Matthias Trimmel, Dipl.-Ing.

A novel laboratory device for simultaneous measurement of retention, dewatering and flocculation under industry-oriented conditions

DOCTORAL THESIS

to achieve the university degree of

Doktor der technischen Wissenschaften

submitted to

Graz University of Technology

Supervisor

Univ.-Prof. Dipl.-Ing. Dr.techn. Wolfgang Bauer

Institute of Paper-, Pulp- and Fibre Technology

Graz, July 2015

AFFIDAVIT

I declare that I have authored this thesis independently, that I have not used other than the declared sources/resources, and that I have explicitly indicated all material which has been quoted either literally or by content from the sources used. The text document uploaded to TUGRAZonline is identical to the present doctoral thesis.

Date

Signature

Abstract

Retention, dewatering and fibre flocculation are three important parameters influencing the sheet forming process at a papermachine. On the one hand each of these parameters affects the forming process by its own. On the other hand retention, dewatering and flocculation are highly interrelated. A change of one of these parameters will have an effect on the other two. In order to achieve a better understanding about these three parameters and especially their interrelations, a novel laboratory device for simultaneous measurement of retention, dewatering and flocculation under industrial oriented conditions was developed. The laboratory device consists of a small pulp chest, a centrifugal pump and several meters of polypropylene tubes. The furnish circulates at headbox consistency through a closed loop. For investigations of the influence of chemical additives three points of dosage and two dosing units are available. The simultaneous measurement of retention, dewatering and fibre flocculation is carried out using a flocculation unit and a dewatering unit. The flocculation unit consists of a transparent rectangular observation channel, a high speed camera and a light source. After image acquisition of the flowing suspension the images are evaluated using a FFT structure analysis. The dewatering unit, used for the measurement of retention and drainage behaviour, consists of a headbox and a dewatering device. At the vacuum supported dewatering zone of the dewatering device, the furnish which is coming from the headbox is dewatered. The filtrate is collected in special tanks and used to evaluate the parameters retention and dewatering. The measurement via the novel device is highly reproducible and sensitive enough to determine change in the range occurring at industrial machines.

Keywords: chemical additives, dewatering, flocculation, laboratory device, retention, simultaneous measurement

Kurzfassung

Der Blattbildungsprozess in einer Papiermaschine wird durch drei miteinander in Wechselwirkung stehenden Parametern - Retention, Entwässerung und Formation - beeinflusst. Diese drei Parameter stehen in Wechselwirkung zueinander wodurch eine Änderung eines einzelnen Parameters einerseits den Blattbildungsprozess, andererseits jedoch auch die Beiden anderen Parameter beeinflusst. Um die Einflüsse auf den Blattbildungsprozess wie auch die Wechselwirkung zueinander besser verstehen zu können wurde eine Anlage im Labormaßstab entwickelt welche unter industrienahen Bedingungen eine simultane Messung dieser drei Parameter ermöglicht. Diese Laboranlage besteht aus einer kleinen Stoffbütte, einer Förderpumpe sowie einem Rohrleitungssystem in welchem die zu messende Faserstoffsuspension im Kreislauf geführt wird. Um den Einfluss chemischer Additive auf Retention, Entwässerung und Flockung untersuchen zu können sind drei Dosierstellen und zwei Dosiereinheiten in den Kreislauf integriert. Die Messung der Faserstoffsuspension erfolgt mit Hilfe einer Flockungseinheit und einer Entwässerungseinheit. Die Flockungseinheit besteht aus einem transparenten, rechteckigem Kanal, einer Hochgeschwindigkeitskamera sowie einer Lichtquelle. Die Auswertung der durch Aufnahme des strömenden Faserstoffes gewonnen Bilder erfolgt im Anschluss mit Hilfe einer FFT- Strukturanalyse. Die Entwässerungseinheit wird durch einen Stoffauflauf sowie durch einen als Langsieb konstruierten Former gebildet. Die Faserstoffsuspension wird nach Verlassen des Stoffauflaufes durch ein dynamisches Sieb entlang einer mit Vakuum unterstützen Entwässerungszone entwässert. Das dabei entstehende Filtrat wird in speziellen Behältern aufgefangen und dient anschließend zur Bestimmung von Retention und Entwässerung. Die Messungen mit dieser Laboranlage weisen eine hohe Reproduzierbarkeit auf und sind sensibel genug um Änderungen die auch auf industriellen Maschinen relevant sind zu detektieren.

Schlagwörter: Chemische Additive, Entwässerung, Faserflockung, Retention, Laboranlage, simultane Messung

Acknowledgements

I wish to thank Professor Bauer, as well as Eckhart Rene for their support, helpful advices and the possibility to write this thesis at the Institute for Paper, Pulp and Fiber Technology.

I want to thank my coworkers at the Institute, who contributed a lot to this thesis and who made my PhD time so enjoyable, working with them has been a great pleasure.

Very special thanks to Christian far all the special accessories he prepared in the mechanic´s workshop. Many, many thanks for the support of the laboratory assistants Manu, Kerstin, Karin, Irmi, Heidi and Hari! Also special thanks to Claudia and Kerstin for heer assistance with administrative issues.

I am greatful for financial support of Andritz Pulp & Paper, Sappi - Gratkorn GmbH, Heinzelpaper - Laakirchen, Mayr Melnhof - Frohnleiten, Smurfit-Kappa - Nettingsdorf, BASF and the Austrian Research Promotion Agency Ltd. (FFG). This thesis would not been possible without their contribution.

I want to thank my family for their support during my whole educational career. This thesis is dedicated to them.

Matthias Trimmel
Graz, 02.07.2015

Contents

List of Figures

List of Tables

Chapter 1

Introduction

1.1 Motivation

During the web forming process on paper machines three interrelated parameters are of great interest to the papermaker. These parameters are called retention, dewatering and formation/fiber flocculation. To increase the quality as well as the quantity of the produced paper, retention, dewatering and formation/flocculation have to be optimized. The constraint of the optimization is the interrelation between these three parameters, a change of one influences the others. Because of these interrelations it is difficult to find the best settings for each paper grade as well as for each paper machine to achieve maximum quality and quantity.
Various chemical additives with different chemical and physical properties are available to support in this task. The diversity of products requires preliminary investigations to find the best solution for each paper grade on each paper machine under cost-efficient conditions. These investigations are performed under standardized conditions on the laboratory scale.
Several researchers and companies have developed different laboratory devices for measuring these parameters using different operating conditions to acquire more information on their interrelation as well as the effects of used chemical additives. Some of these devices are mentioned and described in section 3. The disadvantage of the major part of these laboratory devices is the restriction in measuring only one of the three parameters. To gain some information on the interrelation several devices working under different conditions are necessary making comparisons difficult.
The main goal of this work is the development of a laboratory scale device which enables a simultaneous measurement of retention, dewatering and flocculation under industry-oriented conditions. The possibility of simultaneous measurement facilitates

investigations on the interrelation between retention, dewatering and flocculation under the influence of chemical additives.

1.2 Scope of the thesis

The scope of the thesis was the development of a laboratory device for simultaneous measurement of retention, dewatering and flocculation under industry-oriented conditions. An additional important issue was the requirement to accomplish an easy and fast measurement procedure. Therefore the scale of the device was limited to a sample volume of 40 litres fibre suspension for each trial.
For the measurement of flocculation the basic elements of a previously developed laboratory setup, consisting of an transparent observation channel, a high speed camera and a light panel, was used.
To enable the measurement of retention and dewatering a dewatering unit resembling a fourdrinier former had to be designed which was the main part of the work.
For a simultaneous measurement the elements of the flocculation setup as well as the dewatering unit has to be integrated in a novel device including a circulation loop.
In this thesis the design and operation of the novel device are discussed.

1.3 Outline of the thesis

As mentioned above the main goal of the thesis was the design and development of a small scale laboratory device for simultaneous measurement of retention, dewatering and flocculation.
In chapter 2 the main influencing parameters on retention, dewatering and flocculation are discussed in more detail.
For the measurement of retention and dewatering of fibrous suspensions several devices, presented in section 3.1, are available. Some of these devices designed for the laboratory scale are using small sample volumes and provide an easy and fast measurement. These devices are on the one hand the Dynamic Drainage Jar, discussed in section 3.1.1, on the other hand there are several modifications available, discussed in section 3.1.2. For the measurement close to conditions prevailing in an industrial plant several larger scale devices were developed. These devices are discussed in section 3.1.3. Devices used for the measurement of flocculation are discussed in section 3.2.
In chapter 4 a novel device for the simultaneous measurement of the three main influencing parameters is presented. The developed device consists of two main parts, a flocculation unit as well as a dewatering unit. The main parts of the novel device are discussed in section 4.2.1, the flocculation unit and the corresponding components are presented in section 4.3.1. In section 4.4 the dewatering unit is discussed in more

detail. In general this device consists of a headbox (see section 4.4.1 and a drainage setup (see section 4.4.3).
The measurement procedure, the evaluation of the reproducibility as well as a validation against a commercial laboratory device are discussed in chapter 5. In chapter 6 applications with different representative stock compositions using the novel device are presented and discussed.
Finally in chapter 7 a summary of the main important facts is given.

Chapter 2

Main Parameters influencing sheet formation

In this chapter the main parameters retention, dewatering and flocculation and their influence on sheet formation are discussed in more detail.

2.1 Retention and major influencing parameters

In this section the parameter "retention", especially the influence of retention on the web forming process, will be described in more detail.

Generally in paper making retention is defined as the ability of fibrous suspensions to retain small components such as fines, fillers and colloidal ingredients in the forming section of a paper machine. On the one hand fibres, fines and fillers show anionic charge and therefore repel each other. On the other hand the particle size of fillers, fines and colloidal components is too small to be retained mechanically on the wire. Due to these reasons the small anionic charged ingredients are carried out in the forming process by the water flow during drainage. To improve retention these components need to be bound to the larger fibres which is achieved by using wet end chemicals called retention aids.

A further advantage of increased retention is a reduced rate of charge accumulation in the white water caused by unretained solids. This may lead to the formation of deposits which may cause problems in the white water treatment, may initiate the formation of holes in the paper web to the point of a break in production.
Depending on the used retention system different mechanisms act to retain small components in the furnish on the wire.

2.1.1 Basic mechanism of retention

In general retention aids improve this wet end parameter by aggregation mechanisms [Norell et al., 1999], however the aggregation of particles with the same electric charge occurs by flocculation which directly influences the other two parameters of interest(see section 2.3). In the literature the basic mechanism is classified depending on the size of formed flocs [Unbehend, 1992]. There is the so called "wild flocculation" or "macro flocculation" describing the clumping of fibres. This kind of flocculation leads to basis weight variations which are visible to the eye. Macro flocculation affects the formation noticeably. Flocculation in the micro scale can be classified as "micro flocculation" and occurs between small particles such as fillers. Due to the small scale of these flocs a change in formation is not perceived but optical properties as well as porosity are affected. The ideal retention mechanism would be a combination between macro- and micro flocculation called "hetero flocculation". In this case the flocculation between fibres is limited and flocculation of fines and additives onto the fibres is maximized. Hetero flocculation would give the best retention and a sheet with good formation as well as optical and printing properties.

2.1.2 Mechanism of retention

Retention aids can be grouped depending on their retention mechanism [Norell et al., 1999]. This classification includes

- charge neutralisation
- patch model (charge mosaic model)
- bridging
- complex flocculation
- network flocculation

In this section the listed retention mechanism are described in more detail.

Charge neutralisation

In general particles of the same electric charge repel each other. By charge neutralisation the surface charge of particles becomes neutralized, consequently attractive forces dominate and the furnish components flocculate. Retention aids used for charge neutralisation are generally low molecular weight compounds with a high cationic charge. Examples for these are polyamines, poly-DADMAC or polyethyleneimine [Eklund and Lindström, 1991]. Furthermore polyvalent cations as polyaluminum species also affects charge neutralisation [Norell et al., 1999].

Patch model

In general furnish components such as fibres, fillers or fines have an anionic charged surface. The target of patch flocculation is to form cationic "patches" onto the surface of negatively charged components [Kasper, 1971] . These patches cause attraction of oppositely charged particles. To form such patches, illustrated in Figure 2.1, cationic polymers of high charge density and low molecular weight in a flat conformation are deployed. Flocs formed by this mechanism are compact and small. Patch flocculation in comparison to charge neutralisation and bridging forms flocs which are more shear resistant.

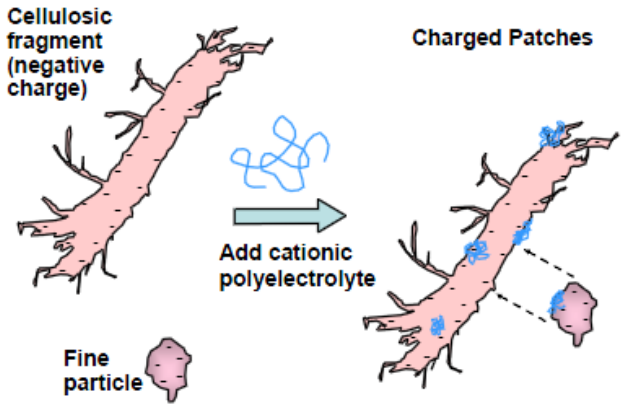

Figure 2.1 The patch mechanism on the surface of a fibre. By adding a cationic polymer of high charge density and low molecular weight patches are formed on the fibre surface. These cationic patches attract oppositely charged particles [Hubbe et al., 2009].

Bridging

The bridging mechanism takes effect when cationic polymers of high molecular weight are used. The polymers adsorb to particle surfaces in the furnish and form tails and loops acting like bridges between particles [Svedberg, 2012].
For this mechanism two parameters of the polymer are important, the anchorage of the cationic polymer on the particle surface as well as the design of the tail and the loops. These parameters are controlled and influenced by several factors such as the charge density of the polymer [Rasteiro et al., 2008], the contact time, the charge balance between polymers and particles and other properties of the suspension like the electrolyte content. The flocs formed by bridging, shown in Figure 2.2, are relatively strong and voluminous but at high shear forces the flocs will break and the polymer degrades. After breaking, reflocculation by bridging will not occur but rather through the patch mechanism.

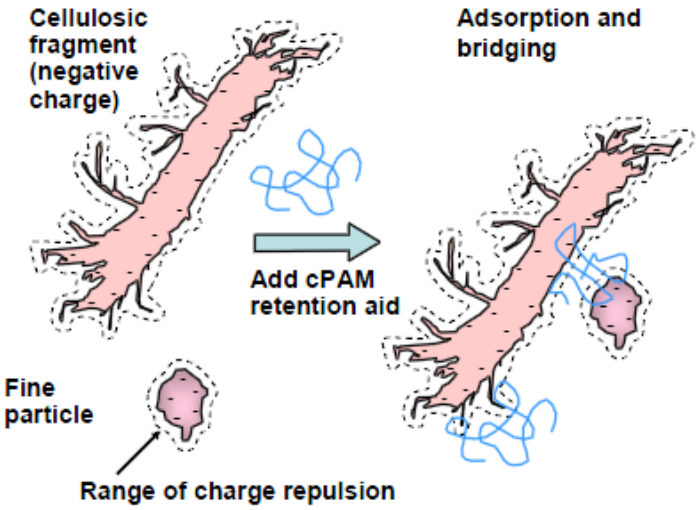

Figure 2.2 The bridging mechanism onto the surface of a fibre. By adding a high mass cationic polymer with low charge density bridging occurs on the fibre surface. The cationic bridges catch oppositely charged particles [Hubbe et al., 2009].

Complex flocculation

Retention caused by complex flocculation can be divided into different types [Norell et al., 1999].

- dual polymer flocculation
- nano and micro particle flocculation
- network flocculation
- site blocking enhanced bridging flocculation

Dual polymer flocculation This mechanism of retention mostly uses a cationic polymer of high charge density and low molecular mass followed by an anionic polymer of high molecular weight and lower charge density[Hubbe, 2001].
In the first step of the dual polymer system flocs are formed by patch flocculation, induced by the cationic polymers. After flocculation of anionic fibres and fillers a shear stage redisperses the flocs. In the last step of this mechanism the anionic polymer is added and reflocculation by bridging occurs. The dual polymer flocculation is shown in Figure 2.3. In some cases of dual polymer flocculation two cationic polymers can be used.

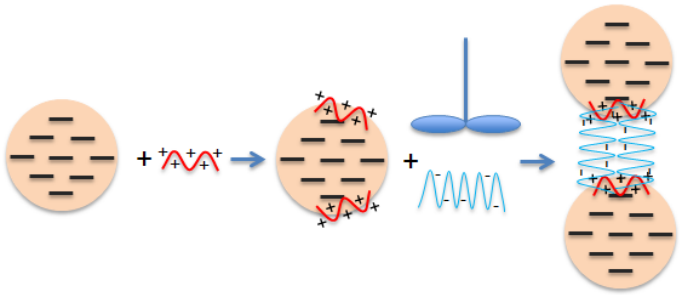

Figure 2.3 Dual polymer flocculation. The two steps of adding polymers and their influence on flocculation. In the first step a cationic polymer with high charge density is added and patch flocculation occurs. Afterwards the high molecular weight, anionic polymer is added evoking reflocculation by bridging after a shear stage [Svedberg, 2012].

Nano and micro particle flocculation To achieve nano and micro particle flocculation a combination of a cationic polymer like starch or polyacrylamide and an anionic micro particle like colloidal silica, alumina or montmorillonite is used [Norell et al., 1999; Swerin and Ödberg, 1996]. By adding the cationic polymer in the first step flocs are formed by bridging. In the presence of high shear rates the formed flocs are destroyed. For reflocculation after the shear stage the anionic nano or microparticle is added into the furnish. By this mechanism very small and dense flocs are produced. Furthermore these flocs are highly shear resistant and show a high degree of reflocculation. Therefore this system is often used for paper machines equipped with gap formers.
There is also the possibility to exchange the anionic particles of the microparticle system by an anionic polymer. In this case a combination of a nanoparticle and a dual polymer mechanism occurs [Kahl, 1997].

Network flocculation Other than in the earlier mentioned flocculation mechanisms network flocculation occurs primarily by hydrogen bonding interactions. Typically for this mechanism is a cationic polyelectrolyte with an anionic component, e.g. phenolic resin and polyethylene oxide or montmorillonite and anionic polyacrylamide [Norell et al., 1999]. In network flocculation diluted transient three dimensional networks are formed. These transient networks trap dispersed materials like fillers and fines by an occlusion process [Lindström, 1984], the mechanism is shown in Figure 2.4.

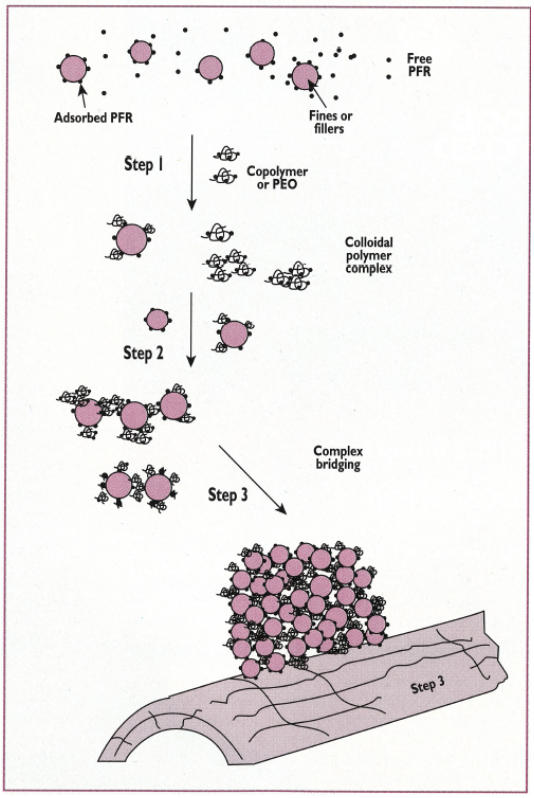

Figure 2.4 The functional principle of network flocculation. In the shown system a phenolic formaldehyde resin (PFR) and a polyethylene oxide (PEO) is used. This system is similar to the dual polymer flocculation and occurs for dispersed components like fillers and fines [Xiao et al., 1996]

.

Site blocking enhanced bridging flocculation For this kind of retention site blocking agents (SBA) are used. By using SBA bridging is improved because of a more favourable conformation. Site blocking agents reduce the surface coverage and lead to reduced collision efficiency factors [Norell et al., 1999]. The polymers used as SBA have low molecular weight and a high charge density. Site blocking mechanism is used additionally in dual polymer and microparticle flocculation systems.

Retention behaviour in the forming section

On the one hand retention and the different mechanism are influenced to a great extent using chemical additives as discussed above. On the other hand because of high shear induced in the forming section retention is affected, too.
The intensity of shear depends on different machine settings as wire speed, elements for drainage and/or the level of vacuum. The higher the wire speed of a former the higher the shear caused by the dewatering elements (register rolls, foils etc.) is.
Due to high shear the size of formed flocs may be reduced, thereby the formation of

the building up fibre web increases, on the other hand retention may decrease. Not retained small particles occur a higher pollution of the white water and may reduce the drainage capacity due to depositions on the wire.

2.2 Dewatering and major influencing parameters

In the production of paper, water is the carrier of all major paper components. A typical furnish in the headbox of a paper machine has a consistency between 0,5-1,5%, that means about 60 up to 200 metric tons of water are necessary to produce one metric ton of paper. This huge amount of water has to be removed as fast as possible to improve the productivity of a paper machine [Norell et al., 1999].

2.2.1 Water removal in the former section

On a paper machine water is removed at different stages beginning at the forming section, followed by the press- and drying section. Dewatering affected by retention and flocculation occurs mainly in the former section.
In general the dewatering process in the forming section can be divided into two zones. The first dewatering stage is the so called drainage zone where water is removed freely by means of gravity in combination with static drainage elements such as foils. This zone starts at the line where the stock jet coming from the headbox impinges on the wire and ends before the first suction box [Norell et al., 1999]. In the drainage zone the consistency of the formed fibre web increases to approximately 8 percent. This zone is important because the largest quantity of water is removed, nearly 94% of the total fluid.
In the second zone the drainage process is supported by vacuum due to a reduction of the free flow of water during drainage, this zone starts at the first suction box and ends when the sheet leaves the suction couch roll.

Enhancing the amount of water removed in the forming section influences several conditions of production and thereby the productivity of a machine [Allen and Yaraskavitch, 1991].

- better wet web strength
- increased refining without loss in production
- use of slower-draining fibres
- reduced press load to achieve equal dry content

2.2.2 Water removal mechanisms on the wire

While the water of the fibre suspension is drained trough the wire two important processes of sheet forming occur:

- Filtration
- Thickening

Filtration

For this mechanism of sheet forming a sharp transition between the already formed fibre web and the fibrous suspension above occurs. The consistency of the liquid phase is approximately constant and included fibres can move more or less freely. The filtration mechanism is also illustrated in Figure 2.5a.

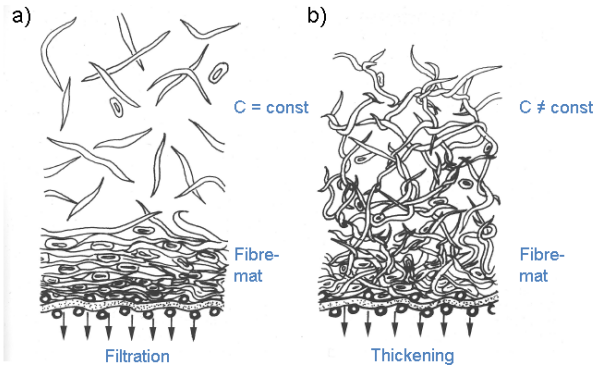

Figure 2.5 The difference between filtration and thickening. a) The filtration mechanism. There is a sharp division between the formed fibre mat and the suspension.
b) The thickening mechanism. In comparison to filtration there is no division, the concentration of the built up fibre mat decreases continuously [Bos et al., 2006].

Thickening

For this mechanism no sharp transition can be detected. The consistency of the built up fibre web decreases continuously starting from the wire. Therefore the fibres are not able to move freely and the remaining water is drained constantly through the forming web. The thickening mechanism is shown in Figure 2.5b.

Both mechanisms, filtration and thickening occur at the same time while the fibre web is formed. Investigations on several paper grades have shown the fact that filtration is the dominant of these two mechanisms [Norell et al., 1999].
In modern paper machines a third mechanism called turbulent thickening occurs. Turbulent thickening means a gradual formation and disruption of the sheet. This leads to a combination of thickening and filtration mechanisms.

2.2.3 Factors affecting drainage

In general the water removal from a forming fibre web along a paper machine should be as uniform as possible, without dead zones as well as sudden high-volume dewatering [Smook, 1989].

Along a paper machine drainage is influenced by several different factors. These are [Norell et al., 1999]:

- moisture content of the stock
- degree of refining
- stock composition
- temperature of the stock
- air included in the stock
- surfactants
- colloidal material and fines
- pH-value of the stock
- polymers and flocculation

The temperature of the stock affects the viscosity. If the temperature of the stock increases, the viscosity will decreases and thus support drainage.
Air, fillers, fines and colloidal components included in the stock influence drainage by blocking available pores.
Yet another important factor is flocculation. Flocculation on the one hand improves dewatering by collecting fillers, fines and colloidal material onto the fibre surface, thereby increasing the free area for water removal. On the other hand, too strong flocculation can decrease drainage because of voluminous flocs which are more difficult to drain.
The influence of polymers on drainage is also very important and will be discussed next.

Influence of polymers on drainage

As discussed in section 2.1 several polymers are used in paper making to improve and control retention and thereby flocculation. Furthermore polymers also affect the drainage performance of the fibrous suspension during the forming process. Depending on the chemistry of the used polymers (charge density, molecular weight) different mechanisms of retention, as mentioned in section 2.1.2, are responsible for

retention of the small anionic charged particles. According to the occurring mechanism the characteristics of formed flocs differ, in other words the size, density as well as the shear resistance of flocs are influenced. Depending on the prevailing floc characteristics the amount and magnitude of interstices in the formed web, necessary for the dewatering process is influenced, therefore the drainage behaviour is affected to a great extent. Allen and Yaraskavitch [1991] gave a summary of the influence of polymers on the drainage performance. In this review several polymers were investigated and the results are mentioned below:

- Single polymer systems of high charge density and low molecular weight as well as dual polymer systems including this component are able to improve free drainage considerably but may downgrade vacuum supported dewatering.
- Microparticle systems normally improve dewatering
- Polymers of high molecular weight and low charge density have less influence on dewatering depending on the furnish.

Norell et al. [1999] mentioned the fact that high molecular weight polymers are able to downgrade dewatering performance through orifices by absorbing energy from the fluid.

2.3 Flocculation and major influencing parameters

A fibre suspension consists of several components like fibres with a given length and width distribution, fines, fillers varying in size and shape and different colloidal components. Also added in fibre suspensions are wet-end chemicals necessary to optimize retention of fines and fillers onto the fibre surface.
Flocculation is an accompanying symptom caused by the reaction of wet-end chemicals like retention aids which act between fibres and other anionic particles. In most cases flocculation and the associated basis weight variations influence the quality of the produced paper. To achieve adequate strength properties as well as optical properties a homogeneous fibre distribution in the forming process is desirable.

On the other hand to retain suspension particles in the forming web and to protect them against washing-out due to drainage agglomerates and flocs are formed. There are two different mechanisms in flocculation, the mechanical flocculation and the chemical flocculation. These two mechanisms are discussed in more detail.

2.3.1 Mechanism of mechanical flocculation

The formation of flocs depends on the concentration of the fibrous suspension. Below a so called critical concentration which could be represented by the concentration of

sedimentation the fibres are allowed for a free rotary movement. As soon as the critical value is reached the free movement of fibres is restricted due to contact of fibres at one or more points. Because of the linkage the fibres are bound to the extent that they can transmit external forces and a three-dimensional network (floc) is formed. The cohesive properties of ordinary pulp fibre networks was shown and measured first by Mason [1954]. The formation of fibre networks by mechanical interaction between fibres depends on several factors

- consistency of the suspension
- fibre length
- fibre coarseness and fibre flexibility
- amount of fibrillation
- fibre curls and kinks

The forces acting between the contact points responsible for mechanical interactions are described by Kerekes et al. [1985]. The mechanical strength of fibre networks depends on mechanical surface linkage, elastic fibre bending as well as surface tension.
Mechanical surface linkage means hooking forces caused by mechanical entanglement due to fibre curls and kinks. Elastic bending is caused by frictional resistance due to normal forces at contact points. Surface tension may have an effect if undissolved gas (normally air) is trapped within the network.

Some of the afore-mentioned parameters like consistency, fibre length and coarseness influence the number of contact points in formation of fibrous networks in a given suspension to a great extent. Kerekes combined three parameters and brought them together in the so called Crowding factor N [Kerekes and Schell, 1992]:

$$N = \frac{2}{3} * C_v * (\frac{L}{d})^2$$

where

$$\begin{aligned} C_v &= \text{volumetric concentration} \\ L &= \text{fibre length} \\ d &= \text{fibre diameter} \end{aligned} \tag{2.1}$$

This definition of the Crowding factor provides informations on the number of fibres present in a spheric volume developed due to the rotation of a single fibre.

Especially for pulp fibre suspension the Crowding factor can also be described as [Kerekes and Schell, 1992]:

$$N = \frac{5 * C_m * L^2}{\omega}$$

where

$$\begin{aligned} C_m &= \text{mass concentration expressed in percentage} \\ L &= \text{length of fibres} \\ \omega &= \text{fibre coarseness} \end{aligned} \tag{2.2}$$

Above the critical value of the Crowding number fibres in suspension show the tendency to build networks. The intensity of fibre flocculation in a given suspension can be described by an equation defined by Wahren [1967]:

$$Flocculation\ intensity = \frac{\sigma_{C_v}}{\bar{C}}$$

In this equation σ_{C_v} represents the standard deviation of a local fibre concentration measured in the volume v. The magnitude of the volume should be chosen similar to the smallest floc. The smaller the chosen volume the higher the standard deviation will be. The average fibre concentration in the stock is expressed by $\bar{C}$. Similar to the flocculation intensity the formation of a produced paper can be expressed as shown [Wahren, 1967]:

$$Formation = \frac{\sigma_{W_A}}{\bar{W}}$$

In this equation the local basis weight from a large number of areas is described as standard deviation σ_{W_A}. $\bar{W}$ is defined as the average basis weight.
This equation in comparison to the equation of flocculation shows the correlation between flocculation and formation. Flocs can be described as a network consisting of fibres; flocculation intensity describes the amount of flocs prevailing in a defined suspension volume. Formation means the variation of the local basis weight in a formed fibre web which depends on the local fibre concentration. Variations of the fibre concentration in a formed web are caused by flocs formed in the fibrous suspension before and during the drainage process in the former section.
Because of turbulences and shear rates present on a paper machine flocs are not permanent. A constant breakdown of some flocs takes place while on the other hand new flocs are being formed. Due to this fact the overall number of flocs will remain constant, however, in a local view the number may change.

2.3.2 Mechanism of chemical flocculation

As described in the previous section 2.3.1, mechanical flocculation occurs between fibres and is influenced by various parameters. In suspensions used for paper making a couple of other components like fines, fillers and colloidal components are included. Due to the fact of high shear rates in the former section of a paper machine fillers and fines may not be retained in the formed fibre web. By using chemical additives, described in section 2.1, the particles are bond by different mechanism onto the surface of the fibres. Furthermore depending on the mechanism of retention as well as on the used additives flocculation is influenced to a certain degree.
The fixation of small particles caused by chemical additives, despite the presence of high shear rates, is achieved by very large electrostatic and electrokinetic forces [Kerekes et al., 1985]. These cohesive forces responsible for the adhesion of fines and fillers onto the fibre surface depend on the chemical properties of the additives like their structure, charge density as well as their molecular weight.
In some cases chemical additives called formation aids are added to reduce macro-flocculation and consequently to improve formation. Kerekes et al. [1985] mentioned two mechanisms affecting macro-flocculation.

- lowering the coefficient of friction between fibres
- changing the rheology of the suspending water

In summary flocculation depends on different mechanical and chemical mechanisms. The mechanical part of flocculation caused by surface linkage and friction forces between fibres mainly depends on the consistency of the used stock. In general these flocs are formed between fibres and do not affect fillers and fines. To retain small particles onto the uniform charged fibre surface chemical mechanisms provided by chemical additives are necessary. In general flocculation is a side effect mainly caused by retention of particles onto the fibre surface induced by chemical additives. The size, density and stability of flocs vary depending on the used chemistry but first of all the number of flocs increases whereby the formation of the produced paper is influenced to a great extent.

Chapter 3

Laboratory Measurement of Retention, Dewatering and Formation/Flocculation

The ability to measure the three important sheet forming parameters retention, dewatering and flocculation/formation is of great significance to the papermaker. Researchers in the past developed various laboratory and pilot scale devices to be able to measure these parameters. In this section some of these devices shall be presented and discussed.

In principle a conventional handsheet apparatus (like Rapid Köthen) is applicable for the measurement of retention under the influence of chemical additives. Furthermore the produced handsheets can be investigated after drying them.
However the sheets form highly diluted suspensions are not comparable to the industrial sheet forming process and flows as well as turbulences during handsheet forming are much gentler than those present on a real paper machine. Therefore the handsheet apparatus is not able to deliver meaningful results when it comes to retention [Hubbe, 2003]. For special applications like for the measurement of mechanical retention, modified handsheet formers can achieve meaningful results [Athley et al., 2012].

3.1 The Measurement of Retention and Dewatering

In this section different laboratory devices as well as modifications used for the measurement of retention and/or dewatering behaviour are discussed, setups resembling a former section up to the pilot scale are presented at the end of this chapter.

3.1.1 The Dynamic Drainage Jar - DDJ

Britt [1973] set a milestone in the history of testing retention in the lab scale. The Dynamic Drainage Jar, also called Britt Jar, fulfils two important tasks necessary for the evaluation of retention. The DDJ provides adjustable and reproducible agitation using an impeller to create turbulences in the system during dewatering. Because of agitation the results are closer to reality due to the hydrodynamic forces, which separate particles attached to the fibres by for example chemical additives. The variation of the impeller speed enables the input of different shear rates and thereby the possibility of different testing conditions [Pelton et al., 1979].

As shown in Figure 3.1 the DDJ consists of a transparent cylindrical jar where the fibre suspension at headbox consistency is filled in. At the bottom of the jar a stainless steel screen is positioned. This thin screen has conical holes with a diameter in the range of $76\mu m$. To achieve hydrodynamic forces during the tests an impeller is positioned above the screen. The distance of the impeller to the screen as well as the agitation speed are adjustable and need to be kept constant from test to test to provide reproducible and comparable results.
Some models of the Britt Jar are fitted with three small baffles placed on the inner wall of the cylindrical jar. By these baffles the rotating movement of the suspension due to agitation is disturbed and thus higher impeller speeds are possible. The outlet of the DDJ is closed by a stopcock or pinch clamp and has to be opened manually to start or stop the drainage sequence.

The Dynamic Drainage Jar is nowadays one of the standard laboratory devices for measuring retention as well as for screening of retention aids (Tappi T 261).

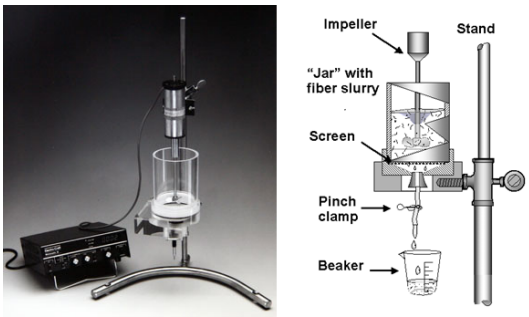

Figure 3.1 The Dynamic Drainage Jar was the first laboratory device which enables the measurement of retention under the influence of variable turbulences. A rotating impeller prevents the formation of a fibre mat during the test and thereby the evaluation of retention of fine particles onto fibres is possible. The DDJ consists of a cylindrical jar, a stainless steel screen on the bottom with a hole diameter of $76\mu m$, an impeller to produce turbulences and a pinch clamp for starting and stopping drainage of the suspension [Hubbe, 2003].

3.1.2 Modifications of the Dynamic Drainage Jar

As described in the section before the Dynamic Drainage Jar - DDJ - is a widespread method for the measurement of retention. Despite the applicability of this device several researchers worked on modifications and upgrades for this system to deliver results even closer to what happens in industrial forming processes.

Dynamic Drainage Analyser - DDA

The dynamic drainage analyser was originally developed to evaluate drainage behaviour of fibre suspensions [Roschy et al., 2002; Wirth et al., 1999]. The DDA, shown in Figure 3.1.2a represents a modification of the earlier presented Dynamic Drainage Jar - DDJ. The suspension for analysis is kept in a cylindrical jar where an impeller is positioned. On the bottom of the jar an exchangeable screen separates the jar from the outlet. Beneath the screen a pneumatic piston allows efficient and easy drainage control. The vacuum unit supports dewatering and acts under the screen after opening the drainage outlet. At the end of each measurement the built up fibre mat can be analysed after couching and drying. The vacuum level during drainage is recorded by means of a pressure sensor and plotted in a diagram over time. The result of the measurement is a manometric drainage diagram which can be divided into three sections, shown in Figure 3.1.2b.

- Section A: In the first step of the measuring procedure the pneumatic piston opens the orifice for drainage. Thereby the vacuum decreases rapidly. In this section drainage starts and a fibre mat is formed.
- Section B: Due to building up and compacting of the fibre mat the vacuum increases as long as there is sufficient underwatered fibre suspension present above the forming fibre mat. The maximum at the end of section B represents the magnitude of dewatering.
- Section C: After reaching the "dry-line" representative for the maximum vacuum level the curve declines rapidly. The reason being that no more suspension covers the web on the wire and air is sucked through the formed fibre mat. The vacuum finally reaches a constant level which indicates the air permeability of the fibre mat.

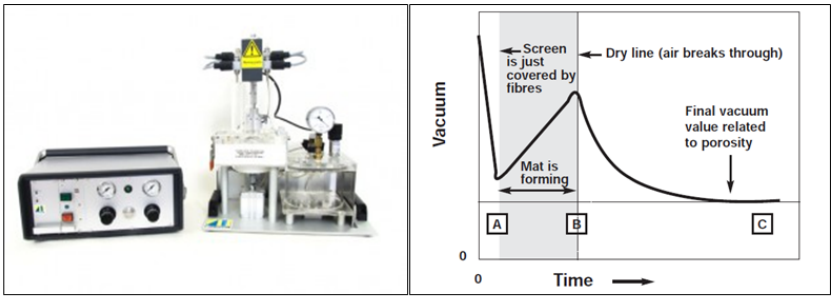

Figure 3.2 The Dynamic Drainage Analyser as well as the result. a) The measuring device. In principle this equipment is a modification of the earlier developed DDJ as presented in 3.1. It consists of a cylindrical jar where the sample is collected, dosing devices for chemicals, an impeller for agitation, a changeable screen, a filtrate chamber and a vacuum device to support drainage, the vacuum level is detected by a transmitter [Wirth et al., 1999].
b) The resulting diagram. On the ordinate axis the detected vacuum level is shown, on the abscissae the measuring time is plotted. The graph represents the manometric drainage curve which can be divided into three sections [Hubbe, 2003].

The measurement procedure of the DDA is controlled automatically by a computer. Before starting a measurement sequence the measurement settings like impeller speed, agitation time, vacuum level as well as dosing time for additives are selected by the user.
The measurement starts by agitation of the suspension sample as well as dosing additives at a set point of time. When the drainage process is started by movement of the pneumatic piston, agitation is stopped and vacuum supported dewatering during formation of the fibre mat occurs. The measurement ends as described above, when air is sucked through the fibre mat at a constant level of vacuum.

Retention and Drainage Tester - RDT

A further modification of the Dynamic Drainage Jar is the so called Retention and Drainage Tester - RDT - developed by Abson et al. [1980] and illustrated in Figure 3.3a. The dewatering principle of this laboratory device is similar to the DDA presented before. The RDT allows the measurement of both dewatering and the simultaneous measurement of retention based on two modes of testing.
In the first mode the measurement of dewatering is performed without agitation and vacuum support. The measurement itself depends on the increase of air pressure in the filtrate chamber caused by the entry of filtrate recorded by a pressure transmitter during drainage.
In the second mode, retention and dewatering are measured simultaneously, the dewatering procedure is supported by vacuum and the suspension is stirred by an im-

peller. Similar to the DDA the measured pressure values are plotted in a diagram over time.

G/W - Retention Tester

The G/W- Retention Tester developed by Gess [1984], [Trepanier, 1992] is used to investigate retention and drainage behaviour under the influence of vacuum. The measuring principle is similar to the dynamic drainage analyser presented above. During the measurement of a suspension at headbox consistency the vacuum level during dewatering is detected and plotted in a diagram divisible into different areas of drainage.

The uniqueness of the G/W- Tester is the sample preparation before the proper measurement starts. A stainless steel fluid vessel is used to segregate a defined volume of sample from the storage tank to the sample port. To eliminate operator errors as well as to achieve measurements in a repeatable manner the whole process is controlled automatically by a computer system.

Dynamic Drainage Device - DDD

Another modification is the Dynamic Drainage Device developed by Chabot and Daneault [2004], shown in Figure 3.3b. The design is similar to the Dynamic Drainage Jar but in addition drainage is supported by vacuum. The vacuum is detected by a transmitter located beneath the filtration screen, the pressure values are plotted in a diagram over the dewatering time. The resulting graph is similar to that of the DDA.

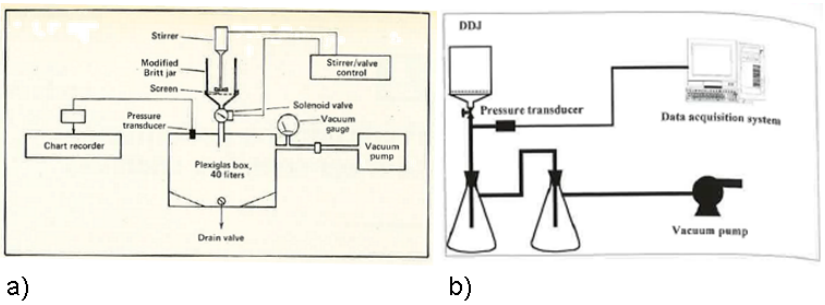

Figure 3.3 a) The Retention and Drainage Tester. The principle of measurement is similar the DDA. By means of two independent modes of testing drainage without agitation and vacuum support as well as dewatering and retention under the influence of vacuum and agitation are possible [Abson et al., 1980].
b) The Dynamic Drainage Device is shown. This device consists of the standard DDJ equipment as described in 3.1 extended with a vacuum device to support dewatering [Chabot and Daneault, 2004].

Drainage-Freeness-Retention Tester DFR

The Drainage-Freeness-Retention Tester DFR developed by BTG-Muetek [BTG-Instruments, 2010] also represents a modification of the earlier developed DDJ.
In general the DFR consists of a cylindrical jar holding the suspension with a consistency of about 1%. The exchangeable screen at the bottom of the jar is protected by a cone-shaped device including a conical impeller used for agitation of the suspension before drainage. To break up the rotary movement during agitation three baffles are installed. Beneath the cone-shaped device a second impeller prevents formation of a fibre mat during drainage when retention is evaluated.
As soon as the drainage procedure starts the conical device is lifted and the suspension accesses both the screen and the second impeller. Beneath the screen a shuttle valve separates the filtrate into pre-filtrate and main-filtrate. The filtrate chamber is positioned on a scale used to control the dewatering process, the filtrate weight is plotted in a diagram over the time. For automated dosage of chemical additives a dosing equipment with three feeders is placed above the cylindrical jar.
The whole measurement procedure is performed automatically and can be adjusted according to parameters chosen beforehand. These settings are:

- agitation time and speed of the two impellers
- dosing time for chemical additives
- starting point and duration of drainage
- filtrate quantity (measured by the scale) that marks the end of drainage

The DFR- Tester enables three modes of measurement, the measurement of retention, dewatering and freeness that have to be chosen before the measurement. For the measurement of retention the DFR- Tester allows the measurement of the turbidity during drainage based on a light transmission sensor. Without this optional equipment retention has to be evaluated separately.
In principle the measurement of retention relies on the principles already used by Britt (TAPPI T 261), the measurement of dewatering is based on formation of a fibre mat and is similar to the Schopper-Riegler measurement (DIN EN ISO 5267/1).

Dynamic Filtration System - DFS An older laboratory device also developed by BTG-Mütek is the so called DFS. The design of the DFS is similar to the DFR and allows the measurement of retention and dewatering via two independent modes of operation. One mode is used for the measurement of dewatering similar to the standard Schopper-Riegler Test (DIN EN ISO 5267/1). The second mode enables the measurement of retention which is based on the standard defined by Britt (TAPPI T 261).

Figure 3.4 The DFR- Tester developed by BTG Instruments enables an independent measurement of retention, dewatering and freeness. The measurement is performed without support of vacuum and with or without agitation. A scale is used to control the drainage process and to detect filtrate quantity over time. The collected data is plotted in a diagram [BTG-Instruments, 2010].

Pulsed Drainage Device - PDD

This laboratory device presented by Sutman [2000] was primarily developed for the measurement of dewatering and retention of newsprint and packaging applications. In some situations the commercial laboratory devices like the DDJ are not the ideal solution because of excessive filtration during formation.
The pulsed drainage device shown in Figure 3.5 consists of a mixing chamber were the sample is stored before measurement. When the measurement starts the suspension is fed through a valve into the test chamber with a screen on the bottom. Below the screen a rotating hydrofoil is installed to generate rapid pressure pulses during drainage. The drainage process is supported by vacuum. To collect data for process control there are two pressure transducers installed in the filtration chamber. One pressure transducer is used to measure the static head on the collected filtrate and the other one is necessary to collect data of the level of vacuum in the filtrate chamber during dewatering. The collected data is evaluated by a computer system and plotted in a diagram.
In summary the PDD simulates dewatering under the influence of vacuum as well as pulsations generated by the rotating foil.

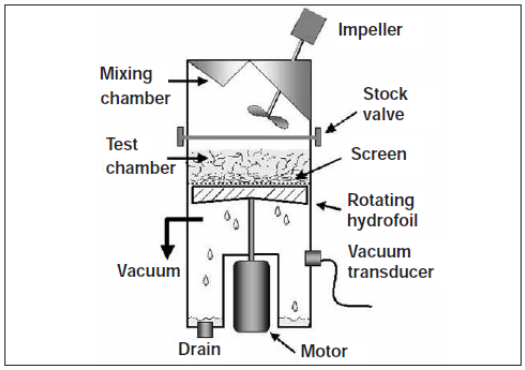

Figure 3.5 The Pulsed Drainage Device is primarily developed to allow an accurate measurement of newsprint and packaging pulps. The rotating hydrofoil beneath the wire prevents the formation of a fibre mat. By means of two pressure transducers the drainage process can be controlled and data for evaluation of the dewatering behaviour is collected [Sutman, 2000].

The Ultrasonic Dynamic Drainage Tester - UDDJ

This modification of the standard Britt Jar was developed by Ju et al. [2011] to simulate the dynamic drainage conditions prevailing in the forming section under more realistic conditions.
In principle this device consists of a drainage jar, a filtrate chamber, a vacuum pump, a scale and a computer. At the bottom of the drainage jar there is a stainless steel wire of 60 mesh and an ultrasonic horn is connected laterally to the screen frame (Figure 3.6). Ultrasound energy is evenly spread over the entire screen during the measurement. Three additional effects occur during drainage. Turbulences are created which reduce colloidal flocculation, acoustic forces on the forming mat open drainage channels as well as unblock the screen itself.
The measurement of drainage in conjunction with vacuum and turbulences induced by the impeller as well as the ultrasound effects should allow more realistic conditions during drainage.

Modified Hercules Dynamic Drainage Tester

The modification of the Hercules Dynamic Drainage Tester established by Davison [1989] is used to simulate dewatering conditions more similar to industrial conditions prevailing in the forming section of a paper machine. In general this device is an enhancement of the earlier developed Dynamic Drainage Jar.
The modification of the testing equipment includes an upgrade of the filtrate chamber by a vacuum-pressure pulsation device. By means of the pulsation the conditions prevailing in the forming section are simulated more realistically. After drainage values for retention and dewatering behaviour are evaluated by gravimetric methods.

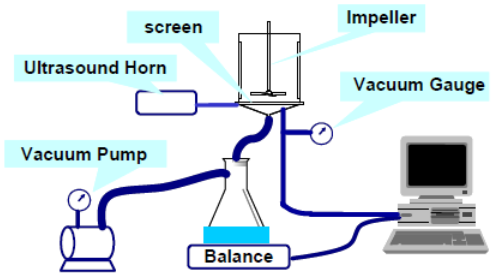

Figure 3.6 This measuring device represents a modification of the Dynamic Drainage Jar. The principle constituents of this device are the drainage chamber equipped with an impeller, the filtration chamber, the balance and the vacuum pump. The special equipment of this device is an ultrasonic horn connected to the wire frame. The ultrasound energy produces additional effects like turbulences and acoustic forces influencing drainage [Ju et al., 2011].

In summary the devices presented in this section show several advantages for the measurement of retention and dewatering. These advantages include a compact design, so that they can be used in standard laboratories, the sample volumes necessary for the measurement are quite low and the testing requires little time. As discussed in this section the Dynamic Drainage Jar developed by Britt [1973] was the first device especially used for the evaluation of retention and dewatering behaviour of fibrous suspensions. In subsequent years several modifications of the DDJ where developed to better simulate industrial conditions prevailing in the forming sections of real paper machines. These improvements includes several approaches to simulate vacuum supported dewatering as well as alternative methods to introduce pulsation.
In some cases these devices are not suitable enough. For example the standard measurement of dewatering using Shopper-Riegler (DIN EN ISO 5267/1) occurs without the presence of turbulences. Furthermore the measurement conditions of the above presented devices (drainage through a static wire, turbulences induced using an impeller) are often not comparable to industrial one and thereby the evaluated effects are not comparable, too. In the following section various devices operating near industrial conditions are discussed in more detail.

3.1.3 Laboratory Devices

In addition to the Britt Jar based laboratory devices presented in section 3.1.1 and 3.1.2 there are a number of other developments used for the measurement of retention and dewatering. These devices differ widely from the measuring principle of the DDJ and try to simulate reality as closely as possible.

Moving Belt Former - MBF

The Moving Belt Former was developed to simulate conditions prevailing on a fourdrinier former of a paper machine more realistically than the earlier developed laboratory drainage devices do [Karrilla et al., 1992; Räisänen et al., 1993, 1995].
The moving belt former consists of a mixing chamber similar to the Britt Jar on top of the device. To prevent sedimentation of the suspension before measuring as well as to generate turbulences during testing an impeller with exchangeable stirrers is installed. The consistency of the stock can be adjusted to achieve a grammage representative for the investigated paper grade. The automated addition of chemical additives is also possible.
At the bottom of the mixing chamber a fixed commercial paper machine wire is fixed (also see Figure 3.7. Beneath the wire a moving perforated and cogged belt generates pulsations due to the scraping effect on the wire during the movement. By means of a vacuum box installed beneath the moving belt the MBF allows vacuum supported dewatering. The speed of the moving belt, the vacuum level, the wire as well as the agitation speed of the impeller can be varied to simulate machine conditions as closely as possible. In comparison to some other drainage testers the measurement of retention behaviour is not based on the white water but on the produced handsheet. After couching, pressing and drying retention, formation as well as other paper properties can be evaluated from the produced sample having a size of $190x190mm$ [Strengell et al., 2004].

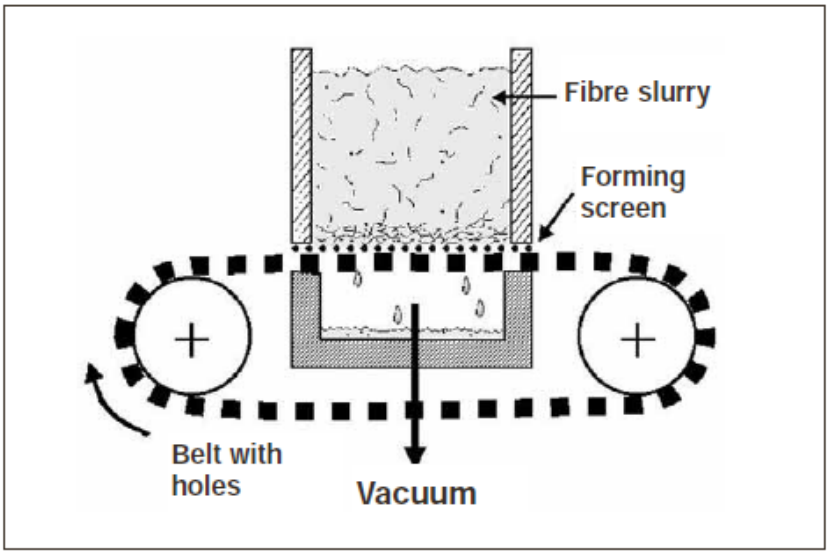

Figure 3.7 The Moving Belt Former. The mixing chamber is similar to the Britt Jar including an impeller as well as dosage equipment. Beneath a commercial wire a moving perforated and cogged belt as well as a vacuum box support dewatering [Hubbe, 2003].

Turbulent Pulse Sheet Former

The Turbulent Pulse Sheet Former - TPSF was developed to produce handsheets under dynamic drainage conditions [Staib, 1991]. In general the TPSF describes a modification of a standard handsheet machine (*NobleandWood*). This modification concerns the drainage process of handsheet machine.
The process can be divided into two stages. The first stage called "turbulent thickening" is dominated by high shear rates due to agitation and high shear pressure-vacuum pulse cycles. Dewatering supported by vacuum and low shear rates represents the second stage of the process. By means of the produced handsheets retention and other paper properties can be evaluated after pressing and drying. The TPSF is presented in Figure 3.8.

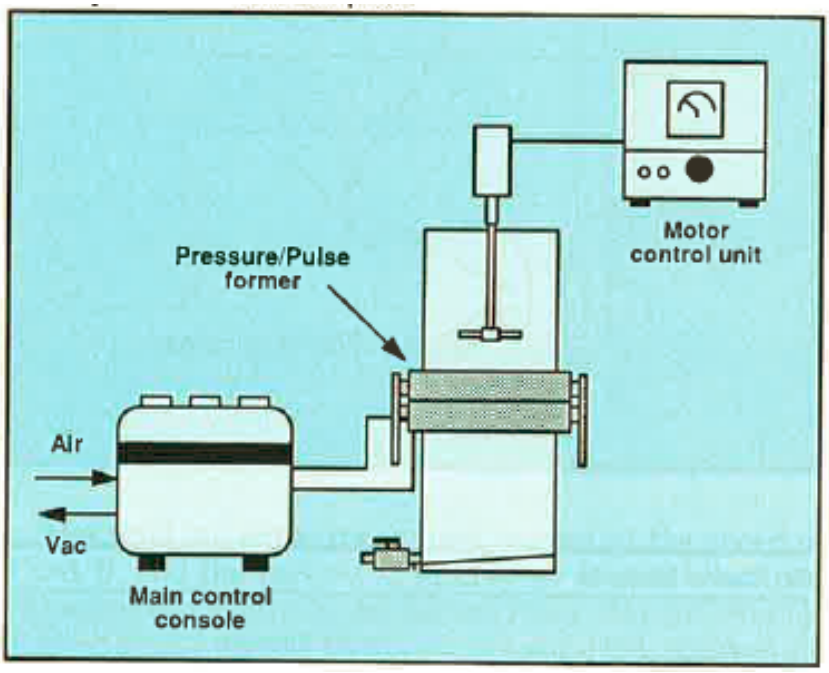

Figure 3.8 The turbulent pulse sheet former represents a modification of a standard handsheet former [Staib, 1991].

Pilot Former Plant - KISU

The pilot former plant KISU implemented at the VTT Technical Research Centre of Finland is a prototype for the measurement of retention, dewatering and formation [Dietz et al., 2010].
In general KISU consists of a headbox and a forming section representing a fourdinier former of a paper machine without press,- drying- or reeling section. The headbox consists of several replaceable built-in components used to generate high shear rates, the top and bottom plate are made of acrylic glass for observation of the flow dynam-

ics.
The dewatering of the fibrous suspension occurs on a length of $20cm$ supported by vacuum divided in four suction boxes. Each suction box is equipped with pressure transducers necessary to control the pressure level. The drained filtrate is collected separately in three different white water lines. The maximum achievable wire speed of the pilot former plant is about $480m/min$. A sample volume of about $1m^3$ prepared at headbox consistency is necessary for one trial.
The floc structure in the forming web is captured during the trial by a CCD-camera in conjunction with a light panel beneath the wire. Information on the dewatering behaviour is gathered based on two different approaches. On the one hand a second camera at the beginning of the forming section is used to observe the position of the dry-line of the drained furnish. On the other hand the built up fibre web is collected by carrier boards placed at the end of the former section. Retention is evaluated based on turbidity measurements of the collected white water.
A big advantage of the KISU pilot former is the number of process parameters which can be adjusted for each trial. These parameters are for example the flow rate, the wire speed, the consistency and temperature of the furnish but also the geometries of the headbox as well as the pipe diameter. Also this device enables the online-measurement of different chemical parameters such as the pH-value, conductivity, turbidity, viscosity as also consistency and ash content.
For dosage of chemical additives like retention aids or fixatives several dosage points are available, furthermore the residence time can also be varied.
In summary the pilot plant former KISU represents a measuring device for the investigation of retention, dewatering and formation under industrial-oriented conditions.

Pilot Paper Machine - FEX

The pilot plant machine FEX is a solution for large scale investigations. Pilot plants like the FEX at Innventia in Sweden are similar to industrial scale paper machines. The main difference is the web width of the produced paper which is only about $1m$. The FEX machine consists of a headbox, a former section as well as a press section. After pressing the samples are reeled up and can be dried off-line on two electrical heated cylinders.
There are two different types of headboxes available, a singly layer headbox and a three layer headbox. Depending on the desired drainage process the chosen headbox can be positioned at different positions in the forming section. The forming section of the FEX is designed as a twin wire section with the possibility to choose one of two available dewatering systems. For uniform drainage the roll former and for pulsating drainage a blade former is selectable. Depending on the dewatering principle the position of the headbox variates. At the end of the twin wire former a short fourdrinier section follows, generally used for forming of the outer ply of board products.

The following press section of the pilot plant machine is designed more or less conventionally, equipped with a double felted and two single felted press nips. As described above, after the press section the paper sample is reeled up and dried off-line.
For the addition of chemical additives to the stock thirteen dosage pumps and a number of dosing point depending on the added chemicals are available to simulate industrial conditions as closely as possible.
To allow the use of two different furnishes, there are two circulations equipped with a wire pit, screen and de-aeration available. For stock preparation the white water, produced during drainage is clarified by a disc filter and used as shower water and to dilute the broke.
The stock storage system of the FEX includes several tanks, with a total capacity of about $540m^3$ which corresponds about 20 tonnes of stock.
For control of the FEX a all components such as motors, pumps and valves are adjustable. During a run different parameters as flow rates, temperature, vacuum and pressure are measured and collected by a computer system.
In summary the pilot plant machine FEX comes very close to commercial paper machine, therefore for operation a relatively large stock volume is necessary. The advantage of such a plant is the lower costs for trials compared to full scale machines and in some cases some investigations and pre-trials are technically too complicated for commercial paper machine trials. Similar pilot machines also are available at machine manufacturers such as Voith or Valmet.

Retention/Formation- Machine

The R/F-machine simulates the short circulation of a paper machine and was developed for investigations of the retention/formation relationship [Svedberg, 2007; Svedberg and Lindström, 2010]. As shown in the schematic diagram in Figure 3.9 this device can be divided into four main sections. The first section includes a step diffuser headbox followed by a fourdrinier section and the reeling section. In order to simulate more realistic conditions the white water is handled in a white water system.
The headbox consists of a dispersion plate, where the inflowing suspension is distributed. This step is followed by a stilling chamber together with the turbulence generator followed by an adjustable slice. By varying the slice opening the jet to wire ratio of the machine can be adjusted. The width of the headbox as well as of the produced fibre web is about 300mm.
The forming section represents a fourdrinier former, including a forming table, foils and suction boxes connected to various vacuum pumps used to set different vacuum levels. By means of foils and suction boxes and depending on the machine settings a dry content of about 20% can be reached. The maximum wire speed of the R/F-machine is about $400m/min$. To enable such high machine speeds the pulp chest has

a volume of about $18m^3$, for the measurement of retention and formation for one trial a volume of about $1m^3$ is necessary.
For investigation of the influence of chemical additives this device includes eight dosing positions where five different additives can be added simultaneously. Depending on the machine settings the residence times can also be varied.
The measurement of the first pass retention is done by analysis of the solid content of the white water. The formation of the produced web after drying is analysed by the FUJI-Beta-Radiograph method based on the measurement of local variations in grammage. Additionally this device allows the evaluation of flocculation by a camera positioned over the transparent headbox.
In summary the R/F- machine represents a sophisticated laboratory device for investigation of retention and formation under industry-oriented conditions. A big advantage of this device is the high amount of possible settings to simulate conditions as close to reality. By means of simultaneous measuring information regarding relationship between retention and formation can be gathered in an accurate manner.

Twin Wire Retention Tester

The twin wire retention tester - also called the high speed retention tester (HSR-Tester) - represents a laboratory device simulating drainage conditions in a gap former [Sivén and Manner, 2001]. The problem of commercial laboratory devices for measuring drainage and retention behaviour is the one-sided drainage process often without support of vacuum. Thus the collected data are not comparable to results achieved with gap formers.
The high speed retention tester represents an alternative method for the measurement of dewatering and retention on the laboratory scale. The big difference to real gap formers is the movement of the main parts. The HSR- Tester consists of a cylindrical frame where the wire is fixed. The rotating parts of the device are the headbox as well as the foil frame. Via the headbox the suspension is injected between the wire and an outer placed fabric in one turn. After injection the headbox stops the movement and the foil frame induces pulsation by rotating at the inner side of the wire. The pulsation can be adjusted by the rotation frequency as well as the number of foils. To improve dewatering and to achieve more realistic conditions drainage is supported by vacuum generated on the outer side of the wire. The dosage of additives is possible at three different points, one before and two after the stock pump, the residence time of the dosed additives can also be adjusted.
Investigations have shown that the HSR- Tester represents a laboratory device capable of measuring dewatering and retention behaviour close to a real gap former.

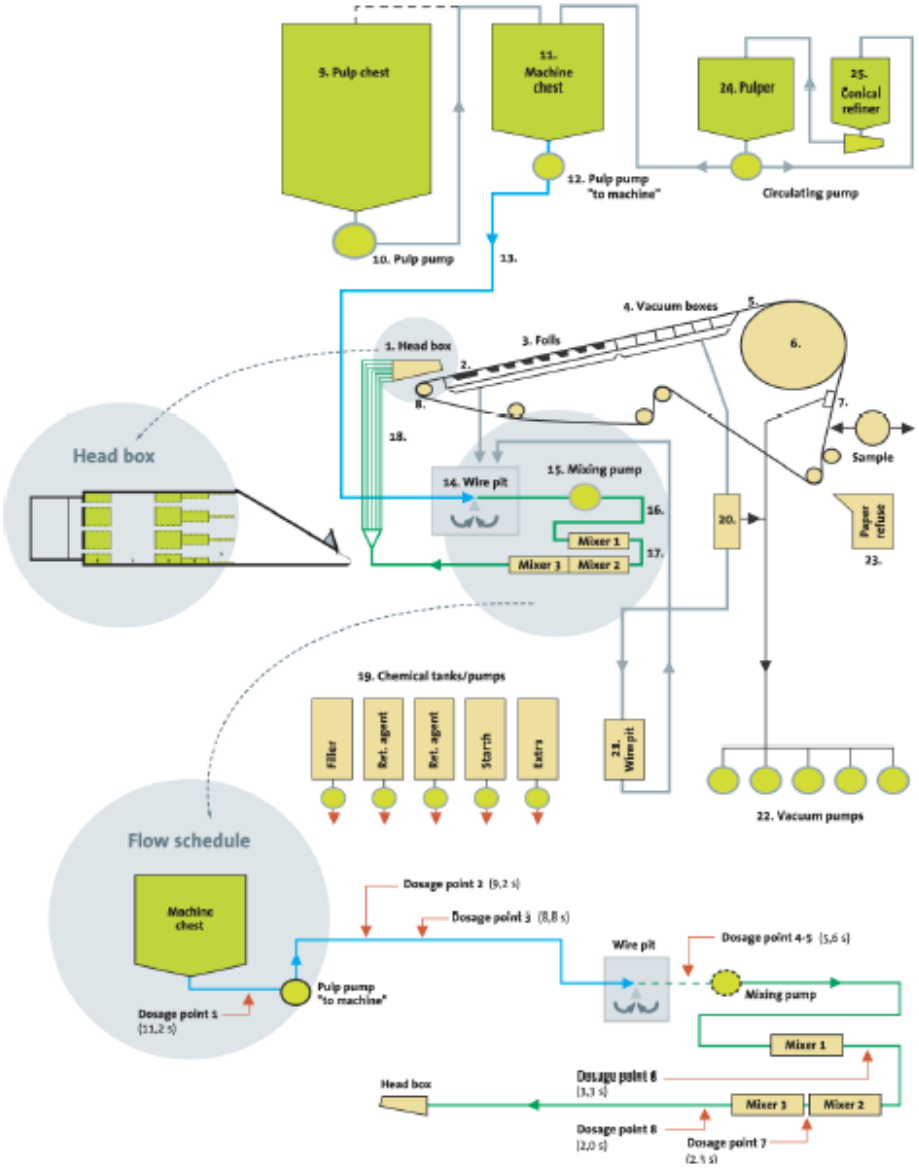

Figure 3.9 Schematic diagram of the R/F- machine with its surrounding equipment, the flow chart of the suspension as well as the white water. This machine consists of a headbox followed by a fourdrinier former, a reeling section and a white water circulation. Drainage is supported by foils and suction boxes connected to several vacuum pumps. Eight points of dosage for five different additives are possible. Because of the large volumes necessary for one trial a pulp chest containing a volume of $18m^3$ and a machine chest with a volume of about $1m^3$ are available [Svedberg, 2012].

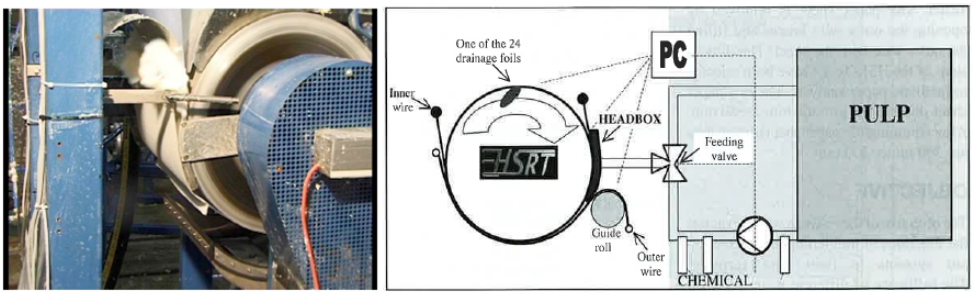

Figure 3.10 The twin wire retention tester represents a methodology to simulate drainage and retention behaviour prevailing in gap formers. a) The cylindrical frame with the two wires and the rotating headbox are shown. b) A schematic diagram of the device including the dosing points [Sivén and Manner, 2000].

In comparison to the DDJ as well as the modifications the presented large-scale devices enables the measurement under conditions close to the reality. Therefore these devices operates as commercial machines using a moving wire, suction boxes and dewatering elements and/or a white water circulation system.
The great disadvantage of these devices (in comparison to the DDJ) is the large sample volume necessary for each trial to achieve realistic conditions. Because of the large volumes these measurements are much more time-consuming and require a great effort.

3.2 Measurement of Flocculation in Flowing Suspension

In general the measurement of flocculation in flowing suspension is carried out based on image analysis. Several researchers in the past designed circulation devices including components to generate turbulences followed by a transparent region for observation and image acquisition. In this section some of these devices are described.

A setup for measuring flocculation in flowing suspension was designed at the by Beghello Beghello et al. [1996]. It is designed as flow loop (see Figure 3.11a and is an image analysis-based floc-measuring device. In principle the design consists of a tank containing the suspension at actual paper machine consistencies. A centrifugal pump in combination with a flowmeter transports the suspension from the slurry tank to the observation pipe with a cross section of $150mm$ in width. For image acquisition a digital camera with high resolution is used. The light source is an electronic flash timed to coincide with the operation of the camera.
Special for this device is the so called "dark room", shown in Figure 3.11b, which represents a plastic box containing white and black plates of plastic. The dark room is positioned in the area of observation and necessary to achieve uniform distribution

of the flash light.
For each trial 10 up to 20 digital images with an area of 80 x 120 mm are acquired. The images acquired in this setup are analysed separately in MD (machine direction) and CD (cross direction) by a one-dimensional Fourier transformation. The final result of the evaluation is the average of all spectra in the MD and CD direction. Before evaluation the images have to be prepared. This pretreatment includes

- conversion into gray scale
- visual inspection with regard to illumination errors
- cutting images to a size of 128 x 128 pixels
- normalization

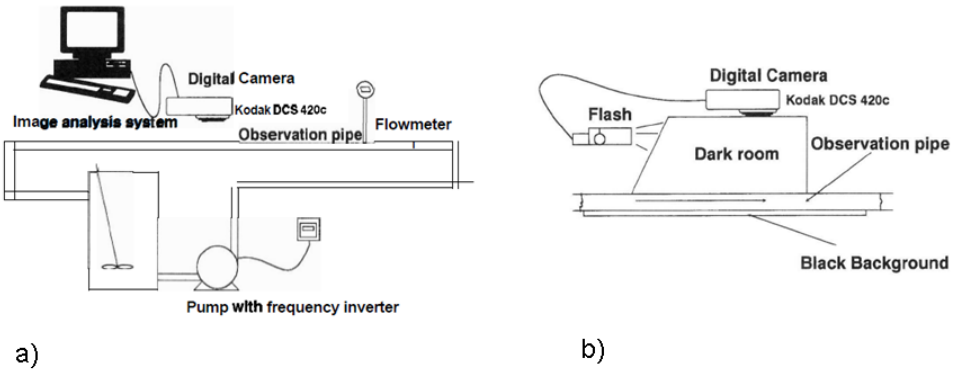

Figure 3.11 The measuring setup is designed as a flow loop for the evalutation of floccualtion in flowing suspension. a) The whole setup of the flocculation device. The device consists of a pulp storage tank, a centrifugal pump a flowmeter as well as an observation pipe. Image acquisition is done by a digital camera in combination with an electronic flash. b) The setup for image acquisition in more detail. A so called "dark room" ensure an uniform distribution of the flash light in the area of observation [Beghello et al., 1996].

Another device designed for the measurement of flocculation was developed by Yan Yan et al. [2006] at the Royal Institute of Technology, KTH. The idea for that setup is the obseration of the flocculation behaviour under realistic headbox flow conditions.
The setup as illustrated in Figure 3.12a consists of a flow loop including a pulp chest equipped with an impeller as well as heating and cooling devices, a frequency controlled centrifugal pump and an electromagnetic flowmeter. The concentration of the suspension normally used for the evaluation is in the range of 2.5 to 7.5 g/l, higher concentrations up to roughly 10 g/l are also possible. For the ability to evaluate the influence of chemical additives on fibre flocculation several dosing points equipped with static mixers are installed.

For the simulation of realistic headbox flows the setup includes a flow distributor and a transparent headbox with a step diffuser. After the headbox a contraction block including the so called headbox-contraction, an extended nozzle followed by a removable secondary contraction, is installed (see Figure 3.12b. By means of the secondary contraction studies of fibre floc rheology are carried out by simulating flow accelerations comparable to the industrial forming process. The measurement of fibre flocculation in flowing suspension is carried out in the extended nozzle. A high speed CCD camera in conjunction with transmitted infra-red laser light is used for image acquisition. The images of fibre flocculation are evaluated by analysis methods like power spectrum and wavelet transform analysis.

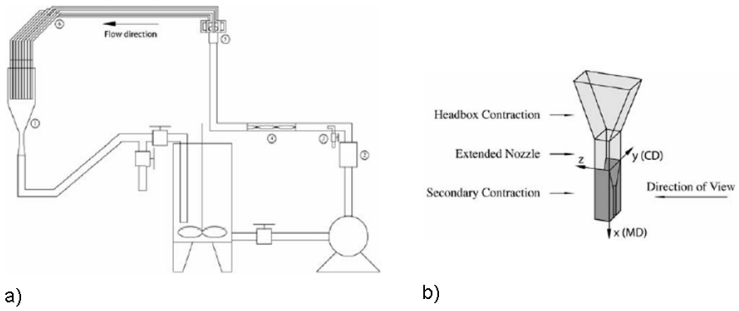

Figure 3.12 a) The flow loop designed by Yan Yan et al. [2006] enables the evaluation of flocculation behaviour under realistic headbox flows. The device consists of a pulp chest followed by a centrifugal pump as well as a flowmeter, several dosing point equipped with static mixers, a flow distribution, a transparent headbox and a contraction block where the measurement is carried out.
b) The contraction block consisting of a headbox-contraction, an extended nozzle followed by a secondary contraction. The measurement of flocculation is done in the area of the extended nozzle by a CCD camera and transmitted light.

Another setup was developed at the Centre Technique du Papier by Huber et al. [2004] to study the influence of retention aids on fibre flocculation. The setup is similar to the already introduced ones except that the suspension flow occurs under static head. A centrifugal pump carries the suspension from the pulp chest to an overflow tank as is shown in Figure 3.13. From the overflow tank the suspension at headbox consistency passes a flowmeter, two points of dosage and an observation channel. At the channel a CCD camera and a light source are installed for image acquisition. The evaluation of the images is carried out by a purpose-built analysis method to obtain the floc size distribution.

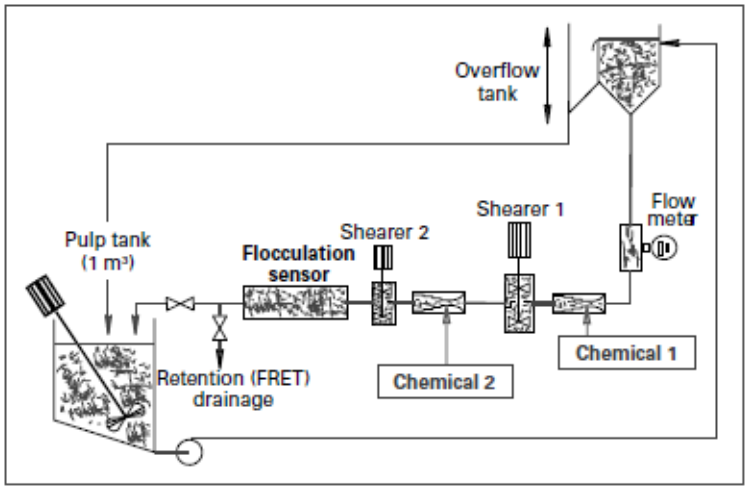

Figure 3.13 The measuring setup for evaluation of flocculation behaviour. This device is similar to the others introduced, but the suspension flow occurs under static head conditions controlled by a flowmeter. The measurment itself occors at the flocculation sensor including a transparent rectangular channel, a CCD camera and a light source [Huber et al., 2004].

In summary the presented methods for the measurement of flocculation are relatively similar. The flocculation behaviour is evaluated by image analysis of the flowing suspension. Differences between the described methods are in the measurement setup allowing different shear rates and in the analysis methods of the acquired images.
The measurement of flocculation used in the device presented in this PhD-thesis is similar to those and will be described in more detail in the following section.

3.3 Remarks

For the measurement of retention, dewatering and/or flocculation there are a high number of laboratory devices available.
The small-scale devices, as discussed in section 3.1.1 and section 3.1.2, enables an easy and fast measurement primarily because of small sample volumes. On the other hand, because of the compact design of these devices the measurement conditions (static wire, impellers) are not comparable to industrial one.
For simulation of conditions closely to reality various large scale devices, as discussed in section 3.1.3, were developed. The design of these devices is more close to industrial machines (moving wire, drainage elements, white water circulation). The disadvantage of these devices is the requirement of large sample volume and therefore the measurements are much more time-consuming (compared to small-scale devices).
Because of that facts a laboratory device combining the advantages of a small sample volume and industry-oriented conditions has to be developed.

Chapter 4

A novel device for simultaneous measurement of retention, dewatering and flocculation

In this chapter a novel device for the simultaneous measurement of retention, dewatering and flocculation is presented. As discussed in chapter 2 sheet forming is influenced by these three parameters to a great extent. On the one hand each of these parameters influences the forming process by its own, on the other hand they are highly interrelated.
In section 3 several devices used for the measurement of each separate parameter or for the evaluation of two parameters in one step were presented and discussed. The laboratory devices enable fast and easy measurement, but the results are difficult to translate to the industrial scale because of the completely different setup. Equipment in larger scale like a pilot plant enables the measurement under industry-oriented conditions, however the necessary suspension volume for a trial is quite large and the effort for preparation and execution of trials is very high.
To combine the advantages of laboratory and larger scale devices a novel device for simultaneous measurement of retention, dewatering and flocculation was developed. This device is presented and discussed in detail in the following.

4.1 Overview

In general the device for simultaneous measurement can be divided into two separate units, the flocculation unit for evaluation of the flocculation behaviour and the dewatering unit for measuring retention and drainage behaviour. A schematic illustration of the device is shown in Figure 4.1. For the measurement the suspension is stored in

a pulp chest containing a volume of 40 litres, a centrifugal pump is used to transport the suspension trough the flow loop to the measuring units.

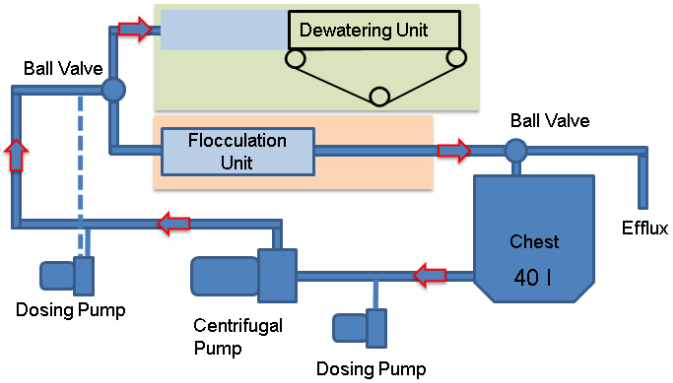

Figure 4.1 A schematic illustration of the novel laboratory device. The device consists of a pulp chest, a centrifugal pump, two dosing pumps and three dosing points. The device is divided into two units, the flocculation unit with a transparent observation channel and the dewatering unit including a headbox and a fourdrinier former section.

Between the pulp chest and the measuring units there are three points of dosage were wet end chemicals can be added using two dosing pumps. To apply high shear after dosage one of the three points of dosage is installed before the centrifugal pump. As mentioned above the presented device includes two measuring units. The flocculation unit consists of a transparent rectangular observation channel. The measurement of the flocculation behaviour is based on images of the flowing suspension. A high speed camera is installed on top of the channel and a light source beneath. Evaluation of the acquired images is based on Fast Fourier Transformation integrated in an especially developed image analysis tool. The setup for the evaluation of the flocculation behaviour was developed by Eckhart et al. [2013] and will be explained in this section in more detail

The second measuring unit included in the setup of the device is the so called dewatering unit. To be able to measure under industry-oriented conditions this unit consists of a headbox used to distribute the suspension evenly, a dewatering device representing a fourdrinier former section including vacuum supported drainage and a white water tank to store the filtrate gathered during the drainage process. Based on the stored white water the parameters retention and drainage efficiency can be evaluated in the laboratory. Figure 4.2 shows an image of the measurement setup.

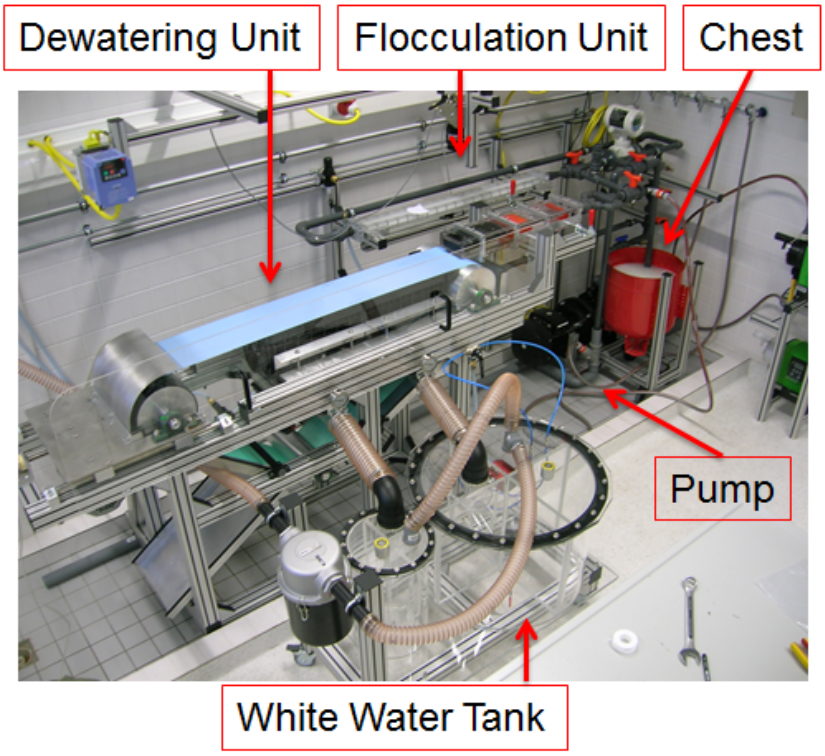

Figure 4.2 The measurement setup of the novel device. The suspension is stored in the pulp chest with a volume of 40 litres. Using a centrifugal pump the suspension is transported to the flocculation unit and the dewatering unit.

To minimize operater errors and to achieve always constant measuring conditions the whole measuring procedure is controlled by a process control system.

4.2 The measurement setup

In this section the laboratory device is presented and discussed in more detail. Section 4.2.1 presents the basic elements of the device including the flow loop as well as the dosage of additives. In the following section 4.3 the flocculation unit is presented including the observation setup, the acquisition process as well as the evaluation of the acquired images. In section 4.4 the dewatering unit of the novel laboratory device including the headbox, the fourdrinier former as well as the white water storage are discussed. Finally the process control system necessary to ensure accurate and reproducible measurements is presented.

4.2.1 Basic elements of the laboratory device

As mentioned in section 4.1 the fibrous suspension has to be pumped trough a flow loop until a stable flow is reached.
The flow loop can be divided into a few basic elements the pulp chest, the centrifugal pump, the dosage points, the loop extension, the flow meter as well as the ball valve necessary to redirect the suspension at the end of the loop. The basic elements are also shown in Figure 4.3.

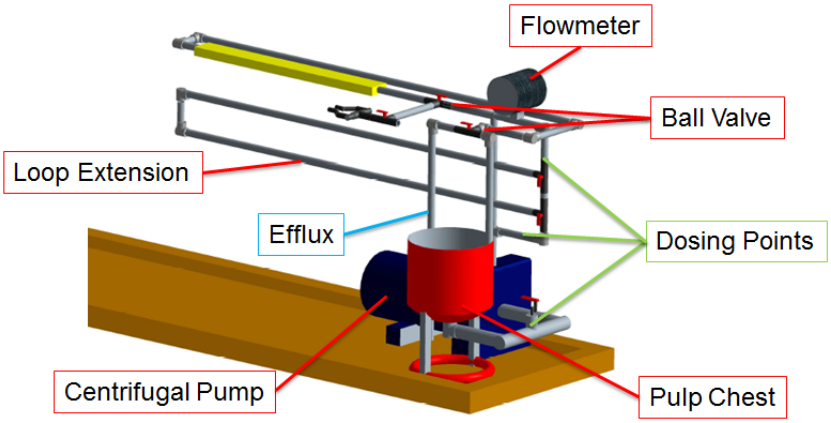

Figure 4.3 The most important basic elements of the novel laboratory device. Elements necessary for the transport in the loop are marked red, the green marked elements are important for dosing additives. When chemicals are dosed, the suspension leaves the closed loop through the blue marked efflux.

For each trial a volume of forty litres of suspension including fibres and fillers is required, the consistency of the sample is in the range of 1%. To prevent sedimentation of the components of the suspension an eccentrically positioned impeller with three blades is used in the pulp chest shown in Figure 4.3.
To transport the suspension trough the loop a centrifugal pump with a radial design is used. The most important performance characteristics of the pump are a conveying capacity of 22.3 m^3/h, a discharge head of 44.3 m and an engine power of 5,50 KW. Because of a viscosity of the fibrous suspension close to water and the very low consistency the radial design was chosen, furthermore this type shows advantages like minimal maintenance as well as robust and simple design. For the adjustment of the the flow speed a frequency converter with integrated PID controller is used.

To evaluate the influence of chemical additives there are three points of dosage installed in the loop. The first point of dosage is placed between the pulp chest and the centrifugal pump. As commented in section 2.3 some mechanism of flocculation, for example complex flocculation, require high shear to take full effect. These high

shear rates are generated by the rotor of the centrifugal pump. The second and third point of dosage are placed after the pump at different positions illustrated in Figure 4.3. The main difference between these two points is the distance between addition and measurment allowing different reaction times of the additives. The whole dosage setup installed is explained in more detail in this section.

An additional component for reaction time variation is the loop extension. The loop extension has a length of about 4 meters and can be, depending on the used additives, connected or disconnected by ball valves. Depending on the flow velocity in the loop the connected loop extension increases the reaction time of additives between two to four seconds.

Another important component integrated into the loop is the inductive flow meter. The flow meter used for this setup is the ProcessMaster FEX 311 manufactured by ABB. The operating area of this flow meter is in the range between 0,06 l/s and 3,33 l/s at a maximum pressure of 10 bar. Because of the working principle of the inductive flow meter without moving parts an accurate measurement especially for fibrous suspension is possible. To achieve constant flow velocity during the measurements the inductive flow meter is connected to the integrated PID- controller of the frequency converter used to control the pump speed.

To channel the suspension flow to different positions of the setup several ball valves are installed, shown in Figure 4.3. Using the valve positioned above the chest the suspension flow can be varied between the pulp chest and the efflux. For the start-up process as well as for measurements without additives the flow is channeled back to the chest, however, as soon as chemical additives are dosed the flow is directed to the efflux to prevent overdosage. The ball valve similar after the flowmeter is used to channel the suspension flow between the two measuring units. In the standard mode the flow is channeled through the flocculation unit.

In Figure 4.4 the flow direction in the loop is shown. The centrifugal pump carries the fibrous suspension from the pulp chest past the dosage points, through the loop extension and the flow meter back to the chest. By setting different positions of the integrated ball valves the extended loop can be de-activated as well as the flow can be channeled back to the chest or to the efflux. The pipes used in this setup are made of unplasticized polyvinyl chloride. The advantage of these tubes are a high chemical resistance and the smooth surface of the of the pipe wall, necessary for the use of chemical additives, as well as a high durability for high pressure purposes (16 *bar* max.). For this setup the used tubes show two different diameters. The diameter of the pipe between the chest and the pump is 63mm. After the centrifugal pump the pipes were designed with a diameter of 25mm. At the diameter of 25mm the flow

velocity in the loop is achieved at medium pump speed. The pump would allow higher flow speeds when special trials would require it. A higher pipe diameter would also relate to a higher loop volume which means a larger chest volume would eventually be necessary. The entire pipe length of the flow loop including the loop extension is about 11 m, this corresponds to a pipe volume of about eight litres. To allow easy drainage of trapped air in the loop the entire pipeline is installed in a gently sloping of about 3%.

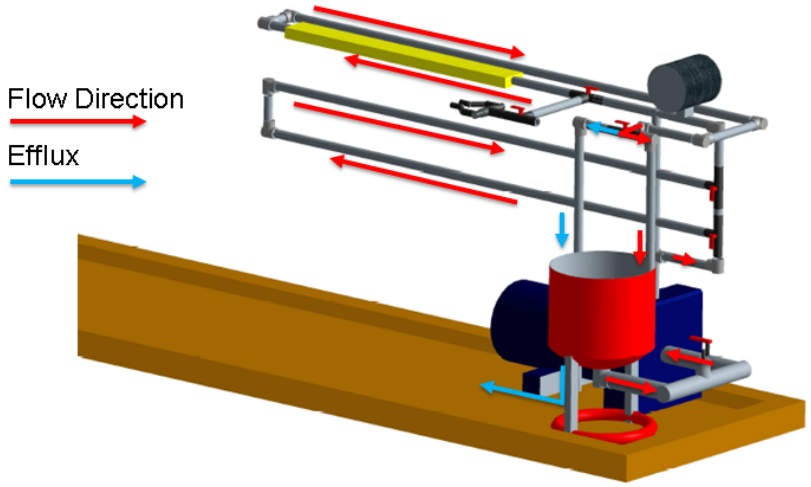

Figure 4.4 The flow direction in the loop setup. Starting from the pulp chest the suspension passes the centrifugal pump, the loop extension as well as the flow meter, after the ball valve at the end of the loop back to the chest. The red bars show the circulation of the suspension in the flow loop, the blue bars show the flow if the suspension leaves the closed cycles.

Dosage of additives

For the evaluation of chemical additives and their effects on retention and drainage behaviour as well as on flocculation there are three points of dosage in the flow loop. In this section the design and the main components of one of these dosage units are explained in more detail.

In general the dosage of chemical additives is done using DME 150 dosing pumps manufactured by Grundfos, as shown in Figure 4.5. The capacity of the DME 150 is in the range of 20 ml/h up to 150 l/h, the dosed volume during the discharge/suction cycle is about 17 ml and is controlled by variation of the stroke speed by a microprocessor. The application area of these dosing pumps for investigations in the laboratory device is in the range of 15 l/h to 45 l/h.

Figure 4.5 The entire dosage unit. The dosing pumps used for the laboratory device are called DME 150 manufactured by Grundfos. The dosing capacity of the DME 150 is in the range of 20 ml/h up to 150 l/h.

The disadvantage of this type of pumps are the pulsations caused by the discharge/suction cycles. Because of the small pipe diameter as well as a low volume of suspension in the loop the pulsations cause fluctuations which influence the measurement accuracy. To minimize these pulsations three additional elements are installed between the pump and the point of dosage. These elements, illustrated in Figure 4.6a beginning from the pump are:

- a pulsation damper
- a pressure-sustaining valve
- and a non-return valve.

To ensure perfect operation of the damper a minimal counter-pressure of 0,5 *bar* is necessary. Because of a medium line pressure of 0,3 *bar* during operation a pressure-sustaining valve is installed between the pipeline and the damper. For optimal working conditions the damper itself is preloaded to a pressure of 0,2 *bar*. To be able to control and adjust these operating conditions pressure manometers are installed as shown in Figure 4.6a. For the injection itself a non-return valve is used. To assure a homogeneous distribution of the dosed additives over the entire cross section the injection needle of the valve is positioned in the centre of the pipe. As shown in Figure 4.6b the drain angle of the needle is 45°.

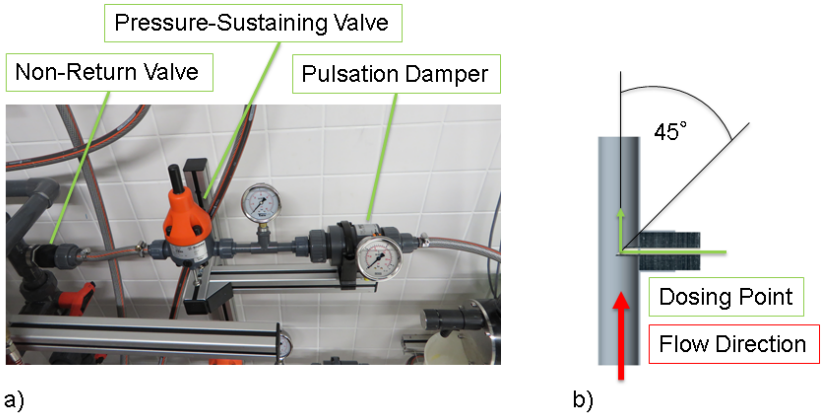

Figure 4.6 a) The elements of the dosing unit. These elements are the pulsation damper, the pressure sustaining valve and the non-return valve. These three elements are necessary to prevent volume fluctuations caused by pulsations. b) The points of dosage in the pipe are shown in more detail. The injection needle of the non-return valve is positioned in the centre of the pipe to achieve a homogeneous distribution of the dosed additives.

4.3 Flocculation Unit

In this section the flocculation unit is described in more detail. This unit, in general developed by Eckhart et al. [2013] is used to evaluate the flocculation behaviour of fibrous suspensions. The unit consists of an observation channel, a high speed camera as well as a LED-light panel necessary for image acquisition in the flowing suspension. The setup of this unit as well as the evaluation procedure based on the acquired images is described in more detail in this section.

4.3.1 Basic elements of the flocculation unit

The measurement of the flocculation behaviour of fibrous suspensions is based on the evaluation of images of the flowing suspension. The elements used for the acquisition are an observation channel, a high speed camera and a light panel. These elements, shown in Figure 4.7, are described in more detail.

4.3.2 Observation Channel

For observation of the flowing suspension a transparent channel with rectangular cross section was designed. The flow channel has a length of 1000 mm, a width of 35 mm and a height of 16 mm. The standard flow velocity at which the measurements are carried out is 2 m/S. To generate turbulences, at the inlet of the channel a constriction block with half the cross section (35 mm wide, 8 mm height) was installed,

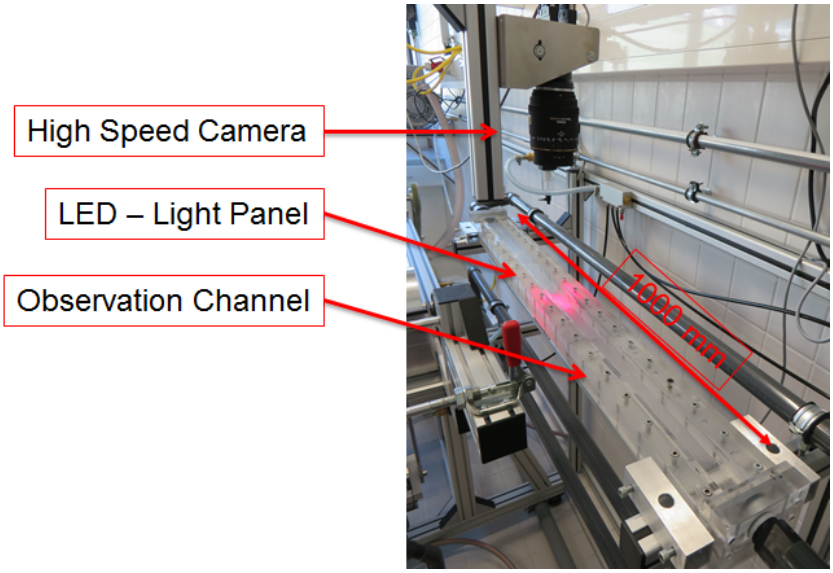

Figure 4.7 The flocculation unit consisting of an observation channel, a high speed camera and a LED-light panel. Using this equipment images of the flowing suspension are acquired.

the corresponding velocity in the constriction is about 4 m/s.

The observation channel, shown in Figure 4.7, consists of a bottom and top plate with a thickness of each of about 12 mm. At the inlet and outlet of the channel there are additional plates which include connectors for the pipeline. The material used for the transparent design of the channel is polycarbonate. The outside dimensions of the channel are 1040 mm in length, 100 mm in width and 40 mm in height.

The sealing between the plates is achieved based on slots in which a sponge rubber is placed and pressed onto the counter-plate by individual screw joints in the distance of 50 mm, also shown in Figure 4.8.

4.3.3 Image Acquisition

For image acquisition of the flowing suspension a high speed camera (Basler A504k) positioned above the channel and a LED-light panel (CCS LDL-60X60) beneath are installed, as shown in Figure 4.7. The hardware as well as the software are adopted from the setup developed by Eckhart et al. [2013].

High Speed Camera The Basler A504k is a high speed camera with a frame rate of max. 500 frames per second at full resolution (pixel size of 1280x1024). The sensor used for this area scan camera is a monochrome 1280 x 1024 CMOS progressive scan with a pixel size of 12 x 12 μm. For the field of application a monochrome sensor is sufficient, on the one hand the use of a color sensor does not provide any additional

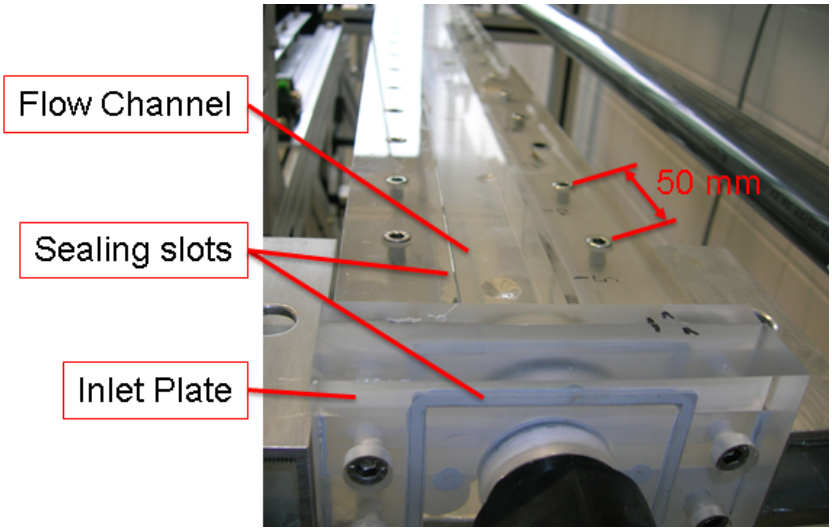

Figure 4.8 The observation channel in more detail. The material used for the transparent design is polycarbonate. The sealing is achieved by slots in which a sponge rubber is placed and pressed onto the counter-plate.

information, on the other hand a monochrome sensor shows a higher light sensitivity which is advantageous for fast moving objects. An adequate exposure is assured by the electronic full frame $TrueSnap^{TM}$ shutter with an exposure time from 10 μs to greater than 33 ms.
The camera lens used for this setup is a Sigma 70mm F2.8 EX DG Macro, the features of this lens are listed bellow.

- Construction: 9 groups / 10 elements
- Field of view: 34,3 deg.
- Aperture: f/2,8
- Minimal focus distance: 0,257 m
- Filter size: 62 mm

To be able to store the high number of images per second the camera is connected with a Framegrabber (Epix Framegrabber PIXI CL3SD). 818 images at full image size (1280 x 1024) can be acquired directly on the framegrabber. Thereafter it is necessary to save the images on a hard disc otherwise they may be overwritten by a new image sequence acquired. The settings evaluated by Eckhart and used for the measurements are mentioned in section 4.3.4.

Image Acquisition Software The acquistion of images of the flowing suspension is done by Epix XCAP. This software is used as the connective link between the camera

and the framegrabber mentioned above. Furthermore the software allows the definition of different settings like frame rate, scan size, exposure time and gain.

Illumination As mentioned above, the illumination unit is placed beneath the channel, the images are obtained in transmission mode. The equipment for illumination consists of a LED- Light Panel (CCS - LD 60x60) as well as a LED- Lightning Controller (*GardasoftVision* PP500). The square shaped light panel with an edge length of 60 *mm* illuminates the observation channel using 144 high power LEDs. The panel operates in the range of the red light at a wavelength of 660 *nm*.
The control and the setting of the panel is done by the LED- Lightning Controller. This unit acts as a connector between the camera and the light panel and the settings can be controlled by the image acquisition software (Epix XCAP). The pulse width supported by the controller is between 20 μs to 999 *ms* and can be adjusted in steps of 20 μs.
Using the Lightning Controller the LED Panel works as a flash and thus the LED panel can be supplied with a power 10 times higher than allowed for standard operating conditions.

4.3.4 Settings for the Image Acquisition

The settings for image acquisition include the position of the camera along the channel, the focus level, the flow velocity as well as different settings in the acquisition software (Epix XCAP).
The optimal position of the camera was evaluated by Eckhart and is chosen 500 *mm* downstream of the inlet. Investigations have shown that at this point turbulences caused by the constriction block at the inlet, have decayed and structures are not subjected to further changes.
Another important setting is the focus level which has to be adjusted by hand. The depth of focus is too low to focus the entire height of the 16 *mm* high channel. Therefore the focus level for this setup is approximately in the centre of the channel.
For image acquisition a couple of parameters have to be chosen before the measurement can start. These are the frame rate, scan size, exposure time and the gain. In Figure 4.9 the operator window of the software (Epix XCAP) including the chosen settings is shown.

For each trial point a series of 200 images is acquired, which means an acquisition time of about 4 seconds, depending on the chosen frame rate. The possible field of view achieved by the equipment is a resolution of 1280 x 1024 pixels which means an area of 40 x 50 *mm*. To limit image acquisition to the dimensions of the channel the scan size is corrected to 1280 x 700 pixels. The exposure time is set at 0.400 *ms* and controlled by the lightning controller (*GardasoftVision* PP500).

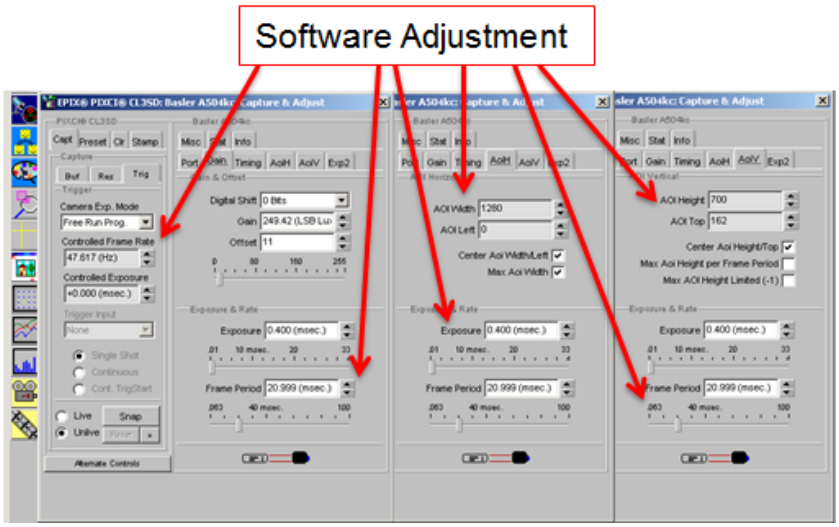

Figure 4.9 A part of the operator window of the image acquisition software. The shown values for frame rate, gain, exposure time, and scan size are used for the measurements in the flocculation unit.

4.3.5 Image Analysis

In this section the pre-treatment as well as the structure analysis developed by Eckhart et al. [2013], [Eckhart, 2008] is presented. The pre-treatment of the images includes three steps. Figure 4.10 shows an acquired image as well as the image after the pre-treatment. At first uneven illumination caused by the acquisition setup (less illumination at the edges of the image) are compensated. In a second step the grey values of the images are normalized and thus the contrast of the images is equalized and comparable. At last the images are cut into quadratic shape with a size of 560 x 560 pixels necessary for the evaluation based on the Fast Fourier Transformation. Flow conditions of the boundary layer caused by the channel wall are eliminated, too. The whole process of the pre-treatment is automated using by algorithms implemented in $Matlab^{®}$ version 7.9.0.529$(R2009b)$.

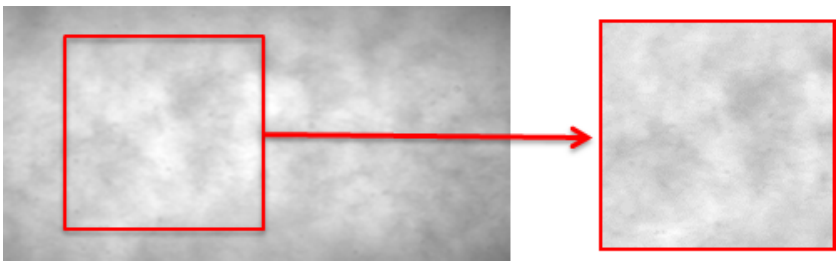

Figure 4.10 The result after the pretreatment of an acquired image. The pre-treatment, including compensation of uneven illuminations, normalizing of the grey values and cutting into quadratic shape is fully automated.

To determine the size distribution of structures in the pre-treated images a structure analysis algorithm (Fast Fourier Transforms) implemented in *Matlab*® is used on a set of 50 images. The results from the analysis are displayed in a diagram as shown in Figure 4.11. On the abscissa the wavelength of the detected structures are shown, on the ordinate axis the variance of the structures is displayed. A high value in variance means a high contrast between specific structures in the respective wavelength.
Figure 4.11 shows the whole process from the acquisition illustrated in a), the images after the pre-treatment shown in b) and the diagram after analysis in c). The samples used for this example are a thermo-mechanical pulp and a chemical pulp. Both samples are diluted in water at a consistency of one percent. In comparison to the thermo-mechanical pulp a chemical pulp shows a higher tendency to build large flocs. The structure analysis illustrates this effect. Above a wavelength of 2 *mm* the detected structures can be considered as flocs. The comparison of the two measured samples shows the increased flocculation tendency of the chemical pulp.

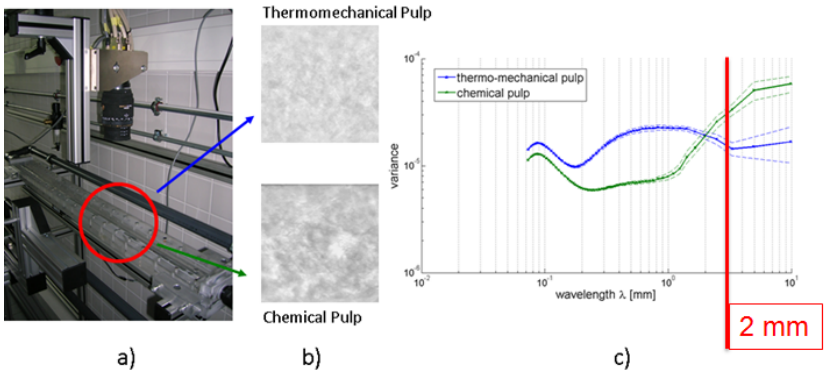

Figure 4.11 The measurement of the flocculation behaviour. a) The measurement setup is illustrated, in b) the acquired images after pre-treatment of two different samples are shown and in figure c) the results achieved by image analysis are presented. Above a wavelength of 2 *mm* the detected structures can be considered as flocs, the chemical pulp shows a tendency to build large flocs.

4.4 Dewatering Unit

In this section the dewatering unit of the device is presented. After the measurement of the flocculation behaviour the fibrous suspension is directed to the dewatering unit using a ball valve (shown in Figure 4.2b. This unit is used for the measurement of retention and drainage behaviour. In general the device consists of a kind of headbox as well as a wire section (see Figure 4.12). The wire section includes components like a commercial wire, a vacuum-supported dewatering zone, a wire tension equipment, wire cleaning equipment and a white water tank. The headbox and its design will be

discussed in more detail in section 4.4.1. The different components of the wire section are presented in section 4.4.3.

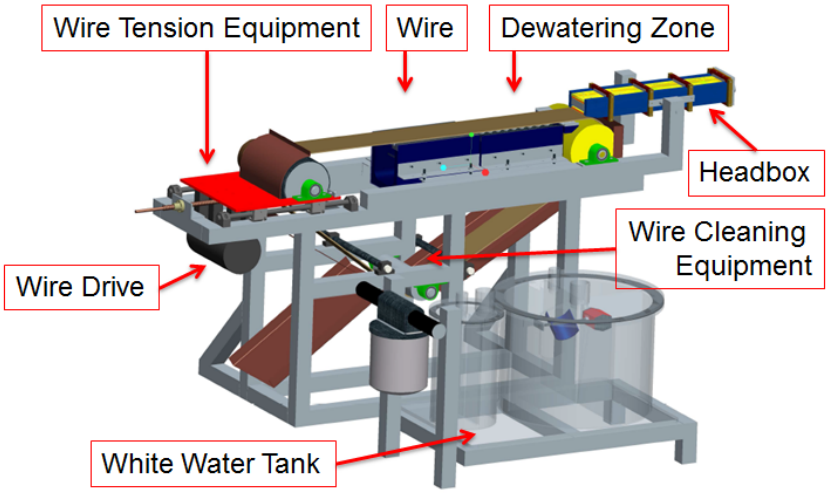

Figure 4.12 The dewatering unit. This unit consists of a headbox, a drainage wire, a vacuum supported dewatering zone, a wire tension equipment, a wire drive, a cleaning equipment as well as a white water tank.

4.4.1 Headbox

In this section the headbox of the dewatering unit is discussed in more detail.
In general this device is used to distribute the flowing suspension evenly over the entire cross section. The headbox is designed as a flow channel with rectangular cross section of $620x140x70\ mm$ (LxBxH). The geometry of the flow channel or inner housing is 600 mm in length, 100 mm in width and 30 mm in height. A schematic illustration of the designed headbox is shown in Figure 4.13.

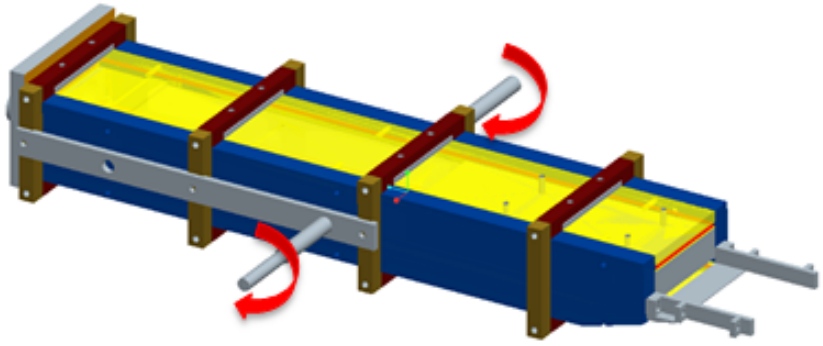

Figure 4.13 The dewatering unit. The side plates, the bottom and top plate of the channel are made of in transparent polycarbonate. For de-aeration the channel has to be turned from vertical into horizontal position using swivel arms fixed on the frame elements.

The inlet and all four walls are made of transparent polycarbonate allowing the observation of the flow conditions in the channel. The sealing of the device is similar to the observation channel discussed in section 4.3.2. In the peripheral areas of each plate slots are machined were sponge rubbers are placed. By screwing the plates together the sponge rubbers are pressed onto the counter plate sealing the housing of the channel.
To improve sealing and to stabilize the cross section of the housing four individual frame elements are placed around the channel. On the top and bottom side of the frame elements there are integrated pressure plates, by means of these plates the top and bottom plates of the channel can be pressed onto the built in components (explained below) individually.
Using swivel arms, installed at the frame elements (see Figure 4.13) de-aeration of the headbox is possible. For de-aeration the headbox is turned from horizontal to vertical position, shown in Figure 4.14. After the channel is filled completely with water it is turned back without any loss of fluid.

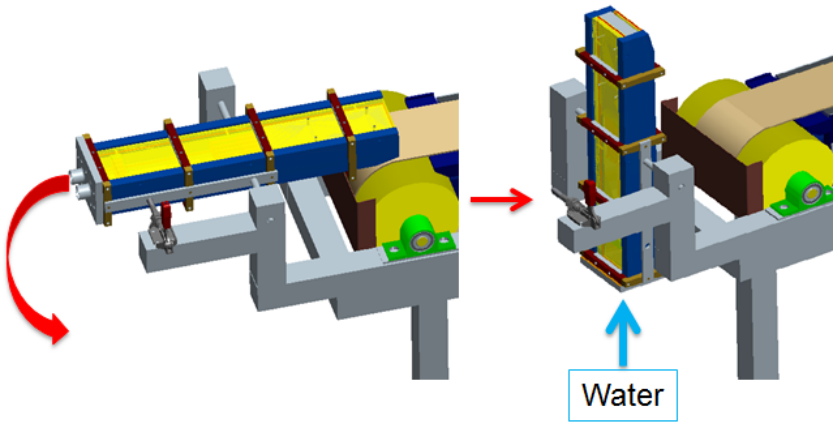

Figure 4.14 The de-aeration process of the headbox. The headbox is turned form horizontal to a vertical position and is filled up with water.

4.4.2 Buit-In Components

As mentioned above the headbox is used for an even distribution of the flowing suspension across the entire cross section. Because of the requirement to operate with low sample volumes (40 litres) the possible flow velocities are limited and in general they are too low for simulation of flow conditions resembling the conditions in an industrial headbox.
During the development of the headbox two versions of built in components were designed and evaluated. These versions shall be discussed in detail.
For the first version components geometrically similar to a real headbox were designed. In Figure 4.15 an illustration of theses components is shown. The fibrous

suspension passes the flow channel through two inlet pipes and is distributed over the entire cross section. For additional distribution and to generate turbulences the suspension passes a hole plate. After a calming zone the suspension is accelerated by means of a two-step diffuser. At the outlet of the diffuser turbulences are generated and thus flocs should be destroyed and re-distributed. In the last step the fibrous suspension has to be accelerated up to machine speed in a nozzle. The nozzle is designed as a spline with a length of one third of the total length of the headbox. At the end of the nozzle representing the exit of the headbox the suspension flows through a gap with a cross section of 5 x 100 mm.

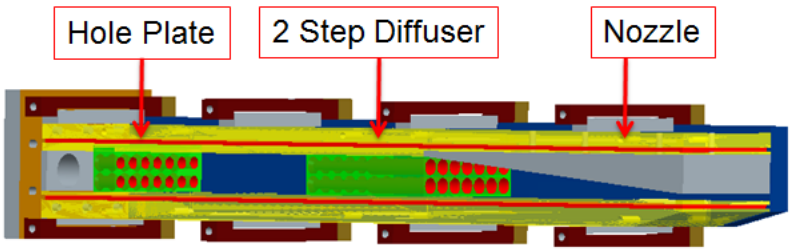

Figure 4.15 The first version of built-in components. The components are a hole plate used to distribute and accelerate the suspension, a two-step diffuser to accelerate the suspension and to generate turbulences at the end as well as a wedge-shaped nozzle.

The first version of the headbox including components geometrically similar to a real heabox caused several problems. Due to an available trial volume of about 40 litres a flow rate between 0.5 to 1 l/s could not be exceeded. Therefore the achievable flow velocities are quite low. For this reason the holes of the built-in components were partially clogged and an even distribution over the entire cross section could not be realized.

Because of that fact a second version of the headbox was designed. In this setup a single channel with flow conditions as well as turbulences similar to these in a diffuser block of a headbox is implemented.

After the inlet zone with a length of 50 mm the suspension passes a diffuser also shown in Figure 4.16. By means of the diffuser the suspension is accelerated, at the outlet of the diffuser the cross section of the channel changes abruptly from 25 x 15 up to a cross section of 35 x 25 mm. In this area turbulences are generated.

As shown in Figure 4.16 the diffuser consists of two parts, the conical inlet and the neck piece. The conical inlet represents a confuser with a length of 80 mm. To prevent detachment of the flow at the beginning of the neck piece, the inlet angle of

21 degree may not be exceeded. The neck piece of the diffuser is designed with a length of 120 *mm* and consists of two plates, the bottom and top plate. The benefit of this design is the possibility for installations of additional reductions of the cross section, as shown in Figure 4.16. To be able to control the flow conditions the diffuser is completely manufactured from transparent polycarbonate.

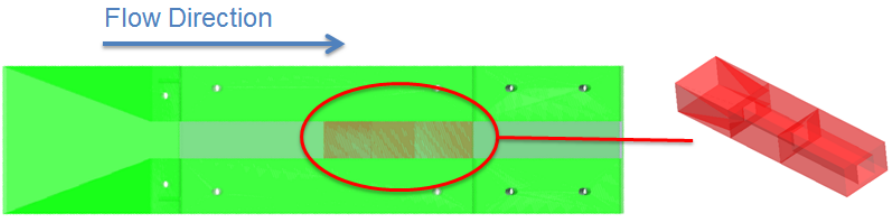

Figure 4.16 The diffuser is made of transparent polycarbonate. By means of the diffuser with an entire length of 260 *mm* the fibrous suspension is accelerated. To achieve flow velocities comparable to a real headbox a step diffuser (red block) can be integrated. At the outlet of the diffuser the cross section changes abruptly from 25 *x* 15 *mm* up to a cross section of 35 *x* 25 *mm* and turbulences are generated.

After the diffuser a nozzle is used to bring the fibrous suspension to machine speed. The cross section of the nozzle (in flow direction) with an entire length of 150 *mm* is 35 *x* 25 *mm* at the inlet and 100 *x* 5 *mm* at the outlet. Especially the cross section at the inlet of the jet influences the flow conditions to a great extent. With increasing cross section in the transition zone the possibility of wake spaces (in this area the flow velocity decreases down to zero) increases and re-flocculation may occur. The material used for the construction is a polyoxymethylene in opaque design. The schematic design of the nozzle is presented in Figure 4.17.

After the outlet (100 *x* 5 *mm*) of the headbox a bottom lip is followed by deckle plates (shown in Figure 4.13). The bottom lip as well as the deckle plates are a single unit, necessary to reduce boundary effects. Depending on these effects the deckle plates can be adjusted by means of a shaft.

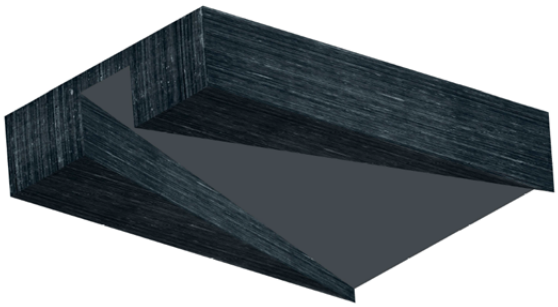

Figure 4.17 The nozzle with an entire length of 150 mm. The cross section of the nozzle is 35 x 25 mm at the inlet and 100 x 5 mm at the outlet.

Using the single channel diffuser clogging can be prevented. Furthermore the flow velocities are higher and turbulences can be generated more effectively. The achievable velocity profile in the headbox using the single channel diffuser is discussed in the next section.

Headbox Flow Velocity As mentioned above, by using the single channel diffuser the achievable flow velocities as well as turbulences are similar to conditions in a real headbox. In Figure 4.18 the velocity profile for different flow velocities along the headbox is shown. Above the diagram the headbox including the presented built-in components of the single channel diffuser is illustrated.
Position 1 represents the flow velocity in the flow loop of the laboratory device. Before entering the headbox the suspension is split into two pipes (position 2) with smaller diameters, the flow velocity remains almost constant. Because of a significant increase in cross section (100 x 30 mm) the velocity decreases at the beginning of the headbox (between position 2 and 3). Through the conical shape of the confuser the velocity (position 4) increases again. The maximum velocity of the flowing suspension (position 6) is reached by an additional reduction inserted into the neck piece of the diffuser. At the beginning of the wedge-shaped jet (also see Figure 4.17) the velocity decreases rapidly and increases again because of a slight reduction of the cross section up to wire speed. The maximum flow rate for the single diffuser pipe is about 0.65 l/s which means a maximum flow velocity (at position 6) of about 7 m/s. Due to a further increase of the flow rate an even distribution in cross direction of the nozzle can not be realized. For trials at higher flow rates an exchange of built-in components will be necessary.

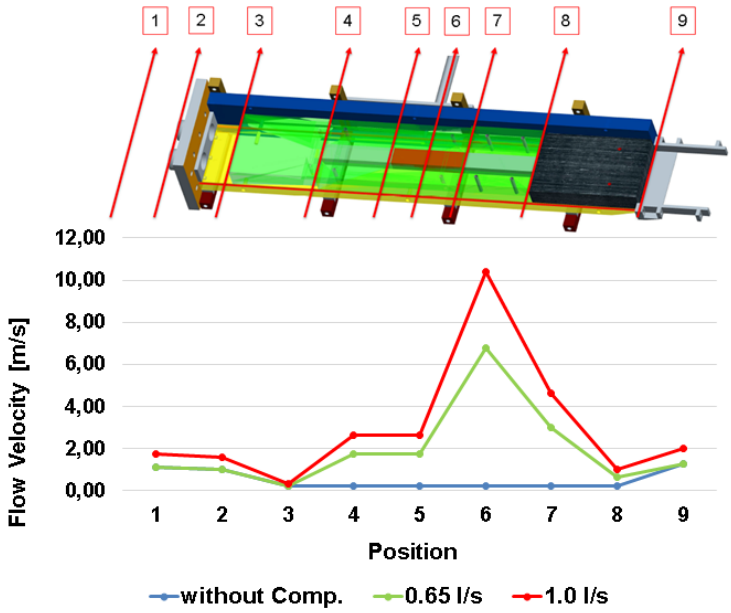

Figure 4.18 The velocity profile of the designed headbox. The green graph illustrates the velocity profile at a flow rate of 0.65 l/s. The profile reached at a maximum flow rate of 1 l/s is shown by the red graph. For comparison the blue graph illustrates the profile without any built-in components.

In summary the designed headbox enables the investigation of different built-in components allowing flow velocities close to that in a real headbox, but restricted to a single diffuser pipe. By the sealing it is possible to de-aerate the headbox before a new trial is started. For an even distribution of the fibrous suspension the maximum flow rate is at 0,65 l/s.

4.4.3 The Dewatering Device

In this section the dewatering section resembling a fourdrinier former including a vacuum-supported dewatering zone is presented. The main components are the frame, the rolls, the drainage wire, the vacuum-supported dewatering zone, the wire tension equipment, the wire cleaning equipment as well as the white water tank and the vacuum unit as shown in Figure 4.19.
In general the fibrous suspension leaves the headbox at the lip(100 x 5 mm) and comes in contact with the commercial drainage wire. Along the entire vacuum-supported dewatering zone the suspension is drained and a fibre web is formed. The resulting white water is gathered in white water tanks. After dewatering the fibre web is removed from the web by air blades. In the last step the wire is cleaned by water nozzles and dried by a second air blade to achieve constant conditions at the headbox.

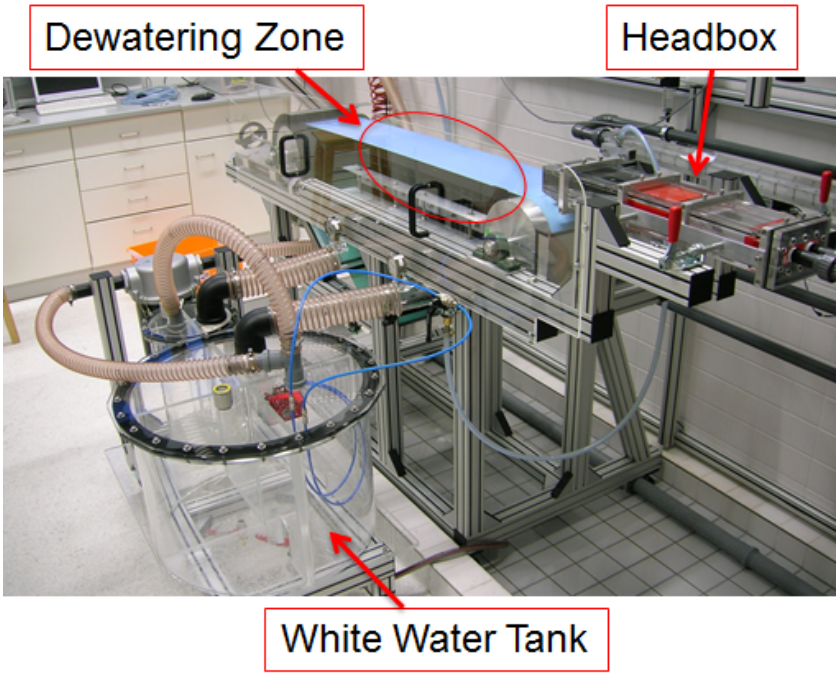

Figure 4.19 The dewatering device: the fibrous suspension is drained in the vacuum-supported dewatering zone. By means of an air blade the formed fibre web is removed after dewatering. The installed wire cleaning equipment cleans and dries the wire. The produced white water is stored in transparent white water tanks.

Basic elements of the dewatering device

In this section the frame and the rolls supporting the wire will be discussed. The dewatering device itself is 1750 *mm* long, 650 *mm* wide and 1047 *mm* high.

Frame The frame of the dewatering device consists of an aluminium profile system, manufactured by *Item*. The used profile system has a quadratic shaped cross section with an edge length of 60 *mm* and a wall thickness of 2 *mm*.
The profile as well as the cross section of the profile is shown in Figure 4.20.
The main advantages of the profile system are ease of handling and simple assembly, furthermore modifications and extensions are always possible with little effort.

Rolls of the Fourdrinier Former The rolls supporting the wire consist of three rolls:

- a breast roll
- a driving roll
- a small deflecting roll.

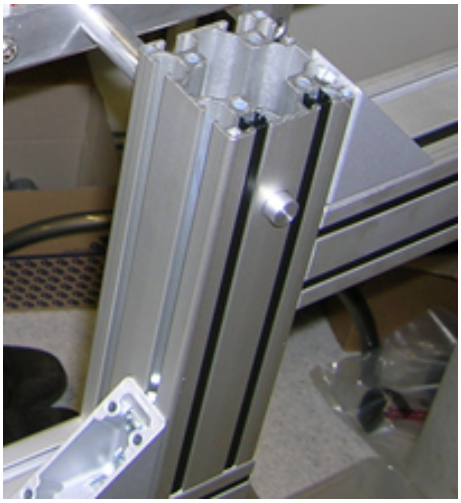

Figure 4.20 The aluminium profile used for the frame of the dewatering unit is presented. This quadratic shaped profile enables an easy assembly and handling.

Each of these rolls is made of aluminium with a width (including the shaft) of 430 mm. The breast roll is designed with a diameter of 200 mm. The deflector roll beneath the suction boxes has a diameter of 90 mm. The roll core of the driving roll, with the same diameter as the breast roll, is also made of aluminium. To achieve higher friction and to stabilize the wire in cross direction the cover of the driving roll is rubberised and convexly arched.
Each roll is equipped with single-row groove ball bearings with installed seals. The bearing bore diameter is 25 mm. In Figure 4.21 the positioning of the three rolls is shown.

Drainage Wire

The rolls of the dewatering device support a commercial drainage wire called $GeoflexX - Change$ used for super-calandered and light-weight-coated paper grades and is manufactured by $AndritzKufferath$. The $GeoflexX - Change$ is a seamless wire of 3300 mm in length and 250 mm in width, the design of the wire is shown in Figure 4.22

The tension of the wire during operation is depending on the width of the wire and specified by the manufacturer with the value of 8 kN/m. The elongation of the wire under tension is about 0.9 percent. The most important facts of this type of drainage wire are summarized in the following list.

- longitudinal threads: 64.0 $1/cm$
- transversal threads: 66.5 $1/cm$
- thickness: 0.87 mm

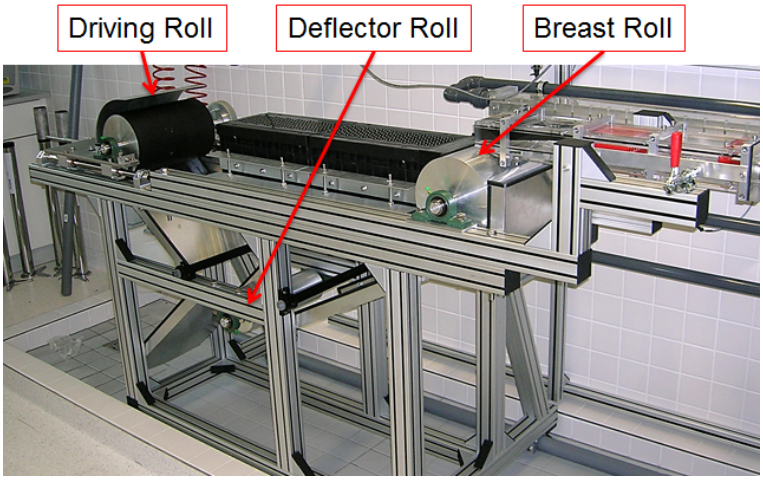

Figure 4.21 The rolls supporting the drainage wire. The dewatering device includes three rolls; the breast roll, the rubberised and convexly arched driving roll and the small deflector roll positioned beneath the suction boxes.

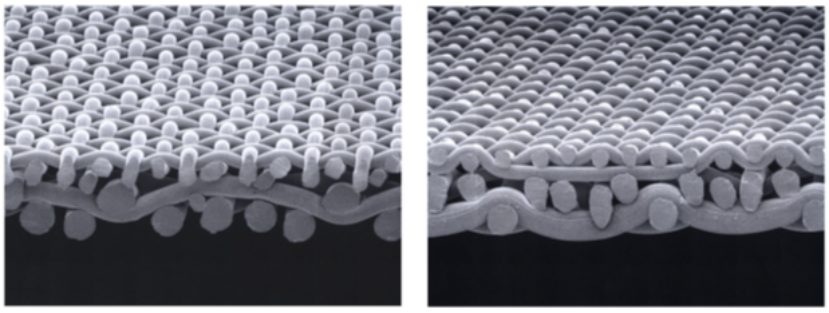

Figure 4.22 The structure of the $GeoflexX - Change$ a commercial drainage wire manufactured by $Andritz - Kufferath$. In a) the warp or longitudinal threads; b) the transversal direction of the drainage wire.

- air permeability: 1650 l/m^2s
- index for drainage: 32.0
- open volume: 57.7 %
- open area: 33.2 %

Wire Tensioning System, Wire Drive System and Wire Cleaning Equipment

In this section the wire tensioning system as well as the wire drive system are presented. These two components of the dewatering device are combined in one unit at the end of the wire section shown in Figure 4.19.

Wire tensioning equipment The wire tensioning system shown in Figure 4.23a consists of a clamping slide, a spindle as well as the driving roll. The clamping slide consists of a stainless steel plate which is fixed to a high-precision shaft (diameter of 20 mm) by four linear bearings. On top of the stainless steel plate the driving roll is fixed. By means of a spindle mounted beneath the plate the clamping slide can be moved along an entire length of 300 mm and the tension of the drainage wire can be adjusted. For the used drainage wire operative tension is the range of 1600 N (based on the specification of the manufacturer, 8 KN/m).
To be able adjust the running direction of the wire the position of the bearing houses of the driving roll can be adjusted by screws shown in Figure 4.23b.

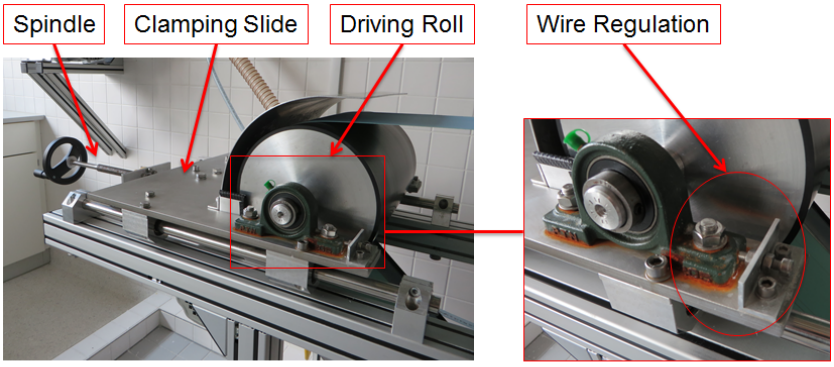

Figure 4.23 The wire tensioning system. a) The wire tension system includes components as the driving roll, the clamping slide as well as the spindle. To adjust the wire tension the clamping slide is moved using the spindle. b) Using the wire regulation the running direction of the wire is adjusted.

Wire Drive System The wire drive system used for the dewatering device consists of an electric motor, fixed beneath the stainless steel plate of the clamping slide. The

motor is connected to the driving roll by a toothed belt.
A three-phase asynchronous motor ($ACA90L-8$) with a power of 0.55 KW and a nominal speed of 690 rpm is used. Control and adjustment of the wire speed is realized by a frequency converter. The above mentioned toothed belt has a transmission ratio of 2.75. At this transmission ratio a maximum wire speed of about 150 m/min can be achieved based on the used asynchronous motor.

Wire Cleaning Equipment To guarantee homogeneous wire conditions a wire cleaning equipment is installed. This equipment consists of two air blades and a water nozzle unit placed in the area of the deflector roll, shown in Figure 4.24. During drainage a fibre mat is built up which has to be removed at the end of the wire section. Furthermore the wire has to be reconditioned before it arrives at the headbox again. In a first step the formed fibre mat is removed by an air blade. The designed air blade consists of a pipe with a single-row perforation with a length of 150 mm. The holes of the perforation are installed in intervals of 5 mm, the diameter of the holes is 1.5 mm. To remove the fibre web the air blade is supported with pressurized air (8 bar).
In a second step the wire is cleaned by an installed water nozzle unit placed above the deflecting roll. The water jets of the unit clean the wire before the deflecting roll and a water wedge building between roll and wire supports cleaning.
After the water nozzle unit a second air blade is installed primarily to dry the wire to provide uniform conditions at the headbox. This air blade is similar to the first one, only the length of the perforation is extended to 200 mm necessary to dry the entire width of the drainage wire.

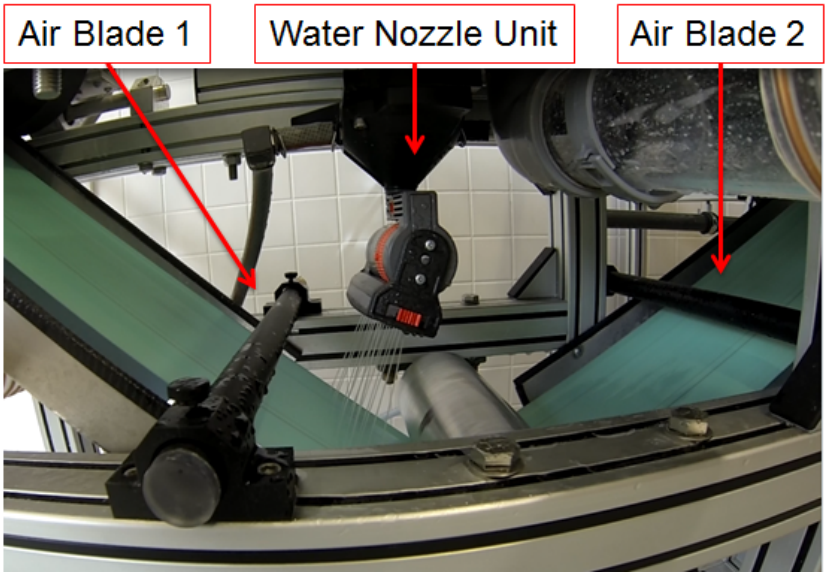

Figure 4.24 The wire cleaning unit. This equipment consists of two air blades with hole perforation and a water nozzle unit. By means of the cleaning equipment uniform wire conditions at the headbox can be achieved.

Dewatering Zone

Drainage of the fibrous suspension is achieved in a vacuum-supported dewatering zone. In general the dewatering zone, illustrated in Figure 4.25, consists of a forming board and two independent drainage boxes with an entire length of about 700 *mm*.
The forming board with a length of about 50 *mm*, fixed onto the frame of the first drainage box, is used as transition between the breast roll and the drainage box as well as for homogenization of the fibrous suspension. In contrary to forming boards used in commercial fourdrinier formers drainage is not desired.
As mentioned above, the vacuum-supported dewatering occurs via two independent drainage boxes. Basically the drainage boxes consist of a collecting basin covered with a perforated drainage plate (see Figure 4.25). Each of these boxes is 350 *mm* long and 175 *mm* wide and has a volume of about 6 litres. Drain pipes (one pipe installed at each box) carry the white water collected during drainage to white water tanks (see section 4.4.3). To guarantee uniform contact with the drainage wire over the entire area of the drainage boxes, height and tilting of each box can be adjusted separately. This adjustment is done using four threaded rods positioned at the edges of each box.

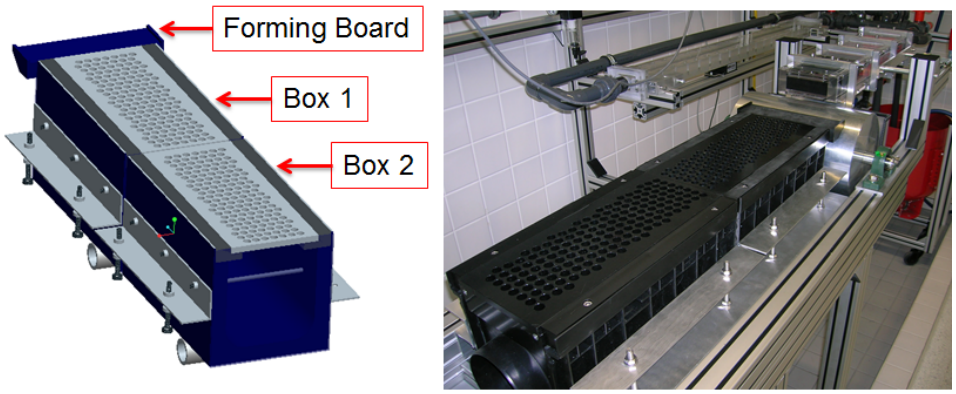

Figure 4.25 a) Schematic design of the drainage zone. The unit consists of a forming board and two drainage boxes. The boxes are covered by perforated drainage plates. b) The construction of the dewatering unit. Threaded rods are used for height adjustment of the boxes to achieve a uniform contact with the drainage wire.

The perforation of the plates is 110 *mm* wide and consists of 23 lines of holes, each line is shifted 4 *mm* compared to the neighbouring ones. This offset assures uniform drainage over the entire area of the built up fibre web. The diameter of the holes is 12 *mm*, the open drainage area is about 50 percent of the available area.
In order to adjust the vacuum level of the vacuum-supported drainage process an alternative drainage plate was designed. This plate, also shown in Figure 4.26b, consists of 10 slots with a length of about 100 *mm* and a width of 8 *mm*. The slotted plate can

be applied as an alternative to the hole plate and has about half the open drainage area. This reduction leads to an increase of the vacuum level.

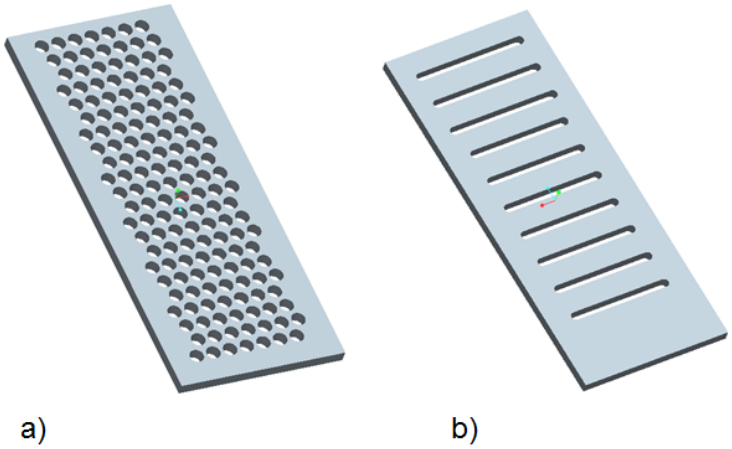

Figure 4.26 a) The perforated plate used in each drainage box. The shifting of the lines of holes assure an uniform drainage as the open area is equal in every line in length direction. b) An alternative drainage plate designed with slots. The open drainage area is reduced by 50 percent.

White Water Tank and Vacuum Unit

In this section the white water tanks, used to gather the filtrate produced during drainage, and the vacuum unit are discussed.

Vaccum Unit The vacuum unit consists of a vacuum fan with integrated frequency converter and a condensate separator used to prevent the fan from humidity coming from the white water tanks. The vacuum fan (2BH7-320 manufactured by Elmo-Rietschle) is designed as a side channel blower with a power of about 3 KW. At a nominal speed of 5000 rpm the vacuum fan produces a vacuum of -560 $mbar$ maximum, the achievable volume flow limit is 110 m^3/h. The performance curve of the vacuum fan is shown in Figure 4.27.

By means of the fan vacuum is applied on the drainage boxes. The fan is connected with both white water tanks by vacuum hoses (see also Figure 4.19). Each of the tanks is connected to one drainage box and the drainage process is supported by vacuum.
To adjust the level of vacuum each tank is equipped with bleed-off valves with a maximum volume flow of 40 m^3/h.
The maximum air flow through the holes of the perforated drainage plates at a volume flow of 100 m^3/h is about 0.8 m/s. A combination of a perforated hole plate in the first drainage box and the slot plate installed in the second box allow a flow velocity through the drainage plates of up to 1.09 m/s. In the presence of the used

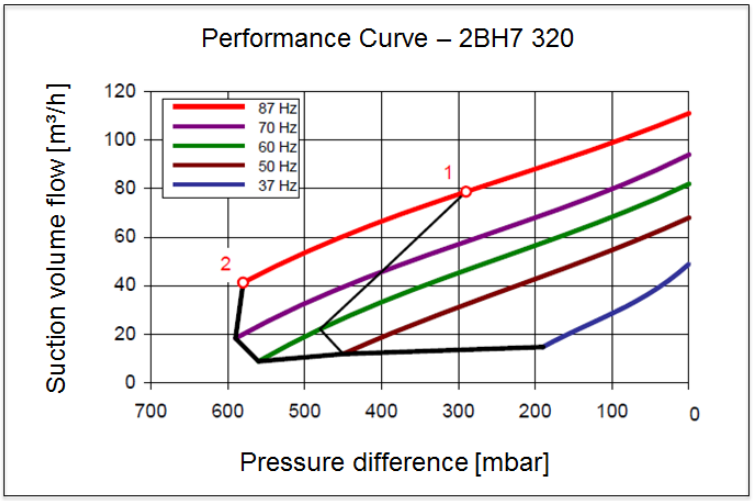

Figure 4.27 The performance curves of the vacuum fan At the ordinate axis the suction volume flow in m^3/h and on the abscissa the pressure difference in *mbar* are shown. At a frequency of 87 Hz the achievable suction volume flow is 110 m^3 [Rietschle, 2013].

drainage wire, with an open area of about 33 % and a fibre web the velocities increase considerably and thereby the resulting level of vacuum increases too.

White Water Tank The filtrate produced during the drainage is collected in the drainage boxes and carried to the white water tanks through pipes.
The white water tank connected to the first drainage box has a diameter of about 550 *mm* and a volume capacity of about 60 litres. Due to the fact that most of the water is drained in this segment this tank is designed much larger than the second one. The second tank with a diameter of about 230 *mm* collects about 25 litres and is connected to the second drainage box.
The white water collected from the first drainage box is used for the measurement of retention and drainage behaviour. To guarantee significant results the first white water tank is divided into two compartments. During the beginning of the dewatering process the collected white water is carried into the first compartment of the tank, as soon as constant drainage conditions are achieved the white water is directed into the second part of the tank. This redirection is done by a deflection shown in Figure 4.28b. Details about the measurement as well as the measurement procedure are discussed in section 5.1.

4.5 Process Control System

In this section the process control system of the novel laboratory device is presented. During operation of the measurement system several motors and valves have to be

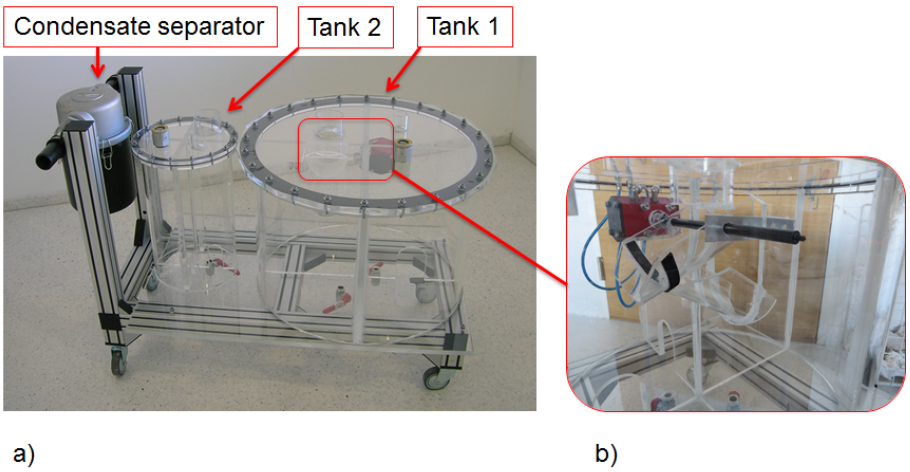

Figure 4.28 a) The white water tanks of the dewatering device. Each drainage box is connected to one of these tanks. For vacuum-supported dewatering the tanks are supplied with vacuum generated by one vacuum pump. The condensate separator is used to protect the vacuum fan from fluid particles of the white water. b) The equipment for sampling the white water. As soon as constant drainage conditions are achieved the white water is directed in a separate compartment.

adjusted in close succession. For an easier operation the control elements are linked via a control platform using a process control software designed and implemented in *Labview*®.
In general the process control system starts and stops the main drives and pumps, settings like the operating frequency have to be adjusted at the actuator directly. The controllable actuators are the frequency converters of the

- centrifugal pump
- wire drive
- vacuum fan

Also controlled by the system are the two dosing pumps (see section 4.2.1), the three way ball valve necessary to switch between the two measuring units (see section 4.1) and the ball valve at the pulp chest used to switch between circulation and efflux of the suspension. Another controllable element is the pneumatic pressure switch installed in the first white water tank (see also 4.4.3) and necessary for a controlled sampling of the white water.
By means of the control platform, shown in Figure 4.29, the incoming signals of the operating elements are processed. The data collector sends the prepared signal to the computer software and collects data from pressure transducers integrated in the flow loop and the drainage zone. The data is are plotted in a diagram over time based on a special software. Control and power supply of the pneumatic pressure switches is realized by way valve control units (see also Figure 4.29).

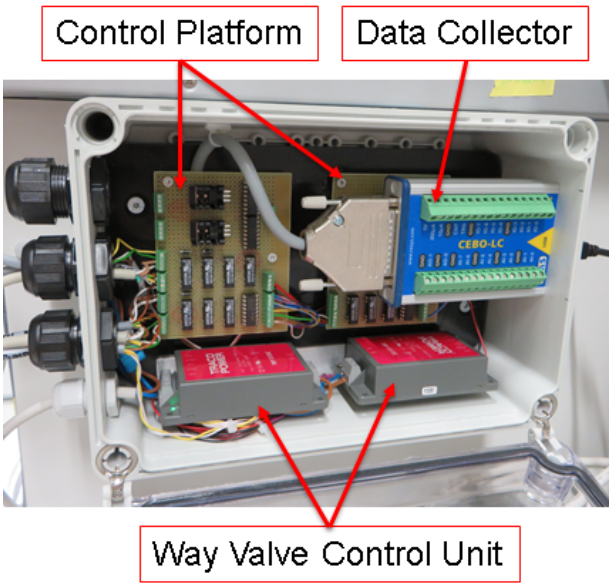

Figure 4.29 The control platform of the process control system. This platform is the link between the operating elements of the laboratory device and the computer software. The integrated data collector records the pipe pressure of the flow loop and the dewatering zone. The way valve control unit controls the pneumatic pressure switches.

The process is controlled by a purpose-built software based on *Labview*®. The user interface of the process control system is shown in Figure 4.30. The background of the interface shows a design drawing of the laboratory device and allowing a better overview of controllable elements. Each of the controllable elements is linked with its own on- and off switch, a red or green button signalises the elements operating state. For a better overview a separate control panel signalises all switched on elements. In the operating area of the white water tank and the sampling device an additional button to set the sampling time is integrated.
Additional to this user interface there are two different process sequences available. These sequences are used for automated measurements with the flocculation unit as well as the dewatering unit.

In summary the process control system enables an easier operation of the laboratory device, facilitates sampling and allows a safe operation.

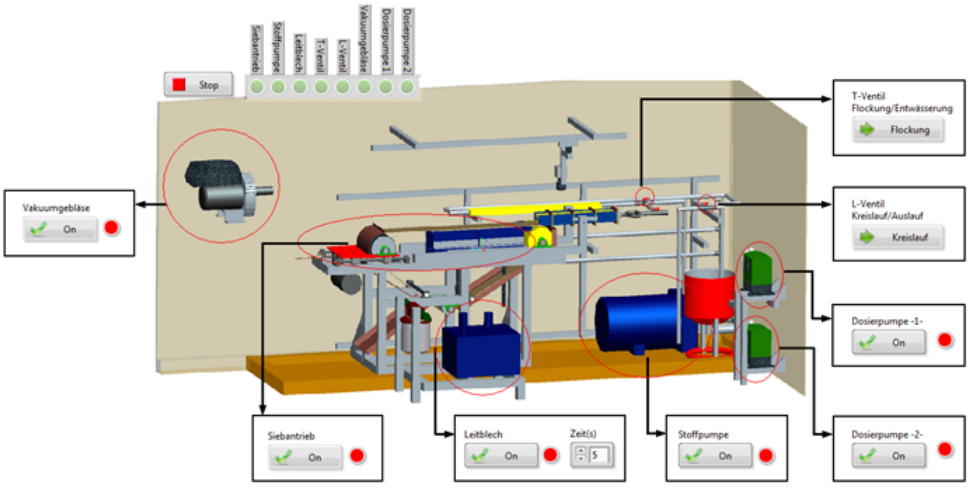

Figure 4.30 The user interface of the process control system. The design drawing illustrates the whole laboratory device and gives an overview of the controllable elements. Each element is linked with an on- and off switch as well as an coloured button which signalises the operating state. The sampling unit in the white water tank is additionally equipped with a timing unit.

Chapter 5

Measurement and Validation

In this chapter the measurement procedure of the novel laboratory device is presented. The measuring sequences as well as the operating conditions and two sequences for an automated measurement started by the process control system (see also section 4.5) are described. The reproducibility as well as the validation of the laboratory device are discussed.

5.1 Measurement Process

The measurement procedure of retention, dewatering and flocculation occurs in two steps starting with the measurement of flocculation followed by a combined measurement of retention and dewatering (see also section 4.1).
A suspension volume of only 40 litres is necessary for the whole procedure. At first the fibrous suspension has to be prepared, which consists of fibres and fillers depending on the furnish recipe. The suspension is prepared at headbox consistency before filling the chest, preparation is done separately for each individual trial. In the following sections the two steps of the measurement sequence are presented.

5.1.1 Measurment of Flocculation

All settings of the process parameters except the frequency of the centrifugal pump are implemented in the process control system. For image acquisition settings have to be adjusted in the software (Epix XCAP, also see section 4.3.3), the standard values were already discussed in section 4.3.4 and are summarized in Table 5.1.

As mentioned above the first step of the procedure is the filling of the chest. To prevent sedimentation of the fibrous suspension the integrated impeller is started.
If chemical additives are dosed the dosing quantity has to be adjusted using the user interface of the dosing pump. In the next step the centrifugal pump is started. To

AOI Height	AOI Width	Resolution	Exposure	Frame Period
700 pix	1280 pix	35 $\mu/pixelm$	0,400 msec	21 msec

Table 5.1 Settings for Image Acquisition

prevent the circulation of air into the chest the L- ball valve (loop/efflux) has to be switched to efflux until the pipe is filled with suspension. The required flow velocity for the measurement of flocculation is 1 l/s and has to be adjusted directly at the frequency controller at the converter. As soon as the system runs stable the dosing pumps (if dosing is requested) are switched on. Three seconds after starting the dosage the flowing suspension is redirected to the efflux (L- ball valve). After six seconds image acquisition is started.
After image acquisition the dosing pumps are stopped and after additional six seconds the flowing suspension is redirected from the efflux back into the chest.
For an easier operation and to ensure stable measuring conditions the process sequence of the flocculation measurement can be controlled automatically by the process control system. The corresponding user interface is shown in Figure 5.1.

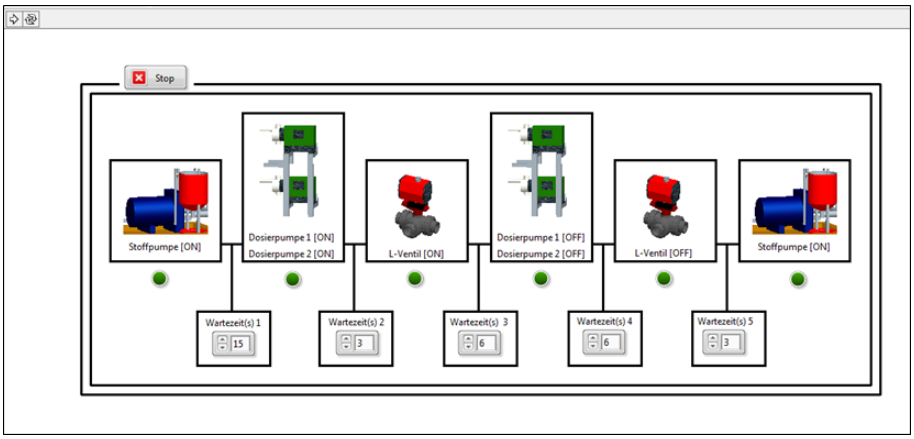

Figure 5.1 The user interface for an automated measurement of flocculation. The automated sequence is divided into six steps and starts with the activation of the centrifugal pump, the dosing pumps, the L- ball valve followed by the deactivation of the dosing pump, the L- ball valve and the centrifugal pump. The time between each step of the sequence can be adjusted in one-second intervals via additional control panels located between the symbols .

In the user interface all the individual aggregates for the measurement of flocculation are implemented. The sequence starts with the activation of the centrifugal pump, followed by the dosing pump and the L- ball valve. After the ball valve directs the suspension to the efflux image acquisition is started. In the next step the switched on aggregates are deactivated starting with the dosing pump followed by the L-ball valve and the centrifugal pump.

The time period between each step of the sequence can be adjusted in one-second intervals by additional control panels located between the symbols. Beneath each symbol a green coloured button signalizes the course of the sequence.

5.1.2 Measurement of Retention and Dewatering

After the measurement of flocculation the measurement of dewatering and retention has to be prepared. Before this measurement can be started different settings have to be modified. The flow velocity is lowered to 0.5 l/s and therefore the dosed amount of additives has to be adapted too. Because of the same cross section of both the pipe diameter and the lip of the headbox the flow velocity of the headbox jet is 0.5 l/s. At this velocity a wire speed of 60 m/min is required to achieve a jet to wire ratio of 1:1. The wire speed has to be adjusted directly at the frequency converter of the wire drive. Based on these settings and at a given furnish consistency of 1% the grammage of the produced fibre web is 50 g/m^2. Using the wire tensioning unit (see section 4.4.3) the used drainage wire has to be tensioned with about 1600 N.
To prevent trapped air in the headbox it has to be vented as described in section 4.4.1. In the last step of preparation the wire cleaning equipment has to be activated by hand.
When all preparations are finished the measurement of retention and dewatering can be started. The centrifugal pump, the wire drive as well as the vacuum fan have to be switched on. After a time period of about 15 seconds these aggregates operate at constant conditions and the flowing suspension can be directed from the circulation to the dewatering unit by activating the T- ball valve. After directing the fibrous suspension through the headbox it passes the wire where vacuum-supported drainage starts. Subsequently the dosing units have to be activated. The addition of chemical additives influences the drainage process, to restore constant dewatering conditions a period of 10 seconds after starting dosage is required. In the next step a sample of the collected white water has to be gathered, therefore the baffle (installed in the first white water tank) is switched. The standard period for sampling is specified with 8 seconds but can be varied via the user interface of the process control system. After collection the measurement is finished and the dewatering unit can be switched off. First the dosing units are deactivated. To carry the remaining additives out of the system the suspension is directed to the dewatering unit for another 5 seconds. Afterwards the T- ball valve turned back and the circulation pump is switched off. To remove the remaining fibre web from the drainage wire a few more seconds are required before shutting down the wire drive and the vacuum fan. Finally the wire cleaning equipment is deactivated.

To evaluate drainage behaviour as well as retention the collected white water has to be transfered into a measuring beaker and weighed. The measured weight rep-

resents the drainage capacity (weight over a set defined time). The measurement of retention is done by a gravimetric analysis in the laboratory.
Similar to the measurement of flocculation the procedure for the measurement of dewatering and retention can be automated using a second side programm of the process control system. The user interface (see Figure 5.2) of this program is similar to that for the measurement of flocculation.

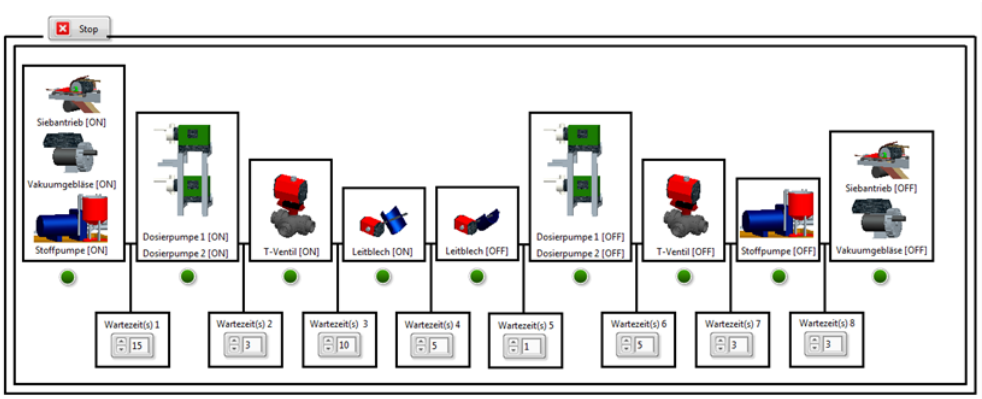

Figure 5.2 The user interface of the program for an automatic control of the dewatering unit. The control sequence can be divided into nine steps. Between each step the duration of the step can be adjusted in one-second intervals. Sampling of white water takes place between the forth and fifth step.

Similar to the user interface for automated control of the flocculation unit the different steps of the control sequence are represented by symbols. Each of the symbols represents the involved aggregates. Additional control panels between each step allow the adjustment of the duration in one-second intervals.
The user interface shows the conditions for a standard measurement of dewatering and retention. In the first step the wire drive, the vacuum fan as well as the circulation pump are activated. After 15 seconds these aggregates operate at constant conditions, therefore the dosing of chemical additives are switched on. The T- ball valve directs the flowing suspension to the dewatering unit. After a time period of ten seconds the white water is collected separately for 8 seconds. After this step the aggregates are successively switched off and the measurement process is finished.

5.2 Reproducibility

In this section the evaluation of the reproducibility of the laboratory device especially the dewatering unit is presented. The evaluation regarding the flocculation unit has been discussed in Eckhart et al. [2013].

5.2.1 Materials

Before the results of the validation are discussed the used fibrous suspension is presented. In general the stock consistency of the suspension is 1%. The composition is 75% fibre and 25% fillers.

The pulp used for the validation was a bleached and refined chemical pulp consisting of a mixture of 90% hardwood (eucalyptus) and 10% softwood (90% spruce, 10% pine). The used pulp was obtained at a consistency of 4.0-4.5%.
The used filler is a ground calcium carbonate (GCC) delivered as slurry with an anionic dispersant and a dry solid content is about 65%. About 60% of the filler particles had a particle size less than 2 micrometers in diameter.
All trials for the evaluation of the reproducibility are performed with the described pulp stored in a mixing chest with a volume of 250 litres. In the mixing chest the pulp was diluted with tap water to a consistency of 0.75%. After dilution the fillers (25% refering to solid content) are added to achieve a consistency of 1%. The standard pulp characteristics as pH- value, conductivity and degree of beating are summarized in Table 5.2.

Consistency, %	1 ± 0.05
Hardwood : Softwood	90 : 10
Temperature, °C	23 - 25
Degree of beating, °SR	24
Conductivity, $\mu S/cm$	534
pH- value	6.95
Zeta- Potential mV	-18.5
Filler content (of total stock consistency), %	25

Table 5.2 Reproducibility - Standard pulp characteristics

For each trial a volume of 40 litres was taken from the mixing chest and filled into the pulp chest of the laboratory device, where the filler was added to reach headbox consistency.
All trials were done within a few hours after dilution to a consistency of 0.75%.

For the reproducibility of the dewatering unit trials with and without a retention aid were carried out. As a retention aid a cationic polyacrylamide called *Percol* 540 delivered by $BASF$ was used. This single-component system has a high molecular weight and low charge density.
The dosage of the retention aid was performed at the first point of dosage after the centrifugal pump, the added concentration was fixed to 100 ppm.

5.2.2 Reproducibility of the dewatering unit

As mentioned before reproducibility was investigated with and without a single-component system in four trials each. For the measurement of retention and dewatering the automated procedure as presented in section 5.1.2 was applied.

The results for the evaluation of the reproducibility of the drainage capacity measurements are presented in Figure 5.3. In these diagrams the ordinate axis shows the drainage capacity in g and in the abscissa the repetition of the measurements is shown.

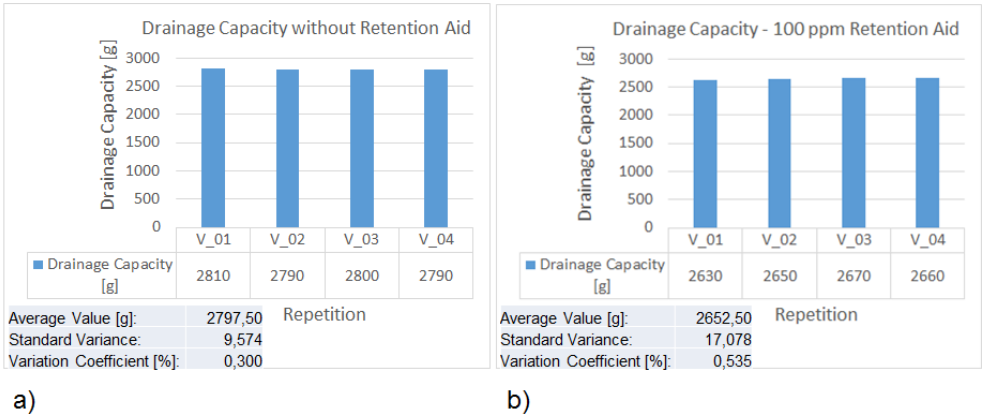

Figure 5.3 Evaluation of the reproducibility of drainage capacity. a) Drainage capacity without retention aids. The value of the variation coefficient is 0.003 %. b) The results of drainage capacity measured when dosing a retention aid. The variation coefficient is 0.006 %. In summary the measurement of drainage capacity show a high level of reproducibility.

In Figure 5.3a the drainage capacity without addition of a retention aid is presented. The measured drainage capacity of the collected white water during a time period of exactly five seconds of four different trials is within the range of 2790 g and 2810 g, a standard deviation of 9,57 and a variation coefficient of 0.003% were reached.
Figure 5.3b shows the drainage capacity of the fibrous suspension when adding the retention aid. The measured drainage capacity is within the range of 2630 g and 2670 g, a standard deviation of 17.07 and a variation coefficient of 0.006% were achieved by the setup.
Comparing these two diagrams the influence of the used retention system becomes evident. Using the cationic polyacrylamide the drainage capacity is reduced by the amount of 150 g.
In summary these results regarding drainage capacity in four trials with and without additives show a high reproducibility of the measurement of dewatering capacity

performed in the dewatering unit.

After the measurement of dewatering overall retention and ash retention of the collected white water were evaluated by gravimetric analysis in the laboratory. First the results of the total retention with and without adding retention aids are presented and discussed. The results are illustrated in Figure 5.4. In these diagrams the ordinate axis shows the total retention in percent.

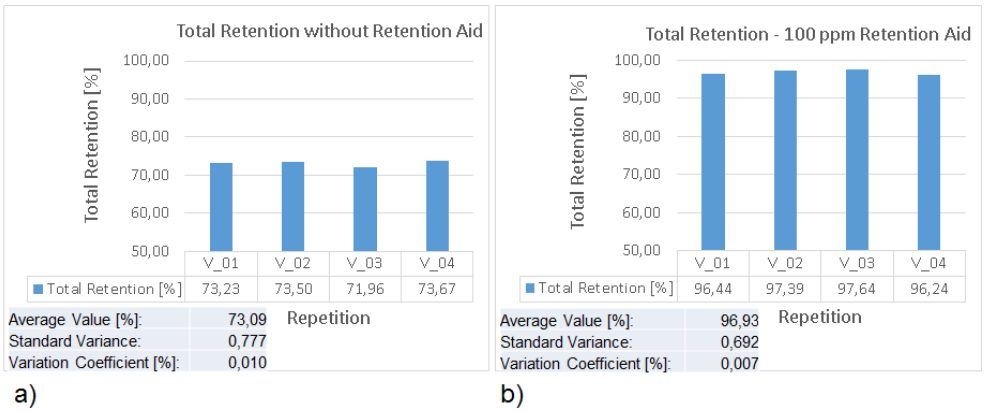

Figure 5.4 Evaluation of the reproducibility of the measurement of retention. a) Total Retention without additives. The average value of four trials is 73% and the variation coefficient is 0.01%. b) Total Retention when adding retention aids. The average retention value increases to 96%, a variation coefficient of 0.007 is reached.

In Figure 5.4a the total retention without adding chemicals is presented. The average value of the total retention at the given suspension is about 73% a standard deviation of 0.77 and a variation coefficient of 0.01% are reached.
Figure 5.4b shows the total retention of the suspension when adding the retention aid. The results of the measurement show an average retention value of about 97% percent, a standard deviation of 0.69 and a variation coefficient of 0.007%.
By comparising these diagrams the influence of the used retention aid is clearly evident. Due to low turbulences in the drainage zone the retention in general attains a high level.
In summary the results of total retention with and without adding retention aids shows a high reproducibility achieved in the dewatering unit.

Finally the ash retention was evaluated and plotted in diagrams presented in Figure 5.5. In the diagrams the ordinate axis shows the ash retention in percent. For the analysis of ash retention the samples were ashed at 575°C.

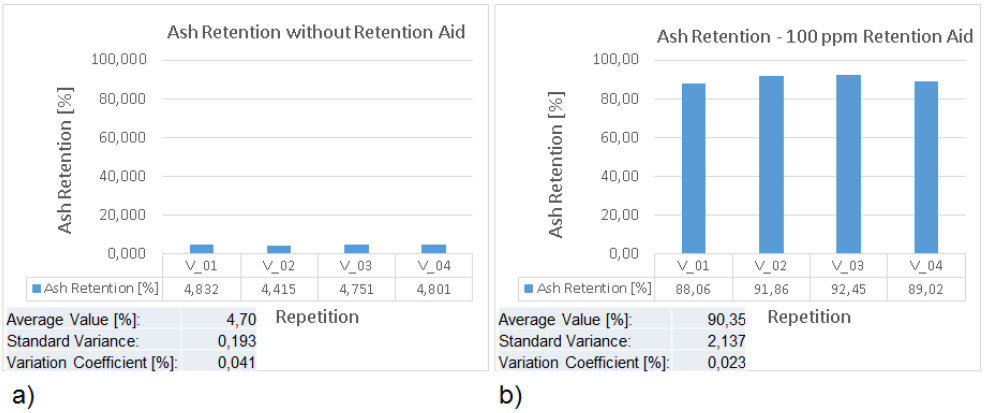

Figure 5.5 Reproducibility of the measurement of ash-retention. a) The results of ash-retention of the suspension without adding retention aids. An average value of ash-retention of 4.7% and a variation coefficient of 0.04% are reached. b) Ash-retention reached using retention aids. An average value of about 90% and a variation coefficient of 0.02% are reached.

In Figure 5.5a the ash retention without a retention aid is presented. The average ash retention is about 4.7%, the standard deviation of the four trials reaches a value of 0.19, the variation coefficient of these trials 0.04%.
In Figure 5.5b the ash retention with 100 *ppm* retention aid is presented. In general ash retention increases dramatically. The average value of these four trials is about 90% and the standard deviation reaches a level of 2.13, a variation coefficient of 0.02% was calculated.
In summary the results for the measurment of dewatering, total retention and ash retention show a low standard deviation as well as a remarkably low variation coefficient proving a high reproducibility of the whole evaluation procedure.

5.3 Validation against the widely used DFR-05

In this section the validation of the dewatering unit against the widely used *Muetek* DFR-05 (see section 3.1.2) for the measurement of retention are presented. Additional to retention and drainage capacity measured with DFR-05 the time for dewatering measured via Schopper-Riegler is used in the comparison.
As mentioned in section 3.1.2 for each measurement a sample volume of 1 litre is necessary. During dewatering the collected white water is separated into pre-filtrate and main filtrate. For the evaluation the collected main filtrate is used. The settings applied for the measurement are summarized in the Table 5.3.

After filling the beaker the measuring program of the DFR-05 starts. In the beginning the suspension at a concentration of 1 percent is agitated for 20 seconds at an impeller speed of 700 *rpm*. In the next step the additives are dosed and the impeller

Pre - Filtrate	20 sec or 40 g
Filatrate	200 sec or 200 g
Agitation Time and Speed before Dosing	20 sec at 700 rpm
Agitation Time and Speed after Dosing	5 sec at 800 rpm
Agitation while Filtration	400 rpm
Screen	mesh 60 (equal to Schopper-Riegler)

Table 5.3 Settings for the measurement using the $Muetek - DFR - 05$

speed increases up to 800 rpm for 5 seconds introduce high shear rates. After this step the filtration process starts and the conical impeller uncovers the screen. The impeller speed during filtration is set at 400 rpm. After 200g of the main filtrate the measurement is finished and the collected filtrate is analysed in the laboratory.
In comparison to the DFR-05 the same measurements are performed on the novel dewatering unit. The settings used for each trial are discussed in section 5.1 and summarized in Table 5.4.

Flow Velocity in the Pipe	0.5 l/s
Jet to Wire Ratio	1:1
Wire speed	60 [m/min]
Points of Dosage	First/Second point after the circulation pump
Time for Collecting the Filtrate	5 seconds

Table 5.4 Settings for using the dewatering device

5.3.1 Materials

The fibrous suspension used for these trials consists of 60% pulp and 40% fillers. A bleached and refined chemical pulp with a mixture of 70% softwood (90% spruce, 10% pine) and 30% hardwood (eucalyptus) was used. The pulp was prepared in a mixing chest with a volume of 250 litres and diluted with tap water to a consistency of 0.6%. As filler a ground calcium carbonate (GCC, 60\$ $< 2\mu m$) deliverd as slurry with a consistency of about 65% was used. For the trials performed with the dewatering unit the pulp suspension was filled into the pulp chest of the device where the filler was added to achieve a consistency of 1%. Different suspension characteristics as conductivity, pH- value and zeta potential are summarized in Table 5.5.

To the furnish a microparticle system was added. The system consists of a cationic polyacrylamide with high molecular weight and low charge density as well as a bentonite as micro particle. To investigate the influence of the added retention system trials with different concentrations were carried out. The concentration of bentonite was kept constant at 3000 ppm, the concentration of the polymer was varied in intervals of 150 ppm from 150-450 ppm.

Consistency, %	1 ± 0.05
Hardwood : Softwood	30 : 70
Temperature, °C	23 - 25
Degree of beating, °SR	18.5 ± 0.5
Conductivity, $\mu S/cm$	505 ± 10
pH- value	7.4 ± 0.3
Zeta- Potential mV	-17.85 ± 0.3
Filler content (of total stock consistency), %	40

Table 5.5 Suspension Characteristics used for the Validation

5.3.2 Results

In the following diagram the results of the drainage capacity obtained with the novel device are presented. The results of the measurement of dewatering time compared to Schopper-Riegler are also discussed. Additionally the results of the measurement of total retention and ash retention are compared between the novel device and the commercial tester DFR-05 are presented and discussed.

Figure 5.6 shows the results of the drainage measurement via the dewatering unit as well as the Schopper-Riegler device. As mentioned before these results are not directly comparable because of different units used, still the comparison of trends is valid.
In Figure 5.6a the influence of the retention system on the drainage time of the fibrous suspension measured via Schopper-Riegler is presented. The ordinate axis shows the drainage time in seconds and the abscissa shows the concentration of the polymer. As mentioned above the concentration of bentonite is kept constant at 3000 ppm. These results show a decrease in drainage time (increased drainage) due to an increase of the concentration of the cationic polyacrylamide.

Figure 5.6b shows the drainage capacity measured using the novel dewatering unit in g. The results of these measurements cleary show the expected correlation between the retention aid and drainage. Between the concentrations of 150 ppm and 300 ppm the major increase occurred, between a concentration of 300 and 450 ppm the drainage capacity of the suspension hardly increases. The results of Schopper-Riegler in comparison show an approximately constant decrease in drainage time. This results might be due to a measurement without any shear during dewatering.

In Figure 5.7 a comparison of total retention as well as ash retention of the dewatering unit to the commercial DFR-05 is shown. In the presented diagrams the ordinate axis shows the retention in percent and the abscissa shows the concentration of the used polymer.

Figure 5.7a shows the comparison of the results for total retention. Generally the

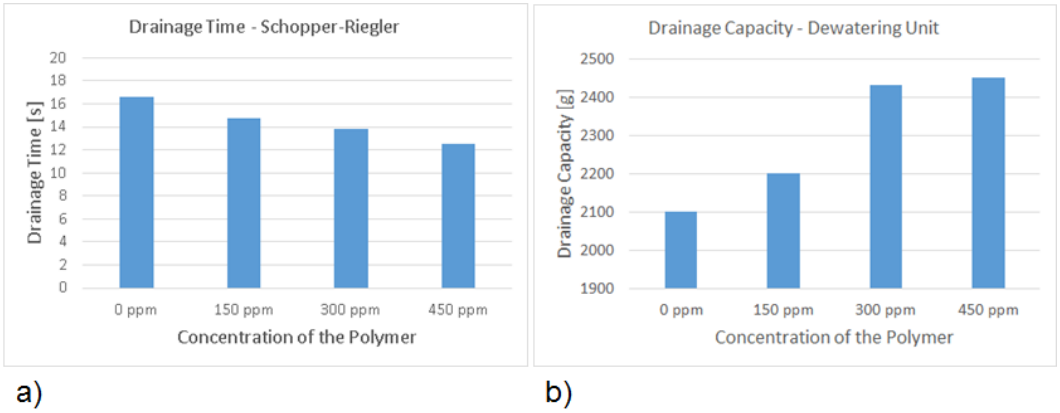

Figure 5.6 a) The drainage time (Schopper-Riegler) of the fibrous suspension. These results clearly show a decreasing drainage time due to an increasing amount of polymer. b) The drainage capacity in g measured via the dewatering unit is presented. Because of an increasing polymer concentration the drainage capacity of the fibrous suspension increases too.

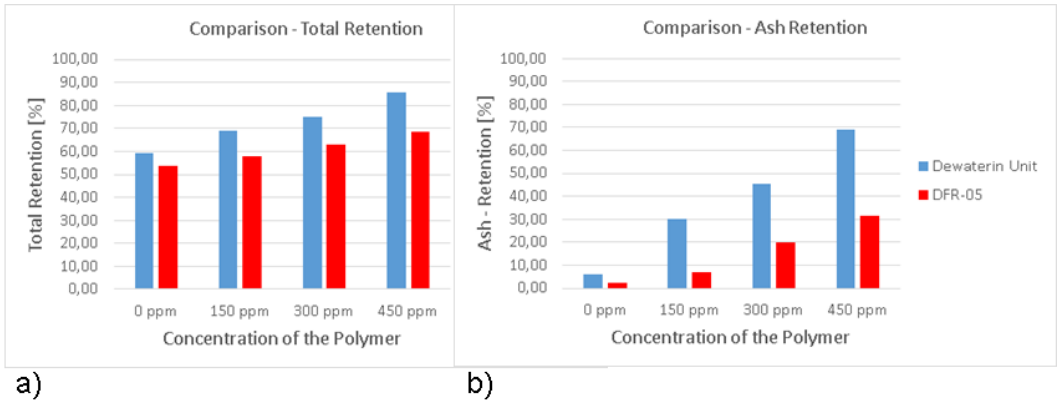

Figure 5.7 A comparison of retention achieved with the novel device and the commercial tester DFR-05. The results of total retention in a) and of ash retention in b) are quite different. Due to lower turbulences during dewatering the retention achieved with the novel device is clearly higher at each point. Between 0 ppm and 450 ppm the gradient for retention is steeper using the dewatering device.

resulats obtained with the dewatering unit are higher than those of the commercial DFR-05 due to a higher level of shear rates during dewatering. Both measurement systems show a very similar trend due to increased polymer addition level.
Figure 5.7b shows the ash retention obtained by these measurement systems. The results of ash retention and total retention are clearly similar.
Despite the similar trend the gradient for total retention as well as for ash retention achieved via the dewatering unit is clearly steeper. The increase of total retention between 0 ppm and 450 ppm is about 25% measured via the dewatering unit and about 15% via the DFR-05. The gradient for ash-retention achieved via the dewatering device is even steeper. This could be seen as an indication that the measurement of retention via the novel device is more sensitive.

5.4 Summary

The evaluation of drainage capacity, total retention as well as ash-retention investigated with and without dosage of retention aids shows low standard deviations and variation coefficients. The results obtained with the dewatering unit are thus highly reproducible. The comparison between the results gathered with the developed dewatering unit and the commercial method clearly show an higher level of retention achieved via the novel device. In general it can be said the trends are very similar. The comparison of the gradients of retention between two devices shows a more sensitive measurement achievable with the dewatering unit.

Chapter 6

Applications with different representative stock compositions

This chapter discusses exemplary applications of the developed laboratory device. The main goal for the developed laboratory device is the simultaneous measurement of retention, dewatering and flocculation under industry-oriented conditions. Therefore three different stock compositions provided by several mills are evaluated. To evaluate the sensitivity of the laboratory device the amount of added retention aids are varied in the range of industrial relevance.
The stock for these trials was prepared as close as possible to the industrial applications (sample collection after the mixing chest, dilution to headbox consistency with process water), furthermore the used retention systems are also identical to those used in the mills.
For each sample the parameters dewatering, retention and flocculation are analysed and discussed. The settings for these measurements are adjusted as described in the sections before (see section 5.1).

6.1 Results obtained for a Fine Paper furnish

The following results of the simultaneous measurement of a suspension are based on a fine paper stock composition. The suspension diluted with process water to a consistency of about 1%, consists of 70% pulp and 30% fillers (GCC). As retention aid a microparticle system consisting of a cationic polyacrylamide (high molecular weight and low charge density) as well as bentonite (microparticle) was used. For the measurement the amount of added microparticle was kept constant at a level of 1750 ppm. The cationic polymer was added in the concentration of 100, 150 and 200 ppm. In these trials the cationic polymer was dosed before the centrifugal pump followed

by the bentonite.

Figure 6.2 shows the results of the image analysis regarding the flocculation of the flowing suspension. For the evaluation of the flocculation behaviour the red vertical line marks a structure size (flocs) larger than 2 *mm*.

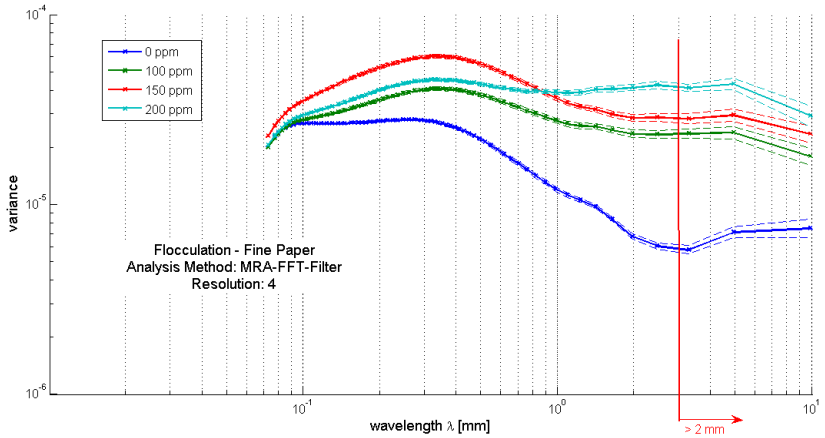

Figure 6.1 The influence of polymer concentration on the flocculation behaviour of the given suspension. The retention system clearly increases flocculation with increasing effect of increased polyacrylamide dosage.

The analysis of the fine paper stock composition shows a clear increase in flocculation. With increasing amount of polymer the intensity of detected structures increases.

In Figure 6.2 the drainage capacity as well as the total retention and ash retention are presented.
The drainage capacity (Figure 6.2a) of the sample measured without the retention system reveals a capacity of 1700 *g* during 5 seconds. Due to the addition of the retention system the drainage capacity decreases at a concentration of 100 *ppm* but increases again at higher dosage levels. The reason for this reduction is probably a higher retention of fine material due to additon of the retention system. The most substantial increase of the drainage capacity is obtained between 100 and 150 *ppm*. The drainage capacity seems to increase again due to stronger flocculation.
Figure 6.2b shows the total retention and ash retention of the fine paper suspension. Without additives a total retention of about 55 % is achieved, the ash retention is below 10%. The strongest increase of total retention (by 20%) is achieved due to adding retention aid and is almost exclusively related to an increasing ash retention. At a concentration of 200 *ppm* a total retention of 82% as well as a ash retention of about 68% is reached.

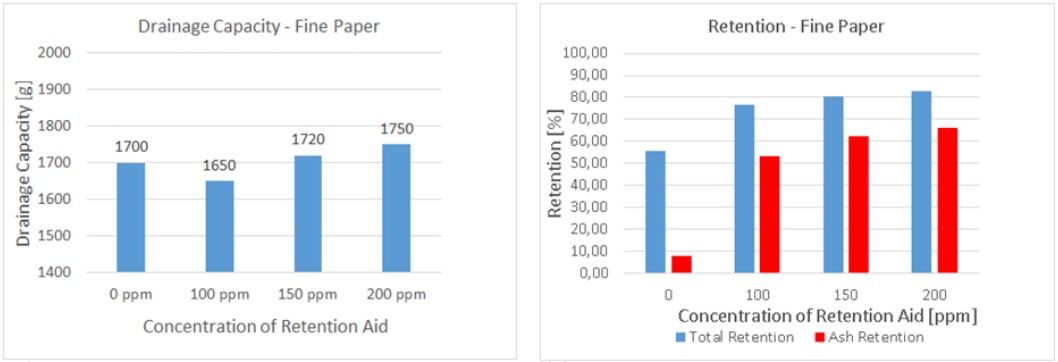

Figure 6.2 a) The drainage capacity of the fine paper stock composition. Without additives the drainage capacity is 1700 g . With addition of the microparticle system capacity decreases at 100 ppm but is improved again with increasing concentration.
b) The evaluated retention of the given suspension. With increasing concentration of the added polymer both total retentin and ash retention are improved. At a concentration of 200 ppm a total retention of about 80% and a ash retention of about 68% is eached.

Comparing these three measurements the interrelation between retention, dewatering and flocculation is visible to some extent. An increasing amount of the retention system improves total retention and ash retention but also increases flocculation. With the clear increase in retention due to the addition of the retention system (100 ppm) the drainage capacity decreases. This is most probably related to a reduction of the free drainage area because the now retained fillers and fines are included into the fibre network. At higher concentrations of polymer the drainage capacity increases again due to a stronger flocculation.

6.1.1 Results obtained for a SC-A paper furnish

In this section a stock sample for SC-A paper is analysed. The suspension consists of 67% pulp including chemical pulp, de-inked pulp and mechanical pulp and 33% fillers including calcined clay and precipitated calcium carbonate (PCC). The pulp was delivered with a consistency of about 5 percent and was diluted for the measurement with mill water to headbox consistency (1%).
To improve retention a dual polymer system with two cationic polymers is used. The cationic polyacrylamide (PAM) with high molecular weight and low charge density is added first in concentrations of 300, 500 and 700 ppm at the dosage point before the centrifugal pump, followed by a cationic polyvinylamide (PVAM) with low molecular weight and high charge density (540, 900 and 1260 ppm) added at the dosage point after the pump.

The results of the measurement of flocculation are shown in Figure 6.3. Four graphs show the results of the structure analysis, the red vertical line marks the wavelength for structures of two millimetres. The blue graph represents the sample without additives and show the least flocculation (low values for the variance distribution at a wavelength higher than 2 *mm*). With the addition of the dual polymer retention system flocculation increases with increasing concentration. The dosage of 700 *ppm* PAM in combination with 1260 *ppm* PVAM clearly shows the strongest flocculation tendency.

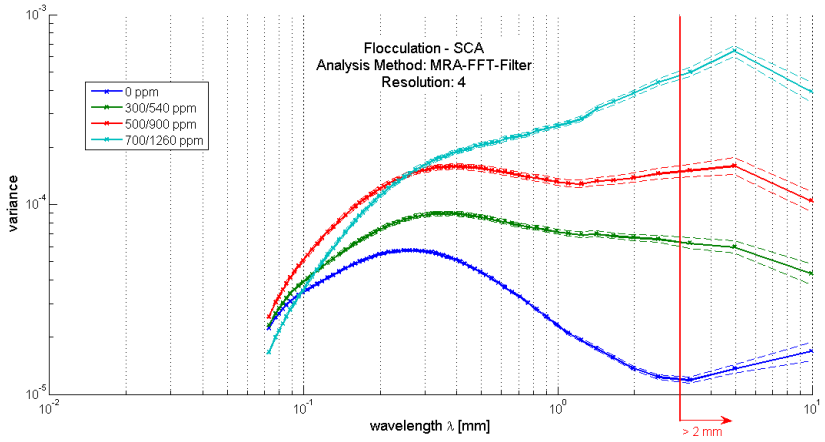

Figure 6.3 The influence of a dual polymer retention system on the flocculation tendency of a stock sample for SC-A paper. The sample without retention aids (blue graph) clearly shows the lowest flocculation tendecy. With the additions of the polymer system flocculation increases with increasing amount. The strongest flocculation is shown as 700 *ppm* of PAM followed by 1260 *ppm* of PVAM are dosed.

The results of the measurement of dewatering and retention are presented in Figure 6.4.
Figure 6.4a shows the drainage capacity. The sample without addition of retention aid has a drainage capacity of 1600 *g*. As the dual polymer retention system is dosed the drainage capacity increases by about 10% (see sample 300/540 *ppm*) up to 15% reached at a concentration of 500 *ppm* PAM and 900 *ppm* PVAM. A further increase of the concentration influences the drainage capacity only marginally.

Figure 6.4b shows the results of total retention and ash retention depending on the concentration of the dosed additives. The results clearly show that by dosing 300 *ppm* PAM and 540 *ppm* PVAM total retention as well as the ash retention cannot be increased remarkably. A total retention value of 60% and an ash retentionvalue of about 25% are reached. With a further increase of polymer addition both the total

retention as well as the ash retention can be improved up to about 90% where total retention is mainly affected due to an increased ash retention.

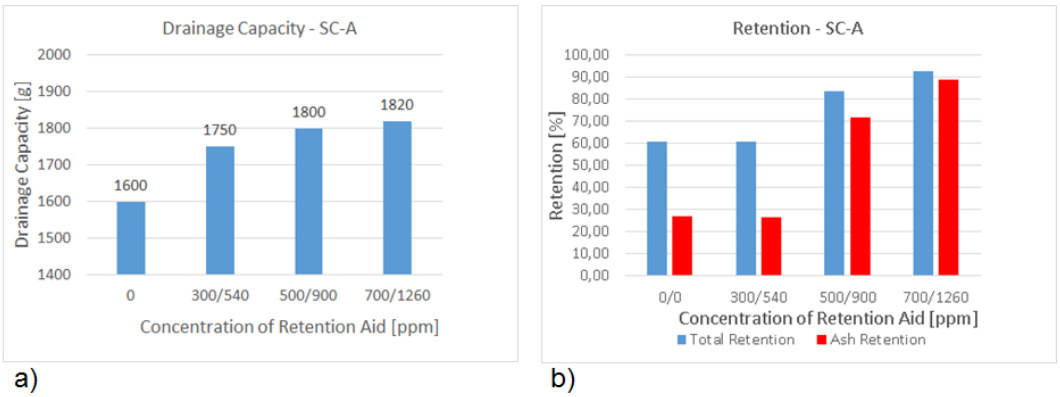

Figure 6.4 a) Evaluation of drainage capacity. Drainage is improved up to a concentration of 500 *ppm* PAM and 900 *ppm* PVAM and remain roughly static.
b) Total retention and ash-retention. Total retention is less affected at a concentration of 300 *ppm* PAM and 540 *ppm* PVAM but is improved due to higher concentrations.

Comparing the three diagrams shows the interrelation between the three parameters depending on the concentration of the used dual polymer retention system. Without dosage the values for flocculation, dewatering and retention are clearly the lowest. The higher the concentration of the polymer system the higher the values for these three parameters are. However, the dosage of 300 *ppm* PAM combined with 540 *ppm* PVAM achieves no influences on total retention and ash retention, despite a clear increase in flocculation and improved drainage capacity.

6.1.2 Results obtained for a Test Liner furnish

The following application shows the measurement performed on a sample for the production of test liner. The suspension consists of waste paper (100%) with an amount of fillers of about 17%. To improve retention a single polymer with a molecular weight and a charge density in the middle range is added at the first dosing point after the circulation pump, the polymer was added in concentrations of 250, 300 and 350 *ppm*.

The results for the evaluation of the flocculation are presented in Figure 6.5. As expected the sample without additives clearly shows the lowest flocculation. The higher the concentration of the single polymer the stronger the flocculation is, however in comparison to the other applications the flocculation intensity is rather low.

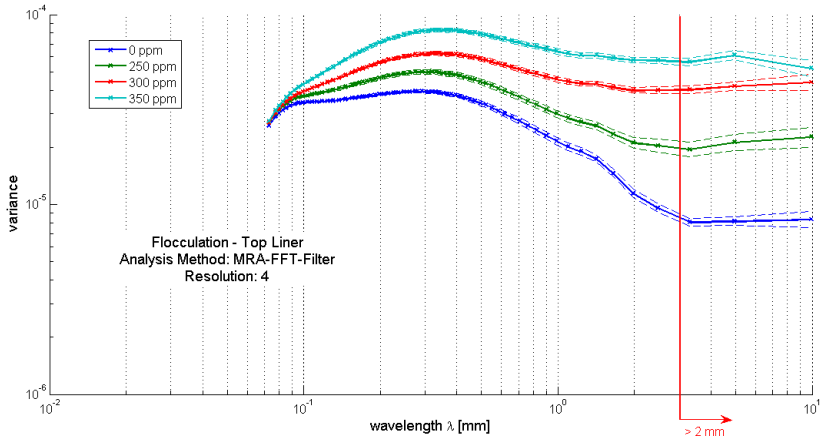

Figure 6.5 The influence of a single polymer retention system on flocculation. The sample without dosage of the polymer clearly show the lowest flocculation. By increasing the concentration of the polymer the flocculation increases too.

Figure 6.6 shows the influence of the single polymer on the measurement of dewatering and retention. The drainage capacity, shown in Figure 6.6a for the sample without additives at 1450 g is clearly the lowest. As expected an increase of the polymer concentration improves drainage capacity up to 1630 g, which is approximately an increase of about 15%.

Figure 6.6b shows the influence of the polymer concentration on total and ash retention. Both total retention as well as ash retention can be improved noticeably with increasing concentration of the retention aid. At a concentration of 350 ppm a total retention of about 90% and an ash retention of about 78% is reached.

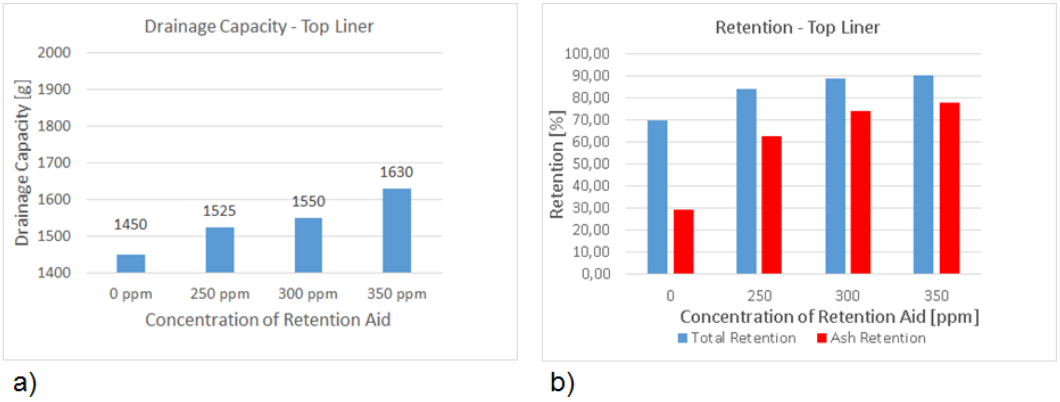

Figure 6.6 The drainage capacity as well as the retention evaluated for the test liner sample. The higher the concentration of the dosed retention aid the higher are both the total retention and ash retention.

In summary the comparison of the three evaluated parameters clearly shows a

similar trend. With increasing concentration of the single polymer system all three parameters increase too. Depending on the concentration each parameter is influenced in a different way. At a dosage of 350 *ppm* of the single polymer primarily the drainage capacity is improved, the parameters flocculation and retention are less influenced. On the other hand at a concentration of 300 *ppm* retention as well as flocculation are clearly influenced, the drainage capacity is less affected.

6.2 Summary

For three different representative stock compositions the three important parameters retention, dewatering and flocculation were measured simultaneous and the interrelation between them was visualized. Depending on the measured stock composition as well as the added retention system these parameters are influenced to different extent. An increase in retention and flocculation caused by increasing concentration of additives do not necessary mean an increase in dewatering, on the other hand dewatering can be improved at higher concentration even tough retention and flocculation may not be affected to a great extent.

Chapter 7

Conclusion and Outlook

Conclusion Retention, dewatering and flocculation are important parameters in the forming process of a fibre web. To get a better understanding regarding these parameters as well as the interrelations between them simultaneous measurements are required.
In this thesis a novel laboratory device allowing a simultaneous measurement of retention, dewatering and flocculation under industry-oriented conditions was developed.
In general laboratory device is designed as a flow loop including two measuring units, the flocculation unit and the dewatering unit.
The measurement of flocculation occurs by image acquisition of the flowing fibrous suspension in a transparent observation channel using with a high speed camera and a light panel. The evaluation of flocculation is realised by a structure analysing tool implemented in *Matlab*® based on Fast Fourier Transformation (FFT).
For the measurement of dewatering and retention a dewatering unit was developed. This unit consists of a transparent headbox including different built-in components and a dewatering device resembling a fourdinier former. The drainage process over a commercial SC/LWC wire is supported by vacuum. The filtrate is collected in white water tanks and used for the evaluation of retention and dewatering. To improve the accuracy as well as the handling of the measurement a process control system is implemented which includes separate routines for both the measurement of flocculation and the measurement of retention and dewatering. To enable a large number of trials with low time effort a comparatively small suspension volume of only 40 litres is necessary for one run. So in one day up to 20 points of measurement are possible. The measuring method is highly reproducible.
Compared to measurements for retention on industrial machines th new device may achieves a higher level of retention because of lower turbulences in the drainage

zone. On the other hand the results of the validation has shown a high sensitivity of the dewatering unit showing that investigations in a low range of concentration of polymers are possible.
Applications on industrial samples (pulp, fillers, additives process water) and variations in the concentration of the polymer relevant to the industry clearly show the effects of retention, dewatering and flocculation as well as the interrelation between retention, dewatering and flocculation.

Outlook At the present moment the essential shear rates after dosage are supplied using the centrifugal pump. Due to different flow velocities depending on the mode of operation the induced shear differ.
In order to obtain defined levels of shear rates after dosage of chemical additives a shear device has to be included in the pipeline. Using the shear device different shear rates could be adjusted. Furthermore this feature would enable investigations of the influence of shear rates regarding the parameters: retention, dewatering and fibre flocculation.
Observations of the formed fibre mat on the wire immediately after the vacuum supported dewatering zone could obtain some additional information about the formation.
As discussed in the thesis the level of retention is quite higher as achieved using other laboratory devices and/or industrial machines. To increase shear in the area of the dewatering zone the perforated plates of the drainage boxes could be exchanged. Possible elements to increase the level of shear are register rolls and foil elements.

Bibliography

Abson, D., Bailey, R. M., Lenderman, C. D., Nelson, J., and Simons, P. (1980). Predicting the performance of shear-sensitive additives. *Tappi Journal*, 63(6):55–58.

Allen, L. and Yaraskavitch, I. (1991). Effect of retention and drainage aids on paper machine drainage: a review. *Tappi Journal*, 74(7):79–84.

Athley, K., Granlöf, L., Söderberg, D., Ankerfors, M., and Ström, G. (2012). Mechanical retention - influence of filler floc size and grammage of the fibre web. *Nordic Pulp and Paper Research Journal*, 27(2):202–207.

Beghello, L., Toivakka, M., Eklund, D., and Lindström, T. (1996). A device for measuring fibre floc size in highly turbulent fiber suspensions. *Nordic Pulp & Paper Research Journal*, 11(4):249–253.

Bos, J., Veenstra, P., Verhoven, H., and de Vos, P. (2006). *Das Papierbuch*. ECA Pulp and Paper,, 2 edition. p. 245.

Britt, K. W. (1973). Retention of additives during sheet formation. *Tappi Journal*, 56(3):83–86.

BTG-Instruments (2010). Mütek dfr-05.

Chabot, B. and Daneault, C. (2004). A methomethod to determine retention and drainage in laboratory. *Paperi ja Puu - Paper and Timber*, 86(6):445–449.

Davison, R. W. (1989). A new vacuum pulsation drainage precedure for determining fine particle retention. *Tappi Journal*, pages 121–126.

Dietz, W., Mannert, C., Lehmonen, J., and Houni, J. (2010). Studying performance and influences on a small pilot plant. *International Paper World*, (6):25–27.

Eckhart, R. (2008). *A new method for single fibre flexibility measurement in flowing suspensions*. PhD thesis, Institute of Paper,- Pulp and Fibre Technology, Graz University of Technology.

Eckhart, R., Trimmel, M., and Bauer, W. (2013). Experimental investigation of the influence of fibre morphology on the interrealtion of flocculation and network strength. In *Fundamental Research Symposium*.

Eklund, D. and Lindström, T. (1991). *Paper Chemistry. An Introduction*. DT Paper Science Publications, Grankulla, Finland.

Gess, J. (1984). A new drainage analysis system. *Tappi Journal*, 67(3):70–72.

Hubbe, M. (2001). Reversibility of polymer-induced fiber flocculation by shear. 2. multi-component chemical treatments. *Nordic Pulp and Paper Research Journal*, 16(4):369–375.

Hubbe, M., Nanko, H., and McNeal, M. (2009). Retention aid polymer interactions with cellulosic surfaces and suspensions: A review. *BioResources*, 4(2):850–906.

Hubbe, M. A. (2003). Selecting laboratory tests to predict effectiveness of retention and drainage aid programmes. *Paper Technology*, 44(8):20 – 22+24–34.

Huber, P., Pierre, C., Bermond, C., and Carre, B. (2004). Comparing the fibre flocculation behavior of several wet-end retention systems. *Tappi Journal*, 3(4):19–24.

Ju, J., Hua, X., and Xiao, H. (2011). A novel ultrasonic dynamic drainage tester. In *PaperCon 2011*, volume 1, pages 195–209.

Kahl, T. (1997). Das positek-verfahren als mittel zur optimierung der füllstoffretention. *Wochenblatt der Papierfabrikation (1) p. 8*.

Karrilla, S., Räisänen, K., and Paulapuro, H. (1992). The moving belt drainage tester (mbdt). In Tappi, editor, *Papermakers Conference: Proceedings*.

Kasper, D. R. (1971). *Theoretical and experimental investigations of the flocculafloc of charged particles in aqueous solutions by polyelectrolytes of opposite charge*. PhD thesis, California Instiute of Technology, Pasadena.

Kerekes, R. and Schell, C. (1992). Characterization of fiber flocculation regimes by a crowding factor. *Journal of Pulp and Paper Science*, 18(1):32–38.

Kerekes, R., Soszynki, R., and Tam Doo, P. (1985). The flocculation of pulp fibres. In *8th Fund. res. Symp., Oxford*, pages 265–310.

Lindström, T. (1984). Network flocculation and fractionation of latex particles by means of a polyethyleneoxide-phenolforaldehyde resin complex. *Journal of Colloid and Interface Science*, 97(1).

Mason, S. G. (1954). Fiber motions and flocculation. *Tappi Journal*, 37(11):494 ff.

Norell, M., Johansson, K., and Persson, M. (1999). *Retention and Drainage*. TAPPI Press: Atlanta, Georgia. p 183-194.

Pelton, R., Allen, L., and Nugent, H. (1979). Measuring fines retention of newsprint pulps. *Pulp & Paper Canada*, 80(12):125–129.

Rasteiro, M., Garcia, F., Ferreira, P., Blanco, A., Negro, C., and Antunes, E. (2008). The use of lds as a tool to evaluate flocculation mechanisms. *Chemical Engineering and Processing: Process Intensification*, 47(8):1329–1338. cited By 37.

Rietschle, E. . (2013). Katalog g-serie.

Räisänen, K., Paulapuro, H., and Karrila, S. (1993). Wire section simulation with the moving belt drainage tester (mbdt). In Tappi, editor, *Papermakers Conference Proceedings*, pages 103–113.

Räisänen, K., Paulapuro, H., and Karrila, S. (1995). The effects of retention aids, drainage conditions, and pretreatment of slurry on high-vacuum dewatering: a laboratory study. *Tappi Journal*, 78(4):140–147.

Roschy, A., Fischer, K., and Kleemann, S. (2002). Vergleich moderner wet-end-methoden zur bestimmung von entwässerung und retention im labormaßstab.

Wochenblatt der Papierfabrikation, (18):1187–1196.

Sivén, S. and Manner, H. (2000). Twin wire retention tester. In *Tappi Papermakers conference and Trade Fair*, volume 2, pages 767–773.

Sivén, S. and Manner, H. (2001). Multicomponent retention systems in twin wire forming. *Appita Journal*, 54(6):523–526.

Smook, G. (1989). *Handbook for pulp & paper technologists*. Joint textbook committee of the paper industry. p. 224.

Staib, R. (1991). Evaluating paper ssize using a turbulent-pulse sheet former. *Tappi Journal*, 74(10):102–105.

Strengell, K., Stenbacka, U., and Ala-Nikkola, J. (2004). Advanced wire part simulation with a moving belt fomer and its applicability in scale up on rotogravure printing paper. *Pulp & Paper Canada*, 105(3):38–42.

Sutman, F. J. (2000). Development and validation of an improved drainage testing methodology. *Tappi Journal*, 83(4):69–70.

Svedberg, A. (2007). Valuation of retention/formation relationships using a laboratory pilot-paper machine. Licentiate thesis, KTH.

Svedberg, A. (2012). *Improvement of the retention-formation relationship using three-component retention aid systems*. PhD thesis, Department of Fibre and Polymer Technology, Royal Institut of Technology, Stockholm.

Svedberg, A. and Lindström, T. (2010). A pilot web former designed to study retentionformation relationships. *Nordic Pulp and Paper Research Journal*, 25(2):185–194. cited By (since 1996)3.

Swerin, A. and Ödberg, L. (1996). Flocculation of cellulosic fibre suspensions by a microparticulate retention aid system consisting of cationic polyacrylamide and anionic montmorillonite: Effect of contact time, shear level and electrolyte concentration. *Nordic Pulp and Paper Research Journal*, 11(1):22–29.

Trepanier, R. (1992). The g/w drainage-retention tester. *Tappi Journal*, 75:139–142.

Unbehend, J. (1992). *Wet end chemistry of retention, drainage and formation aids, in Pulp and Paper manufacture (R.V. Hagemeyer, Ed.) 3rd edn.,*, volume 6. TAPPI Press: Atlanta,, 3 edition. p. 137.

Wahren, D. (1967). Proposed definitions of some basic papermaking terms. *Svensk Papperstidning*, 70(21):725–729.

Wirth, B., Gralke, R., Fischer, K., and Kleemann, S. (1999). Labormethoden zur bestimmung von retention und entwässerung. *Wochenblatt der Papierfabrikation*, (17):1100–1107.

Xiao, H., Pelton, R., and Hamielec, A. (1996). Novel retention aids for mechanical pulps. *Tappi Journal*, 79(4):129.

Yan, H., Norman, B., and Lindström, T. (2006). A flow loop system for study of fibre suspensions flocculation. *Nordic Pulp & Paper Research Journal*, 21(1):19–23.