Preferred citation: M. Mayr, R. Eckhart, A. Thaller and W. Bauer. Characterization of fines quality and their independent effect on sheet properties. In **Advances in Pulp and Paper Research, Oxford 2017**, *Trans. of the XVIth Fund. Res. Symp. Oxford, 2017*, (W. Batchelor and D. Söderberg, eds), pp 299–322, FRC, Manchester, 2018. DOI: 10.15376/frc.2017.1.299.

CHARACTERIZATION OF FINES QUALITY AND THEIR INDEPENDENT EFFECT ON SHEET PROPERTIES

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ABSTRACT

It is widely accepted that pulp fines (particles passing a 200 mesh screen) largely affect pulp properties, sheet consolidation and the final paper properties. Especially fines produced during refining – so called secondary fines – showing a more fibrillar character compared to primary fines already present after the pulping process, have a positive effect on strength properties. Although this is common knowledge within the paper physics community, it is still largely unclear which detailed properties of fines influence pulp and paper properties to what extent. As fines show some similarity to MFC, this question is also of interest regarding the use of MFC as an additive in papermaking. We apply established and new methods for fines characterization, such as the secondary fines content, the swelling ability and data on fibrillation and fibrillary material together with a suitable experimental setup to isolate the technological impact of fines in the final product. Thus we are able to evaluate the technological effect of fines with different characteristics in terms of the above mentioned properties. Our results clearly show that categorizing primary and secondary fines is not sufficient when it comes to their technological impact and only in depth analysis of the fines present in a given pulp allows to understand their effect on paper properties.

INTRODUCTION

Pulp fines are most commonly defined according to their size as all particles in a papermaking furnish passing a 200 mesh screen (76 μm hole diameter; SCAN-CM 66:05). Fines definition based on commercially available fibre analyzers is different by categorizing particles smaller than 200 μm in length as fines (ISO 16065–2). Besides these two standard classifications authors have also used other definitions and methods in their work. Therefore, whenever fines are addressed in literature, one has to keep in mind that the respective definitions may vary and direct comparison may be difficult.

In addition to the size definition chemical pulp fines are further distinguished in primary and secondary fines according to their origin. Primary fines are those fines present after the pulping and bleaching process mainly consisting of ray cells, parenchyma cells, fragments of the middle lamella and only a small portion of fibrils. Secondary fines contain fibrillated and lamellar material originating from the fibre wall, as a result of mechanical forces acting on a fibre during beating [1]. When it comes to mechanical pulp fines, Brecht and Klemm [2] classify flour stuff (granular or powdery fiber pieces, cells) and slime stuff (mucillage material) according to the morphology.

Another category used in literature when referring to the fine fibre fraction is crill, defined as the part of cellulosic material that does not necessarily become entangled in the plug of fibres during plug flow [3], [4]. Based on its definition the crill of chemical pulps consists mainly of slender fibrillar particles. For PGW crill includes both flour stuff and slime stuff.

Luukko *et al*. [5] introduced the terms flake-like (non-fibrillar) and fibrillar material, which are applied for mechanical as well as for chemical pulp fines [1], [6], [7]. Flake-like material contains fibre wall fragments, thick lamellae, and ray and parenchyma cells whereas fibrillar material contains fibrils and thin lamellae as well as fibrils that are attached, for example, to a fibre wall fragment.

Prior to characterization fines have to be separated from the fibre fraction which is performed mainly using the Britt dynamic drainage Jar [1], [8] or the Bauer McNett classifier [9]–[11]. Fines fractions were further separated into subfractions based on screening techniques [8], [12], [13], tube flow fractionation [14], [15] and sedimentation [13], [16].

Chemical composition was analyzed by several groups, e.g. [10], [13], [17]–[22] showing among other differences, that fines have higher lignin and extractives content compared to the fibre fraction [10], [19]. The higher lignin content is most prominently attributed to primary fines but also to secondary fines originating mainly from the outermost fibre wall [10]. Charge and charge related measurements like zetapotential [8], [23], total charge by conductometric titration [24], total and surface charge by polyelectrolyte titration [25]–[27] and methylene

blue adsorption [22], [28] have been applied. Specific surface area was estimated by settling techniques [16], [29], [30], turbidity or transmittance measurements [31], [32] and Congo red adsorption [17].

Morphological characterization is mainly performed applying optical imaging using commercially available flow cells and optical microscopy but also using techniques based on fractionation and measurement of specific surface area which were already introduced. Other non – imaging but optical methods include the crill measurement (based on the difference in the signal under UV- and IR illumination) [33] and dynamic and static light scattering [34]. A quite rarely used non-optical technique is the electric sensing zone method [35]. These methods allow the evaluation/characterization of a certain part of the fines fraction (e.g. crill) and/or yield the corresponding size distributions. In addition to the information on content and size, imaging methods allow the evaluation of particle shape. Still, the flow cell analyzers are mainly used for the determination of fines content but not for further characterization of this fraction related to insufficient detectability of fines in aqueous media due to their transparency [36]. High resolution microscopy was mainly applied for qualitative characterization of fines and visualization of the effects of these on paper structure, e.g. [37]–[39]. Static light microscopy was used qualitatively and quantitatively for content size and shape characterization by several authors, e.g. [9], [19]. The most frequently cited and used method was introduced by Luukoo and coworkers [5], first applied on mechanical pulp fines but later on also used to characterize chemical pulp fines. This method uses staining prior to evaluation and distinguishes flake-like and fibrillar material based on grey level differentiation. Flake like material appears darker than fibrillar material and thus can be separated by an image processing routine. Size of flake like material, length of fibrillar material and fibrillar content can be determined.

The swelling characteristics representing the ability of the material to absorb or retain water was measured using size exclusion determining the fibre saturation point [40], or a variety of modifications of WRV measurements [10], [17], [42]–[44].

Due to their similarity in character methods for fines characterization are also applied for MFC or methods used for MFC might be also suitable for fines [44].

Fines in general are known to affect sheet properties [2], [3], [9], [10], [19], [46]–[48]. They increase tensile strength and z-strength, which is attributed to the high surface area and conformability yielding a high bonded area and better stress distribution in sheets containing fines [49]–[51]. Between two bonding fibres a bonding layer was identified [51] only existing in refined chemical pulps. When secondary fines are present this layer's thickness increases, while it is reduced when the fines are removed. Macrofibrils (thick bundles of microfibrils) form a network structure and microfibrils seem to fill the space between the macrofibrils. It is suggested that the microfibrils are the ones predominately contributing to bond strength. Besides,

secondary fines were found to cover the surface of fibres, as they are attracted to the fibres by capillary forces thereby also covering the edges of the fibre bonds. It is also suggested that fines have more effect on bonding than external fibrils still connected to the fibres [49], [53]. Together with tensile strength, density is also increased by the addition of fines. On the one hand fines pull the fibres closer together by capillary forces which are more pronounced for thin fibrillar fines, on the other hand they fill the voids within the network structure. Air permeability is reduced for similar reasons and the dewatering resistance is increased [46]. Dewatering resistance is increased even further because of the high swelling ability of fines [53]. Light scattering was found to be improved by mechanical pulp fines, but reduced for chemical pulp fines as more interfaces are created by the stiffer mechanical pulp fines whereas they are reduced by the flexible, fibrillar chemical pulp fines.

Differences in quality of mechanical pulp fines were discussed in detail and quantified based on the fibrillar content and the swelling of the fines [5], [9], [24], [46], [54]. The higher the fibrillar content and the swelling ability the higher the impact on strength properties. Connections were also drawn to size and shape of the material, but these parameters seem play a minor role in paper property development for mechanical pulps [9], [54]. When it comes to chemical pulps primary fines have a lower impact on paper properties than secondary fines [55], again attributed to the higher swelling ability of secondary fines [10]. Differences in swelling,, extractive content and specific surface area of fines from different wood sources and pulping processes were found to be related to strength development of sheets [17], [19], [40]. Sub fractionation of chemical pulp fines shows that the impact of these fractions on tensile strength is differing probably depending on morphology and chemical composition [19]. Changes in quality and quantity were observed when using different refining aggregates [47], [56]. It was also found that the particle size of secondary fines is larger and the number of fines lower in late wood than in early wood related to the lower S2 fibril angle and fiber wall thickness of latewood fibers promoting a cutting action [48].

As listed above a wide range of methods was applied resulting in different conclusions regarding the effect of fines quality and quantity on sheet properties. While for mechanical pulp fines the parameters fibrillar content, swelling and fines content seem to describe quite effectively their impact on sheet properties, the parameters finally determining sheet properties for chemical pulp fines are not that clear. To relate fines properties to product properties various research groups used different approaches. Sheets were formed with and without fines, allowing no clear differentiation between quality and quantity as variations in fines content in a sheet also imply a variation of the fibre content. When it comes to secondary fines most of studies used laboratory refining, where only the duration of the refining treatment is changed but not the intensity and thus merely the quantity of secondary fines is influenced but not their quality. If different aggregates were

used, only differences in size and quantity of the produced fines were discussed. As especially secondary chemical pulp fines are of a heterogeneous nature, the morphology of fines cannot be described by size only. Variations in paper properties achieved by the addition of fines from different wood sources are not related to a distinct characteristic, as the character of these fines is different on a wider range. Additionally one has to consider potential variations in fines content due to differences in fines retention in the standard sheet forming process.

In this work we will first present two improved methods dealing with the characterization of the swellability (WRV) and the fibril area of chemical pulp fines. These two methods are then applied to describe differences in fines quality, relevant in terms of their impact on paper properties. Fines were produced from two different pulps using an industrial disk refiner. In order to achieve differences in fines quality of a certain pulp, refiner settings were varied. These fines are added to different fibre qualities in sheet forming based on a trial matrix. The experimental setup thus allows to separate the impact of a given fines fraction on sheet properties.

EXPERIMENTAL SETUP

Samples

Softwood bleached kraft pulp (SBK) and softwood bleached sulfite pulp (SBSU) were refined in an industrial single disk refiner at five different refiner settings varying flow rate and refiner pressure. The refiner was operated in a range, relevant for production, leading to different settings for SBK and SBSU pulp. Details regarding the refiner settings are not given in this paper as the intention is mainly to evaluate the effect of secondary fines of a different character on paper properties. The detailed refiner settings are given in [57].

Samples were taken from the unrefined pulps as well as from the five different refiner settings for each pulp. Corresponding samples were taken within the time frame of five hours to minimize the impact of variation in pulp quality.

Methods

Pulp characterization and fractionation:

The drainability according to the Schopper-Riegler (SR) method (ISO 5267-1:1999), the gravimetrical fines content determined by the Britt Dynamic Drainage Jar (SCAN-CM 66:05) and the fibril perimeter determined by a L&W Fiber Tester^{+} (FT^{+}), were evaluated on all pulp samples.

The unrefined and refined pulp samples were fractionated using a lab-scale pressure screen [58]. The pressure screen was equipped with a perforated plate (hole

diameter 100 μm). The material passing through this plate was defined as the fines fraction and collected in a separate tank. The pulp was recirculated until the remaining volumetric fines content (measured with a L&W Fiber Tester^{+}) was below 0.5%. This fraction was defined as the fibre fraction. The fines were allowed to settle for three days, before the supernatant was removed and approximately 1% solids content was reached. Fibres were centrifuged to approximately 30% consistency.

Fines characterization

Secondary fines content:
The secondary fines content of a given sample was determined based on the fines content of the unrefined pulp and the fines content of the refined pulps (measured using the BrittJar method SCAN-CM 66:05), assuming that primary fines are not changed during refining.

Water retention value (WRV):
Measuring the WRV according to the standard procedure (ISO 23714) requires the formation of a pad of defined mass by filtration. Due to blocking of pores of the filtering element and the high dewatering resistance of fines this approach is not feasible for the pure fines fraction. The method applied in this work allows the use of the standard procedure also for fine material, without modification of the experimental conditions. Samples with a defined fines content (5, 10, 15 %) are prepared and WRV is measured according to the standard (ISO 23714). Based on the results linear regression (Eq. 1) allows the determination of the WRV of fines (WRV_{Fines}), inserting a fines content of W_{Fines} = 100% (Eq. 2) [59].

$$WRV = k^{*}w_{Fines}[\%] + d \tag{1}$$

$$w_{Fines} = 100\% \rightarrow WRV_{Pulp} = k * 100 + d \triangleq WRV_{Fines} \tag{2}$$

Microscope method – Determination of Fibril Area:
The method applied in this work is based on the method proposed by Luukko *et al*. [5] Luukko's method was mainly designed for mechanical pulp fines. The segmentation process was discussed for these fines, giving a clear picture which particle corresponds to flake – like and which to fibrillar material. As can be seen in Figure 1 (*first row*) mechanical and chemical pulp fines are highly different in their morphology. Staining of fines is important for contrast enhancement allowing a complete detection of the fines material, especially microfibrillar fines. For chemical pulp fines the dye adsorption varies between different wood sources and different morphological entities (raycells, parenchymacells, fibrillar fines) within

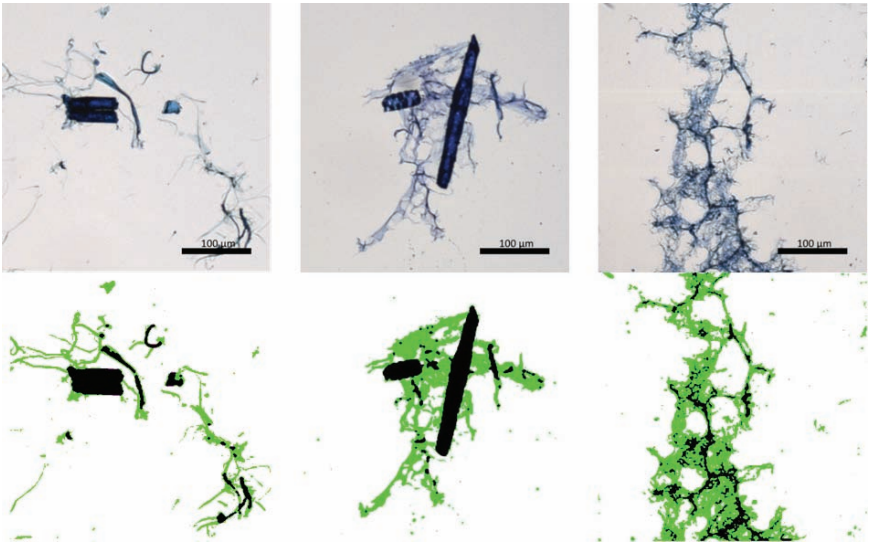

Figure 1. Microscopic images of PGW fines: (a) BSK primary fines, (b) BSK secondary fines, (c) of fines (*first row*) and segmentation of these images into flake-like materqal and fibrillar material (*second row*). Black (dark) areas correspond to flake-like fines (ray cells, parenchyma cells, fibre fragments, macrofibrillar backbone); green (light) areas correspond to fibrillar material (fine fibrils or micro fibrils).

the sample. Most probably the differences are related to the differences in their chemical nature affecting the charge content (methyleneblue adsorption was used as a fast determination method for fibre charge [28]). We found that addition of tall oil levelled out these differences and gives uniform coloration independent from chemical composition (see Appendix 1).

The microscope method determining the morphology of fines consists of three parts; sample preparation, image acquisition and image processing.

A 0.01% fines suspension was prepared in a test tube. A tall oil – water emulsion was prepared by adding 0.01 g commercially available crude tall oil to 5 g deionized water and the mixture was emulsified at 80 °C in an ultrasonic bath for 30 minutes. 0.06 g of this emulsion was added to 5 g of the fines suspension and homogenized by vigorous shaking. 0.1 g methylene blue solution (1 wt.% in water) was added and homogenized again.

Three droplets of the prepared fines suspension were placed on a microscope slide (76 × 26 mm), fixed with a cover glass (60 × 24 mm) and dried on a heating plate. A conventional transmission light microscope (Leica 301-371.010) equipped

with a standard CCD camera (Jai AM-200GE/AB-200GE) and an automated stage control (Märzhäuser Multicontrol 2000), operated by the open source software ImageJ was used for automated capturing of 700–800 single images per microscope slide (1600 × 1200 pixels per image; image area: 1380 × 1035 μm^2). An image analysis routine was programmed on the basis of Luukko's method in MATLAB. OTSU thresholding (standard routine implemented in MATLAB) was applied for multilevel thresholding, distinguishing between fibrillar material and flake–like material based on grey level differences.

In Figure 1 (*second row*) an example of a segmentation result for PGW fines, BSK primary fines and BSK secondary fines is shown. Lighter areas are classified as fibrillar material, consisting of microfibrillar fines, whereas darker areas are classified as flake-like material. Flake-like material contains ray cells, parenchyma cells but also the macrofibrillar backbone of fibrillar secondary fines. Particle size and shape of the detected segments is determined as follows. The pixels of each segment are transferred into μm^2 unit representing the segment area (A_i). After that the segments undergo a skeletonizing process and the pixels comprising the skeleton are counted and transferred to μm units. The length of the skeleton is used to approximate particle 'length' (L_i).

As a size parameter for flake-like and fibrillar segments the circle equivalent diameter (CED_i) is determined for each single particle with Area (A_i) (Eq. 3). Based on this parameter the area weighted mean size ($CED_{,q2}$, Eq. 4) is determined. The width (W_i) for segments is determined by dividing the Area (A_i) by the skeleton approximated length (L_i, Eq. 5). The aspect ratio (AR_i) is defined as the length (L_i) divided by width (W_i, Eq. 6). Mean AR (q_0) was determined according to Eq. 7.

The Fibril Area is defined as the sum of the areas of fibrillar material (A_{fibril}) divided by the sum of the areas of fibrillar material (A_{fibril}) and flake-like material (A_{flake}) (Eq. 8) of all images taken from one microscope slide. Thus Fibril Area gives the microfribrillar fines content of a fines fraction, the CED for flake-like and fibrillar material, express the size of these fragments connected after segmentation and the aspect ratio corresponds to the slenderness and branching of these segments. [60]

$$CED_i = \sqrt{\frac{4 * A_i}{\pi}} \tag{3}$$

$$\overline{CED_{q2}} = \frac{\sum_{i=1}^{n} CED_i^3}{\sum_{i=1}^{n} CED_i^2} \tag{4}$$

$$W_i = \frac{A_i}{L_i} \tag{5}$$

$$AR_i = \frac{L_i}{W_i} \tag{6}$$

$$\overline{AR_{qo}} = \frac{\sum_{i=1}^{n} AR_i}{\sum_{i=1}^{n} 1} \tag{7}$$

$$Fibril\ Area = \frac{\sum_{i=1}^{n} A_{fibril,i}}{\sum_{i=1}^{n} A_{fibril,i} + \sum_{i=1}^{n} A_{flake,i}} \tag{8}$$

Recombination of fibres and fines fractions

A first, rather simple trial setup was used to illustrate the effect of secondary fines quantity on fines characteristics and paper properties (Figure 2). Samples of unrefined and refined pulp were taken from an industrial disk refiner during production without choosing defined refiner settings. Different ratios of primary fines (SBSU uref/SBK uref) and fines after refining (SBSU ref/SBK ref) were blended and added to the sulfite fibres obtained from the same refined pulp in a constant ratio of 91% fibres and 9 % total fines with varying ratio of primary and secondary fines (Figure 2). Thus six blends containing SBK (VS_1:3) and SBSU fines (VE_1:3) with different secondary fines content were established.

A more complex trial setup was developed to look into the effect of secondary fines quality (Figure 3). Refined pulp samples were produced by changing refiner settings and separated into fines and fibre fractions as described in the beginning of this chapter. These fractions are then applied based on a sample matrix including ten different fines fractions from refined SBK (HS_1:5) and SBSU pulp (HE_1:5) and 4 different fibre fractions, SBK (HS_3,5) and SBSU (HE_3,5), also originating from these refined pulps. 9% of each fines fraction was blended with 91% of each fibre fraction yielding forty sample points. In this setup quality as well as quantity of secondary fines is changing as in industrial refining not only the quality of produced secondary fines is influenced but also the quantity. The influence of fines quantity on paper properties is however reduced, as a constant amount of 9%

	Fiber fr.	Fines fractions			
	SBSU ref	SBSU uref	SBSU ref	SBK uref	SBK ref
VS_1	91			4	5
VS_2	91			2	7
VS_3	91			0	9
VE_1	91	4	5		
VE_2	91	2	7		
VE_3	91	0	9		

Figure 2. Trial setup for evaluation of the influence of secondary fines quantity: 91% sulfite fibres were blended with 9% BSK fines (green) increasing secondary fines content in the order of VS_1 to VS_3 and with 9% sulfite fines (yellow) increasing secondary fines content in the order of VE_1 to VE_3. Primary and secondary fines were blended in different ratios.

			Fines fractions									
			HS_1	HS_2	HS_3	HS_4	HS_5	HE_1	HE_2	HE_3	HE_4	HE_5
			y1	y2	y3	y4	y5	y6	y7	y8	y9	y10
Fibre fractions	HS_3	x1										
	HS_5	x2										
	HE_3	x3										
	HE_5	x4										

Figure 3. Trial setup for evaluation of the influence of secondary fines quality and quantity: Blends including 10 different fines qualities, five BSK fines qualities (HS_1:5, $y_{1:5}$ green) and five SBSU fines qualities (HE_1:5, $y_{6:10}$ yellow) and four different fibre qualities, two BSK fibre qualities (HS_3.5, $x_{1:2,}$ green) and two SBSU fibre qualities (HS_3.5, $x_{3:4,}$ yellow). Forty combinations containing 9% fines and 91% fibres of these different qualities are obtained; prepared and measured combinations (grey coloured fields). Fines quality was influenced by applying different refiner settings.

fines was added and the impact can be related to the characteristics measured from the pure fines fraction. Only the grey coloured combinations in Figure 3 were actually prepared and tested due to time reasons.

Sheet preparation

Handsheets of 60 g/m^2 were prepared on a Rapid-Köthen sheet former (ISO 5269-2:2004) using white water recirculation [45], [55] in order to obtain comparable retention of fines in the sheets. The first five sheets were discarded until stable fines content in the sheets was achieved. Eight sheets were formed and wet pressed (150 bar, 90 sec) between two blotting papers, directly after sheet formation. Wet pressing was performed to limit the influence of capillary forces and their effects on sheet consolidation. It was found in pre-trials that the influence of fines on some sheet properties (e.g. light scattering, tear strength), became more pronounced after wet pressing. As in industrial production the paper is always wet pressed, although under highly different conditions than applied in laboratory, this setup should yield results showing higher industrial relevance.

Pulp and sheet testing

Water retention value (WRV; ISO 23714:2014) and drainability Schopper-Riegler (SR: ISO 5267-1:1999) were measured for the pulp blends. Density (ISO 534:2011), Bendtsen air permeability (ISO 5636-3:2013), tensile index (EN ISO 1924-2), z-strength (Scott Bond, ISO 15754:2009) and tear strength (ISO 1974:2012), were measured for the paper sheets.

Data processing

Data processing was applied to minimize the impact of random variation on sheet properties. To give an example: Blending fibre quality x_1 and fines quality y_1 might affect sheet formation differently than blending fibre quality x_2 and fines quality y_1. If the effect is purely related to the fines quality and thus apparent in all sets prepared with fines quality y_1 independent from the fibre type, this effect will be also visible after data processing. If the effect is only evident in one single data point it is considered random variation, which will be evened out by the data processing routine described below.

The first row ($x_{i=1}$) was taken as a basis and the mean difference ($\overline{\Delta x_{i,j}}$) between the row ($x_{i=1}$), and the rows ($x_{j=1:4}$) was calculated applying Eq. 9 (see Figure 4). The same routine was applied with the other rows ($x_{i=2:4}$) taken as a basis.

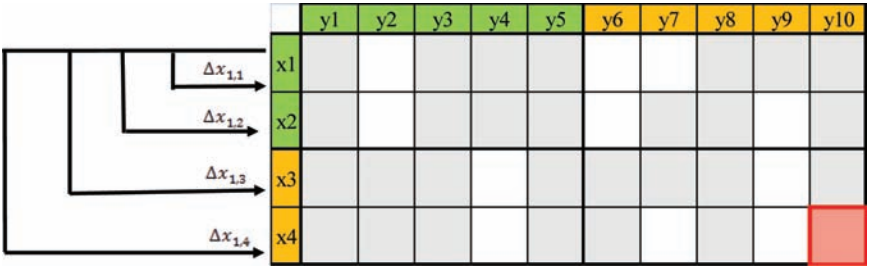

Figure 4. Data processing, determination of differences between basis row ($x_{i=1}$) and the rows ($x_{j=1:4}$).

In a second step the mean differences ($\overline{\Delta x_{i,j}}$) were added to the first row ($x_{i=1}$) and the matrix $X_{i=1}$ was calculated (Eq. 10). Applied also for the other basis rows ($x_{i=2:4}$), four 4 × 10 matrices $X_{i=1:4}$ were calculated.

The same routine was applied in the other direction, thus taking columns ($y_{k=1:10}$) as basis and determining the mean differences ($\overline{\Delta y_{k,l}}$) (Eq. 11), adding these differences to columns ($y_{k=1:10}$) ten 4 × 10 matrices $Y_{i=1:10}$ were generated (Eq. 12). The average matrix was calculated from the 14 matrices and represents the processed data matrix (XY) (Eq. 13). This operation allows also the interpolation of sample points which were not measured. This interpolation gets more uncertain when only one data point is measured in a row or a column, thus column y_9 (Figure 4) will be not considered in the discussion.

$$\overline{\Delta x_{i,j}} = \frac{\sum_{n=1}^{10} x_i, y_n - x_j, y_n}{\sum_{n=1}^{10} 1} \tag{9}$$

$$X_{i=1:4} = x_{i,j} + \overline{\Delta x_{i,j}} \tag{10}$$

$$\overline{\Delta y_{k,l}} = \frac{\sum_{m=1}^{4} x_m, y_k - x_m, y_l}{\sum_{m=1}^{4} 1} \tag{11}$$

$$Y_{k=1:10} = y_k + \overline{\Delta y_{k,l}} \tag{12}$$

$$\overline{XY} = \frac{\sum_{i=1}^{4} X_i + \sum_{k=1}^{10} Y_k}{\sum_{i=1}^{4} 1 + \sum_{k=1}^{10} 1} \tag{13}$$

Presentation of data

In the data matrix a reference point is set (marked red in Figure 2 and Figure 3) and mean differences to that reference point are calculated. The data is presented as is shown in Figure 5 (secondary fines quantity) and Figure 7 to Figure 12 (fines quality) respectively. The reference point is set to 1 and the relative difference to that point represents the difference arising due to changes in fines quality. The averaged measured value of this reference point is added in the diagram as a number (in red text).

RESULTS:

Variation of secondary fines quantity at equal quality:

As is discussed by several authors [55], [60], secondary fines show a more beneficial influence when it comes to paper properties compared to primary fines. This topic will be addressed in the first trial series (see Figure 2) to show the capabilities of the applied methods for evaluation of fines characteristics and their effect on paper properties in a simple setup. Therefore the effect of the quantity of secondary fines on tensile index and fines characteristics is investigated. Different quantities of secondary fines were achieved by blending primary fines with fines from pulp after refining in different ratios (see Figure 2). Thus a variation in secondary fines content of constant quality was achieved (Figure 5(a)).

Looking at the fines characteristics with the above described methods it is evident that the water retention value (WRV) of fines as well as the Fibril Area measured by the microscope method rises continuously with increasing quantity of secondary fines (Figure 5(b), 5(c)). This reflects the expected higher swelling ability and more fibrillar character of secondary fines. When evaluating their impact on sheet properties it has to be pointed out that the fines content in the sheets was held constant at 9%, varying only the ratio between primary and secondary fines. The influence of fibres on sheet properties was held constant by blending these fines always with the same fibres.

Tensile index showed an increase with increasing amount of secondary fines as those fibrillar secondary fines are more bondable than the ray and parenchyma cells containing primary fines. This is in accordance with the findings in the literature.

That sulfite pulp achieves lower strength properties compared to kraft pulp is well known, but here it is shown that this is also the case for the isolated behavior of the corresponding fines fraction. Although the secondary fines content is higher in the blend for sulfite pulp (Figure 5(a)) fibril area is comparable and WRV is even lower compared to kraft fines, related to the lower tensile index achieved for the sulfite fines [40].

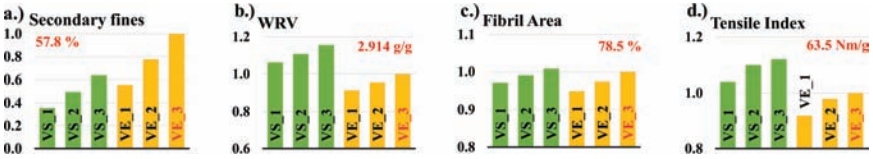

Figure 5. Trial setup for evaluation of the influence of secondary fines quantity: fines properties: (a) share of secondary fines in the fines fraction containing primary and secondary fines; (b) WRV of fines, (c) Fibril Area determined by the microscope method, (d) Tensile Index measured from sheets with 91% fibre (of same quality) and 9% fines with varying quantity of secondary fines.

Variation of secondary fines quality and quantity

Characterization of pulps

Pulps obtained by the variation of refiner settings in terms of flow rate and refiner pressure were characterized prior to fractionation. Due to these refining variations different quantities of secondary fines were produced resulting in a change of the total fines content in the refined pulp samples (Figure 6(a)). Also variations in the fibril perimeter determined by the FT^{+}, representing external fibrillation of the fibres, are measured (Figure 6(b)). Although fines production and external fibrillation is related, the trends are not directly comparable (Figure 6(a), 6(b)). The SR represents a sum parameter not differentiating between fibre properties and fines properties (Figure 6(c)). Still, when it comes to paper properties work in literature shows that external fibrillation has less effect than secondary fines content most probably due to the higher freedom of fines moving to any possible position within a network [48], [51], [52]. In this respect it is also of interest to separate the impact of fines from the impact of fibres on pulp and paper properties.

Characterization of fines quality

Fines quality was characterized from the separated fines fractions with the methods described above yielding WRV representing swelling ability and the

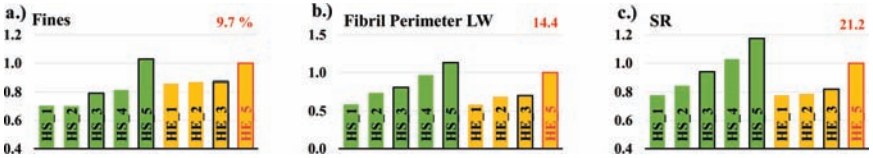

Figure 6. Characterization of pulps obtained by variations of refiner settings: (a) fines content after refining; (b) fibril perimeter; (c) SR.

Fibril Area, representing quantity of microfibrillar fines (Figure 7(b), 7(c)). As only secondary fines are produced during refining, the quantity of secondary fines of the separated fines fraction (Figure 7(a)) will change according to the change in total fines content of the pulp (Figure 6(a)). Sample HS_2 possessing one of the lowest secondary fines contents in the provided samples shows the highest WRV and Fibril Area (Figure 7(b), 7(c)) which is totally different to the trial series above where secondary fines content showed clearly the same trend as WRV and Fibril Area. This underlines that not only quantity of secondary fines is of interest when it comes to the properties of fines, but to a considerable extent also the fines quality. These big differences in fines quality could not have been observed when refining pulp in a lab device (e.g. in the PFI mill), as only the duration of the treatment is changed but not the intensity of the impact on the fibres which is achieved in industrial refining when changing the specific edge load and specific energy consumption. WRV correlated well with the measurement of Fibril Area for BSK fines (HS_1:5) but not for sulfite fines (HE_1:5). Especially in sample HE_5 the low Fibril Area is in contrast to the high WRV, indicating that Fibril Area and WRV are not measuring the same property, although this would be assumed considering results from the trial series on the quantity of secondary fines. An explanation will be given below based on the in depth morphological characterization of the fines networks.

Figure 8 shows parameters obtained by the microscope method additional to the Fibril Area. The first row shows parameters of the flake like material, the second row parameters of the fibrillar material. The circle equivalent diameter (*CED*) describes the average size of a connected fragment after segmentation, representing the area of the fragment. The aspect ratio (AR) describes the slenderness and/or the branching of the segment. Fibril Area (Figure 7c) is related to the size of flakes (*CED* flakes) and fibrils (*CED* fibril). For BSK fines at similar secondary fines content a decrease in flake size is accompanied by a decrease in fibril size (HS_1 ≥ HS_2) or vice verca (HS_3 ≥ HS_4). This indicates the size of secondary fines networks – comprised of flake – like and fibrillar segments – as is exemplarily shown in Figure 9 (*first row*). The size of flake like material in

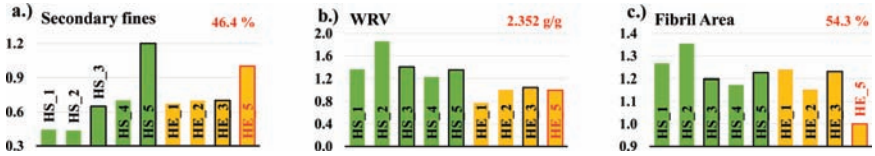

Figure 7. Trial setup for evaluation of the influence of secondary fines quality and quantity: Characterization of separated fines fractions of fines obtained by the variation of refiner settings: (a) secondary fines content; (b) WRV; (c) fibril area.

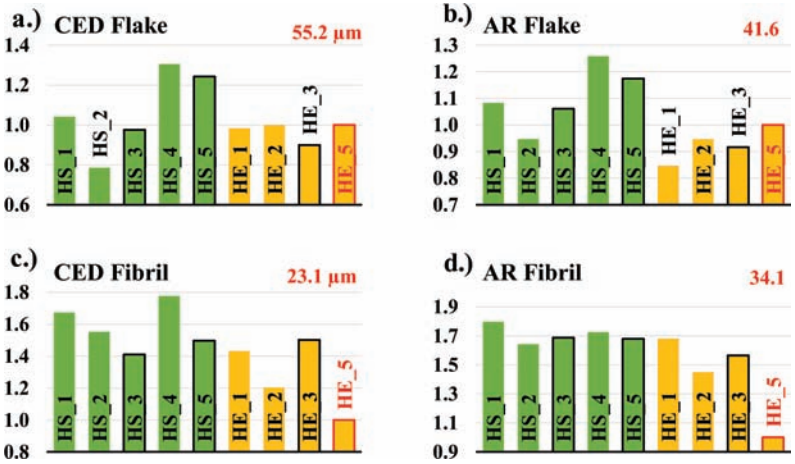

Figure 8. Parameters obtained by the microscope method in addition to the Fibril Area; size of segments expressed by the *CED* of flake – like (a) and fibrillar material (c); aspect ratio (*AR*) indicating slenderness and branching of segments for flake-like (b) and fibrillar material (d).

sulfite fines remain rather constant for HE_1,2,5 while fibril size decreases, leading to a reduction in fibril area. This change of morphology representing the change in the size ratio between flake like and fibrillar segments is illustrated in Figure 9 (*second row*). Therefore the WRV does not reflect Fibril Area as it is the case for kraft fines, because the high secondary fines content in HE_5 shows a high proportion of flake like segments yielding a low Fibril Area. Still, the high proportion of macrofibrillar backbones in the secondary fines networks has a higher WRV compared to ray or parenchyma cells contained in primary fines.

Impact of fines quality on pulp and sheet properties

The above discussed results of fines and pulp characterization will now be related to pulp and sheet properties measured on the blends of 9% fines and 91% fibres (see Figure 3) based on the results after data processing.

The WRV of the different fines fractions derived from the results after data processing (Figure 10(a)) correlates well with the WRV measured directly on these fines fractions (Figure 7(b)). The differences between these two values represent the difference in the amount of fines addressed. In the directly measured values the variation represents 100% fines whereas in the processed results the variation represents the impact of 9% fines in a blend with the fibre fraction.

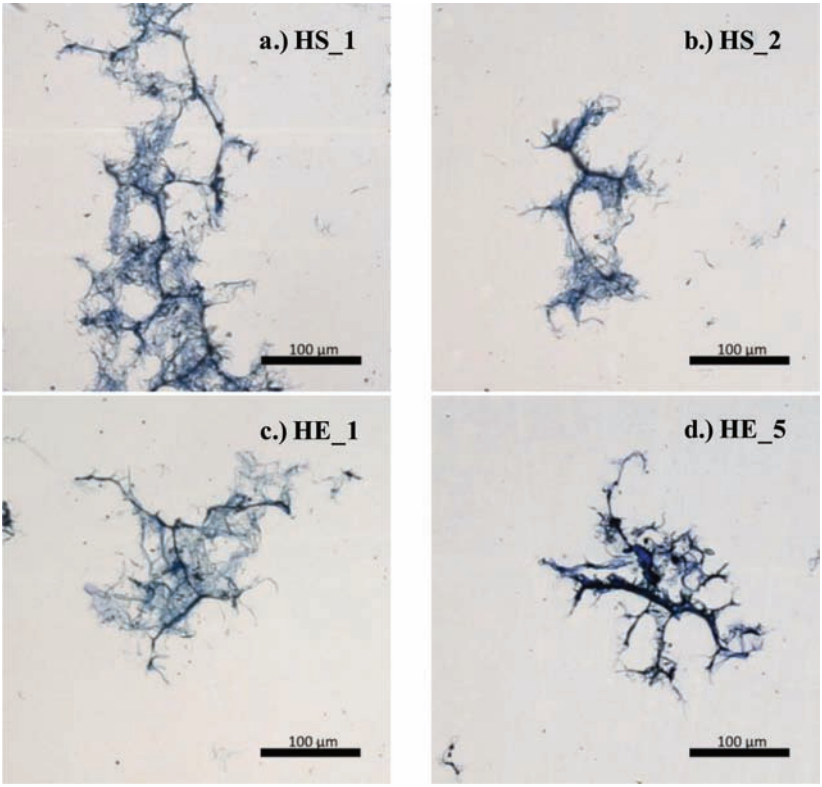

Figure 9. Microscope images demonstrating the differences in fines morphology determined by the parameters obtained from these images; Size of fines networks ((a), (b)); Ratio between fibrillar and flake – like material of the fines network ((c), (d)).

Keeping that in mind not only the trend but also the absolute values match quite well. This high correlation between directly measured and extracted data confirms that the trial setup and the data processing routine yield correct and reliable results for the impact of the different fines fractions.

SR shows the same trend as the WRV (Figure 10(a)–(b)), indicating that the impact of the fines fraction on the SR is directly related to the volume change of fines due to swelling.

The apparent sheet density (Figure 11(a)) of the wet pressed sheets is also affected by the fines quality. For example when comparing HS_1 and HS_2, containing a comparable quantity of secondary kraft fines, sheet density is higher for HS_2. This fines fraction also shows a higher WRV and fibril area and may

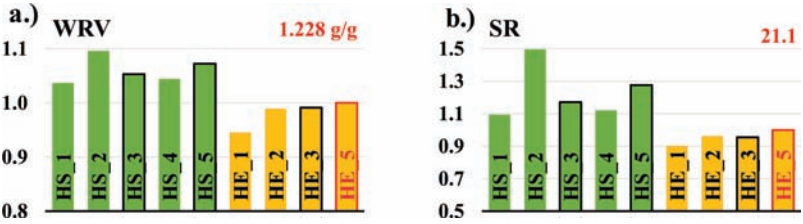

Figure 10. Impact of variation of fines quality on (a) WRV and (b) SR of blends containing 9% fines and 91% fibres.

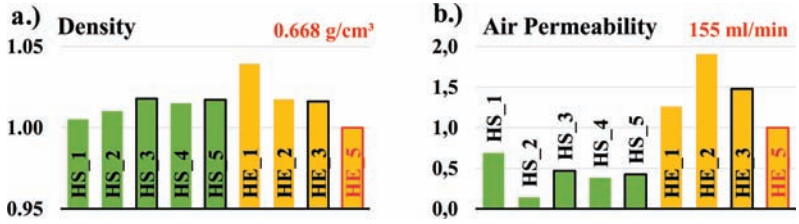

Figure 11. Impact of variation of fines quality on (a) sheet density and (b) air permeability of blends containing 9% fines and 91% fibres.

be capable of filling up voids more easily due to the more fine fibrillar character. Considering the trend in sheet density for sulfite fines, sheet density seems to be to a greater extend affected by the fibril area (microfibrillar fines), than by the WRV, as in this case WRV does increase while fibril area decreases together with decreasing density.

Air permeability (Figure 11(b)) shows a large difference between BSK and sulfite fines quality with sulfite fines obtaining higher air permeability at similar sheet density. That BSK fines show a lower air permeability is most probably also related to the higher swelling of these fines qualities (HS_2 showing the highest WRV and Fibril Area also exhibits the lowest air permeability). Apart from that a correlation between fines characteristics and air permeability is not obvious but porosity will most probably be affected by sheet density, fibril area, WRV and the secondary fines content to different extents that cannot be separated and quantified based on this data.

Scott Bond (Figure 12(b)) is especially for BSK fines highly consistent with the development of fibril area. Also Nanko and Ohsawa [51] considered the microfibrillar fines predominantly responsible for bonding in refined chemical pulps. The high variation in sulfite fines qualities in terms of flake-like and fibrillar character does not allow to link Scott Bond development directly to Fibril Area.

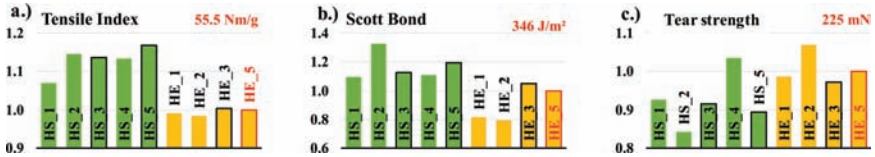

Figure 12. Impact of variation of fines quality on strength properties: (a) tensile index and (b) Scott Bond (c) tear strength, of blends containing 9% fines and 91% fibres.

Nevertheless the difference in Scott Bond between HE_2 and HE_3, showing similar secondary fines content, WRV and sheet density, is most probably related to the difference in Fibril Area. Tensile index (Figure 12(a)) develop somewhat different from Scott Bond. In addition to the Fibril Area (microfibrillar fines), secondary fines content (macrofibrillar backbone + microfibrillar fines) seems to affect this parameter. For example HS_1 and HS_2 with the lowest secondary fines content achieve lower tensile index in relation to Scott Bond. HS_5 on the other hand with the highest secondary fines content shows the highest tensile index, but not the highest Scott Bond, which was achieved for the sample HS_2 with the highest Fibril Area. The secondary fines content implies the quantity of macrofibrillar backbones of fines, influencing tensile index, were the sheet is loaded in plane during measurement to a higher extent than Scott Bond, applying the load out of plane as there might be an orientation of such structures predominantly in plane. This would be reasonable considering SEM images of fines stretching between voids and covering the surface of fibres [37].

That this backbone contributes to tensile index is also evident in the higher difference between SBSU and BSK fines when it comes to tensile strength, compared to Scott Bond. Sulfite fibres and fines are widely accepted to show considerably different character than kraft fibres and fines especially when it comes to tensile properties [40], [61], [62] which is also shown in the appendix for the 91% fibre fraction of sulfite and kraft pulps (Appendix 2). Thus the macrofibrillar backbone consisting of oriented microfibrils might have similar characteristics than a fibre.

Also tear strength (Figure 12(c)) is influenced by fines quality. Fines were found to decrease tear strength due to their higher bonding ability compared to fibres [45], [48]. This is evident from HS_2 fines with the lowest tear strength at highest Scott Bond. Additionally tear strength shows a similar trend than the *AR* of flakes for both kraft and sulfite fines (Figure 8(b)). The *AR* is a measure for the slenderness but also for branching of the macrofibrillar backbone. A higher *AR* indicates a higher total backbone length seeming to give higher tear strength.

CONCLUSION

Chemical pulp fines are highly heterogeneous in terms of morphology and chemical composition and their impact on sheet properties cannot simply be described by their quantity or by their origin. In this setup fines quality was varied by applying different refiner settings thus affecting fines morphology in terms of size, aspect ratio and fibrillar character. As all fines varieties were produced from two different pulps, a discussion of the influence of different pulp chemistry is not possible, which of course is also important for bond formation and thus strength properties. The measured differences in quality are only described in terms of morphology and swelling ability determined based on methods implemented especially for this task.

This study shows that such an in depth fines characterisation together with a suitable experimental setup allows a better understanding of the impact of fines quality on paper properties, which is usually superimposed by the impact of the fibre fraction and the quantity of fines. Such insights are not possible with standard testing and without a setup to separate the impact of the fines fraction from that of the fibre fraction.

Based on the presented results it is obvious that a differentiation in primary and secondary fines usually applied to address fines characteristics is not sufficient as secondary fines can show quite different properties in terms of fibrillar character, size and swelling ability. Depending on the sheet property one or more fines characteristics are descriptive. Strength properties for example are highly related to fine fibrillar material and the respective swelling ability of the material.

ACKNOWLEDGEMENTS

The authors acknowledge the industrial partners Sappi Gratkorn, Zellstoff Pöls AG, Norske Skog Bruck and Mondi Frantschach, the Austrian Research Promotion Agency (FFG), COMET, BMVIT, BMWFJ, the Country of Styria, and Carinthia for their financial support of the K-project FLIPPR°.

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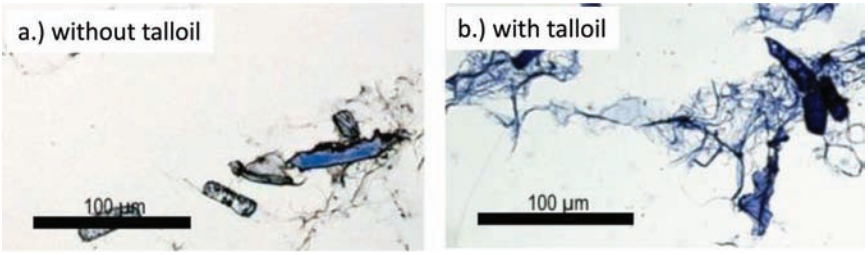
APPENDIX



Appendix 1. Sample preparation microscope method, staining of BSK primary fines: (a) without talloil, (b) with talloil.

Appendix 2. Development of strength properties as a result of changing fibre quality: (a) Tensile Index, (b) Scott Bond.

Transcription of Discussion

CHARACTERIZATION OF FINES QUALITY AND THEIR INDEPENDENT EFFECT ON SHEET PROPERTIES

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Jean Francis Bloch Grenoble Institute of Technology

What is your understanding of water retention value for fines? Where is the water, inside? Or outside, due to capillary effect? And if it is the case, in fact you have twice the effect of the fibrillation.

Melanie Mayr Graz University of Technology

Yes, the water retention value is a quite heterogeneous parameter, so that we have differences of course when we look at fibres, with the swelling of the fibre wall and also the swelling of the external fibrils on the fibres. When we go down to the fines we have seen we have a kind of macrofibrillar backbone in the secondary fines, so also there will be a kind of internal swelling and in the microfibrils of course. There might somehow also be the capillary effect where the water is held between the interstices, but I think as long as they are not elementary fibrils, we have also swelling inside this material.

Hahn-Ning Chou Thepharak

In addition to virgin fibres, I am curious to hear what you have to say about the effects of using recycled fibres, such as OCC, and what you think the results would look like?

Discussion

Melanie Mayr

I have not used any recycled pulp in this study, we just have used virgin pulp. However there are already differences when we compare the bleached kraft pulp and the bleached sulfite pulp and I would also expect that the recycled pulp would react differently. However, I think that the fibril area and water retention value will continue to provide good predictive access to the technological properties of recycled pulp.

Hahn-Ning Chou

Thank you. Fines play a more complex role in recycled fibre, so I think it will be an interesting study for you.

Joel Panek WestRock

I want to ask you to clarify some of the work. If you can go back to slide 18, I just wanted to make sure I'm straight on how you went through the data and got to your conclusions. So you were showing on the bottom graph that you are increasing the refining Specific Edge Load and the y-axis shows the relative effect of a property. So you are interpreting as you increase a parameter how it relatively changes a property. What was the difference from the lighter-coloured SEC to the one next to it?

Melanie Mayr

The difference between the lighter colour and the darker colour is their Specific Energy Consumption. We have lower Specific Energy Consumption applied for the lighter colour and the higher Specific Energy Consumption for the darker colour.

Joel Panek

So you are comparing different Specific Energy Consumptions and then seeing the relative effects and then the red ones versus the grey ones, those are two different types of pulps?

Melanie Mayr

Yes, our main interest was there to validate our methods for fines characterization, so yes it is nice to see that we have done the variation in Specific Energy Consumption, but our major goal was to prepare different fines with different characters.

Joel Panek

So, if you go back to slide 10, the one that has secondary fine content. So you looked at these conditions to see how the secondary fines content changed. What were the other characteristics you looked at for the fines?

Melanie Mayr

The water retention value and also the fibril area.

Joel Panek

Is that on the next slide? So, this shows, as you go up in SEL there is a larger increase in the water retention value, but there is not much difference . . .

Melanie Mayr

In secondary fines content? So, we produced in both cases secondary fines, but we can really see the impact of increasing Specific Edge Load only when we look at the water retention value.

Joel Panek

So, I would like to turn to the audience now. Is anyone surprised by this, is this what you would expect to see?

James De Witt SAPPI

I guess I have a problem with the measurement of fines water retention values by themselves. In my experience anyway, when you go to a high fines furnish like a BCTMP, you get the phenomenon of the filter blinding over. This essentially does not dewater the mat and you have quite high water retention values and I wonder how you ensured that you weren't in fact blinding the filter. It is a centrifugal measurement, I assume, with a standard method and you know this method again in my experience is very reliable on things like unrefined kraft pulp, but when you get to furnishes with higher fines, they tend to be erratic in their results.

Melanie Mayr

So, we don't exactly measure the water retention value of the pure fines, but we blended these fines with fibres at different fines ratios and what we can see here is

we get a linear relationship between the water retention value and fines content, so we measured this water retention value for the different fines with the standard method, and what we get out of this is, then by calculation is, the water retention value of both fines and fibres.

James De Witt

I guess I would expect the method to be less accurate despite your correlation. I think you might sometimes get a good correlation and other times not, but just based again of my experience.

Melanie Mayr

Yes, we have done this for 16 different pulps and we calculate the water retention value of the pulp and then we correlate the water retention values of the pulp directly measured and the water retention values we get by calculation, and we got a very good correlation including mechanical pulps as well as chemical pulps.

Bill Sampson, University of Manchester

We too use this method where you add different amounts of fines and use the rule of mixtures really to calculate what the water retention value of the fines is. We find it very reproducible. It does work very well and it does get you past the problem of trying to centrifuge a mat of fines.