result, fluid penetration into the pore structure of paper must be measured during coating. The measurement results obtained with copy paper (Supplement III) show that the probing depth of OCT is limited. Due to the highly scattering nature of coating pastas, problems might arise, if fluid penetration into ground paper is measured by OCT. Solving these problems requires experimental work. In the paper industry, there is an incessant requirement to enhance the production efficiency of factories. One approach to raising efficiency involves online measurements, which are particularly important for the coating process. Unfortunately, there is no experimental evidence as yet of the applicability of the OCT method to online measurements during paper manufacturing.

During the printing of paper, an ink mixture comes into contact with the paper surface, usually with the coating layer. If the solution fails to penetrate the surface rapidly enough, the ink may inhibit the transportation of ink from other printing nips. On the other hand,

too fast ink penetration reduces cost efficiency, because a larger amount of ink will be needed. Thus, ink absorption by the substrate is an important consideration in almost all types of printing and covers aspects such as ink setting, trapping, print gloss and print density [11,60,61].

Despite the fact that commonly used printing inks are suspensions, contrary to the liquids used in this thesis, this work's most important contribution centres on evaluating phenomena related to printing. The fact that OCT can be used to investigate the liquid sorption behaviour of papers as spatially and temporally dependent phenomena offers new avenues for exploring ink penetration [61,60].

The OCT method seems more suited for laboratory applications than for online testing. Although this thesis focused on liquid sorption measurements in the paper industry, the sorption characteristics of porous scattering materials play an important role in many other fields as well, including the food, pharmaceutical and textile industries. It is quite obvious that the method is also applicable to these areas.

### ***5.2.3 Limitations of OCT***

The obtained OCT measurement results are very promising for investigating the paper wetting process. Unfortunately, the results also show that the OCT method suffers from certain limitations. This chapter discusses these limitations and offers some suggestions on how to overcome them.

A conventional OCT system has a resolution of about 10-15  $\mu\text{m}$ . In a paper sheet, the dimensions of the smallest raw components fall below that resolution limit [13]. As a result, determining the width of the gaps that appear during paper swelling was not a reasonable expectation. Nonetheless, the development of OCT has produced devices with a resolution better than 1  $\mu\text{m}$  in the depth direction [62,63]. This resolution should be good enough to enable a detailed study of swelling behaviour in paper.

Another observed limitation of the OCT method was insufficient probing depth. The results presented in Supplement III prove that, in copy paper wetted with glycerol, the probing depth is less than 102  $\mu\text{m}$ , which prohibits effective liquid penetration measurements. In addition, limited probing depth constrains penetration measurements to the front face of the paper sheet.

It is well known that OCT's probing depth can be improved by refractive index matching. Industrial liquid sorption applications require evaluating water penetration properties within paper. Since the refractive index of water is 1.33, the OCT method does not avail itself to penetration measurements in normal copy paper.

To circumvent this problem, the wetting measurements conducted during this study were made using glycerol. However, owing to its very high viscosity, glycerol penetrates into paper at a relatively slow rate, thereby imposing restrictions on liquid sorption measurements in pure cellulose sheets. The limited scanning rate of the used OCT device disallowed fast liquid penetration measurements such as 3D measurements of droplet behaviour in paper structure.

Another way of avoiding the scanning rate limitation involves using more sophisticated OCT systems, such as full-field optical coherence tomography (FF-OCT) or

Fourier-domain optical coherence tomography (FD-OCT). In FF-OCT, a CCD camera is used as a detector array and a 3D OCT image can be achieved by single axial scanning [64]. In this way, the achievable imaging rate can be improved significantly compared to the imaging rate of the TD-OCT system used in this thesis. In the FD-OCT method, one full depth profile is recorded in parallel with a spectral analysis of the backscattered light [65]. The method enables an imaging rate in excess of video imaging rate [66], thereby allowing the detection of even very fast liquid penetration processes.

All the measurements described in this work were performed in a non-controlled atmosphere. Since the wetting process depends on circumstances, it will inevitably introduce a degree of non-systematic variation into the results. This should be taken into account as the results are compared with those obtained in different circumstances.

## 6 Conclusion

The main ambition of this thesis was to develop an optical method for measuring liquid penetration into paper in all three directions. Underlying the optical approach was the idea that the scattering properties of wetted and dry paper differ strongly, and optical measurement methods enable the detection of dynamical changes in scattering properties.

Two different optical devices, streak-camera and optical coherence tomography, were used. Results from the streak-camera measurements clarified the relationship between paper properties and light migration within a paper sheet, while the contribution of the OCT measurement results centred on liquid sorption measurements.

Chief among the practical results of this thesis are the following:

1. The delay before liquid penetration begins is caused by the fact that the pores and pits of paper surface must be filled with liquid before penetration into paper can start.
2. The border between the wetted and dry area in the depth direction of paper can be investigated during the paper wetting process. Moreover, the liquid penetration velocity seems to be slower at the beginning and end of the penetration process.
3. The effect of swelling on the optical properties of paper can be measured independently. The scattering coefficient of a swelled sample decreases.
4. Swelling behaviour can be measured as a dynamical process concurrently with the liquid penetration measurement. The place and moment of structural changes caused by swelling can be determined.
5. The effect of three different coexistent subprocesses related to paper wetting can be detected. Only the effect of liquid migration along fibres could not be distinguished by OCT.

Of the used method's limitations, the most important were insufficient resolution, probing depth and depth scanning rate. However, restrictions related to resolution and scanning rate can be resolved by the use of more sophisticated OCT devices. Using a liquid whose refractive index was about 1.55 resulted in the attainment of an adequate probing depth.

The obtained OCT measurement results are very promising from viewpoint of developing an effective paper wetting measurement device for industrial applications. Even if this thesis focused on paper wetting, it is reasonable to assert that the presented

ideas and obtained results have more general value in terms of explaining liquid penetration into porous structures and offer an alternative method of evaluating that process.

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