Quantifying the contribution of fines production during refining to the resulting paper strength

Daniel Mandlez · Sarah Koller · Rene Eckhart · Artem Kulachenko · Wolfgang Bauer · Ulrich Hirn

Received: 29 March 2022 / Accepted: 16 August 2022 / Published online: 31 August 2022 © The Author(s) 2022

Abstract Pulp refining is an essential process step prior to paper production. The contribution of fines production during refining to the resulting paper strength so far has mostly been considered qualitatively. A quantitative and experimental evaluation regarding their effect has not yet been published. Unbleached softwood kraft pulp was refined using a PFI mill and a disc refiner at different refining intensities. Prior to handsheet forming, fines were removed in a lab scale pressure screen from one part of the refined and unrefined samples in order to investigate the difference in tensile strength between sheets with and without fines, which were furthermore produced with and without additional wet pressing. It was found, that fines formed in a disc refiner at 250 kWh/t are responsible for up to 25% of the breaking length increase, while the PFI mill at 10,000 revolutions fines only contribute to a maximum of 12%. In terms of fines efficiency, the disc refiner was able to achieve higher results compared to the PFI mill, which however might be attributed to the higher fibre flexibilization in the PFI mill. Thus fines formed in the refining process are of high importance for strength development especially for the disc refiner.

Keywords Refining · Beating · PFI · Disc refiner · Fines · Strength properties

Introduction

Motivation

The refining of pulp fibres is an essential process step in stock preparation prior to paper production in order to obtain specific paper properties required for the respective paper grade. Refining is used to achieve a certain strength potential as well as specific porosity properties. In principle, the consequences of refining on pulp and paper properties are well known. For example the tensile- and burst strength increases, while opacity and tear index decrease (Herbert and Marsh 1968; Töppel 1993; García et al. 2002). A negative side effect of refining is the increasing dewatering resistance (Herbert and Marsh 1968; Töppel 1993; Park et al. 2006), which affects the paper production process. On the fibre level different effects of refining can be observed.
Refining effects on pulp/fibre level

As a consequence of the mechanical treatment during refining, fibres are modified in different ways which can be divided into internal fibrillation, external fibrillation and fibre shortening (Higgins 1961; El-Sharkawy et al. 2008; Park et al. 2006).

*Internal fibrillation* means the delamination of the cell wall structure, which leads to higher flexibility and conformability of the fibres (Genco 1999). The flexibilized fibres create a more dense paper network which leads to an increased relative bonded area (RBA) which in turn improves the paper strength (Ingmanson and Thode 1959; Rennel 1969). According to Hartman (1995), internal fibrillation is the most important refining effect in terms of tensile strength improvement.

*External fibrillation* describes the partial destruction of the fibre cell wall. The mechanical impact on the fibre surface causes the fibrils to tear out (Ferreira et al. 2000) generating a roughened fibre surface leading to an increased fibre surface area. This not only increases the fibre-fibre bonding area, but also enhances mechanical interlocking between fibrils from different fibres (Kang and Paulapuro 2006; Motamedian et al. 2019).

*Fibre shortening or cutting* is the third effect of refining. First, the shortened fibre length leads to a reduced flocculation of the pulp (Kerekes and Schell 1992), but however it also leads to a decrease in paper strength (d’A Clark 1987).

*Fines* formation is a result of external fibrillation (Higgins 1961) and fibre shortening. In contrast to so called primary fines, which are a result of the pulping process, secondary fines are produced due to the mechanical impact during refining (Odabas et al. 2016). According to ISO 16065-2 fines are defined as particles smaller 200 μm, where the particle size is determined using an optical fibre analyzer. Another common way to define fines is classification using a 200 mesh (76 μm hole diameter) screen in a Britt Dynamic Drainage Jar (BDDJ) according to SCAN-CM 66:05, where all material passing the screen is defined as fines. Furthermore, fines can be classified by their morphology. Here they are divided into fibrillar fines and flake-like fines (granular material) (Odabas et al. 2016; Mayr et al. 2017), as depicted in Fig. 1.

![Image: Sketch of the cell wall structure and various types of fines grouped in fibrillar fines and flake like fines, adapted from (Odabas et al. 2016)](image)

The primary fines mainly consist of flake-like fines, including ray cells, parts of “pores” e.g. border pits, or fragments of the middle lamella and primary cell wall (Odabas et al. 2016). Chemical pulps usually have a primary fines content of about 5% (w/w). In contrast to that, secondary fines result from torn out fibrils and fibril bundles from the fibre wall due to the mechanical stresses during refining (Genco 1999). Therefore, secondary fines mainly exhibit a strong fibrillar nature (Ferreira et al. 2000; Odabas et al. 2016; Mayr et al. 2017). Due to their strong fibrillar character, secondary fines have a higher bonding ability compared to primary fines (Mayr et al. 2017). Nevertheless, most recent studies have shown, that also unbleached softwood kraft (UBSK) pulp primary fines contribute to fibre-fibre bonding and therefore increase the tensile strength (Mandlez et al. 2020). Next to fibrils and fibril bundles, the group of fibrillar fines includes colloidal material and loose slender fibrils (Kibblewhite 1975; Odabas et al. 2016). This fines material which is known as crill (Steenberg et al. 1960; Sandgren and Wahren 1960) appears invisible to conventional imaging systems (Mayr et al. 2017) used in ISO 16065-2. Common crill measurement is based on the different response of fines and fibres on ultra violet and infrared light (Osong et al. 2016).

Effects on fibre network/paper level

While refining is modifying the pulp at the fibre level, its actual intention is to improve the properties of the paper network. Apart from the optical properties and porosity, sheet strength is of high interest. Besides mechanical interlocking, which is mainly caused by external fibrillation (Motamedian et al.
tensile strength can be enhanced by increasing the (RBA), and the sheet density (Rennel 1969; Ingmanson and Thode 1959; Retulainen et al. 1993)

Sheet densification based on refining seems to be the key parameter to improve tensile strength properties as has been shown in several studies (Kibblewhite 1975; Koskenhely et al. 2005; Mayr et al. 2017; Motamedian et al. 2019) pointing out a strong linear correlation between densification and tensile strength (Koskenhely et al. 2005; Nordström 2016).

The densification is caused by internal fibrillation of the fibres and formation of secondary fines. Fibre shortening is not contributing to densification (d’A Clark 1987), external fibrillation, however, might slightly increase fibre flexibilization and thus paper density. However, it should be noted, that fibre shortening can yield fragments that that can be defined as fines. Therefore, fibre shortening can be both, a modification of the fibre as well as the basis for fines formation.

Sheet densification based on wet pressing: Nevertheless, refining is not the only cause of sheet densification during paper production, as also wet pressing increases the sheet density. On industrial paper machines wet pressing is an essential step for water removal and leads to consolidation of the fibre network at the same time. Exactly this consolidation increases the sheet density and therefore also the RBA resulting in an increase of tensile strength (Vainio and Paulapuro 2007). Rennel (1969) and later Nordström (2016) compared the increase in strength properties between refining and wet pressing for handsheets. They found that for equivalent sheet density, refined sheets had a higher paper strength than wet pressed sheets. In both cases—wet pressing and refining—a predominantly linear relation between density and tensile strength has been found, though the slope and offset of the linear relationships were different.

Refining equipment

Refining equipment can be divided into continuously operated industrial refiners and laboratory devices such as Valley Beater, Jokro mill and PFI mill which are operated in batch mode (Eibinger 2005; Gharehkhani et al. 2015). For laboratory refining the PFI mill is discussed in more detail, as it is used for the present research study. Basically, the impact of refining on fibre modification and fines creation differs depending on the used refining equipment and refining conditions. As depicted in Fig. 2 the resulting breaking length at a given dewatering resistance is higher for the PFI mill compared to a refiner.

During PFI refining the mechanical treatment is mainly caused by impulses of the beating body bars, which causes predominantly internal fibrillation (Yasumura et al. 2008; Kerekes 2005; Wang et al. 2007; Gharehkhani et al. 2015).

In addition to densification industrial refiners induce considerable fibre shortening and external fibrillation (Kerekes 2005; Yasumura et al. 2008). In summary both equipment cause internal- and external fibrillation and production of fines. However PFI refining has a tendency towards more internal fibrillation while industrial refining leads to more pronounced external fibrillation, fines production and fibre shortening.

Aim of the work

The role of external fibrillation (Ferreira et al. 2000; Kang and Paulapuro 2006; Motamedian et al. 2019), internal fibrillation or densification (Genco 1999; Koskenhely et al. 2005; Mayr et al. 2017; Motamedian et al. 2019; Mandlez et al. 2020) as well as fibre shortening (d’A Clark 1987) has been widely discussed in the literature. In contrast, apart from work based on fibre network modelling (Motamedian et al. 2019), the role of fines produced in refining on the
resulting development of paper strength has mainly been discussed qualitatively (Luukko and Paulapuro 1999; Mayr et al. 2017b). However, it has not yet been evaluated in a quantitative experimental investigation. Therefore, the aim of this work is to attribute the strength development due to refining to either secondary fines production or fibre modification i.e. internal fibrillation, external fibrillation and fibre shortening. The effect of secondary fines is quantified as a percentage of the total strength increase due to refining. Thereby the focus is set on commonly used refining equipment at their typical range of refining intensities.

For this purpose, the fines fraction of refined and unrefined pulps was removed and the tensile properties of handsheets produced with and without fines were evaluated in this study. Thus, the strength increase of the pulp without fines represents the effect of fibrillation and fibre shortening only. The effect of fines is determined by comparing pulp with and without fines. Additionally, the effect of wet pressing on the influence of fines on strength properties was investigated.

Experimental

Materials

An industrial, once dried, UBSK pulp with Kappa number 40 was used in all trials. The primary fines mass content of the pulp determined according to SCAN-CM 66:05 was 5%. Fig. 3 shows the cumulative length weighted fibre length distribution of the unrefined reference pulp.

Experimental procedure

Figure 4 shows the three main steps of the experimental procedure also giving an overview of the conducted measurements on the pulp and paper samples:

1. Refining: Using the UBSK reference pulp unrefined and refined samples were prepared at neutral pH conditions. For the refining, an industrial pilot disc refiner and a laboratory PFI mill were used at different refining intensities.

2. Fines separation: Fines were removed from one part of the unrefined and refined pulp samples prior to handsheet forming, while fines remained in the other part

3. Handsheets: From all of the above-mentioned fines containing and fines free pulp samples, two sets of handsheets were prepared—one with additional wet-pressing and one without additional wet-pressing.
Refining

**Disc refiner**

For refining a pilot scale 12-inch single disc refiner was used to produce samples at two different refining intensities of 100 kWh/t and 250 kWh/t at a specific edge load of 0.3 Ws/m. Prior to refining, 55.8 kg\text{ad} of dry pulp was diluted to a consistency of 3.9% (w/w) according to DIN EN 20638 corresponding to the above mentioned tank volume \( V_\text{tank} \) of 1430 L. 24 h after dilution, disintegration was carried out in the feed tank with a duration of 30 min using the integrated propeller stirrer. Deflaking was done by passing through the refiner with a volumetric flow \( \dot{V} \) of 190 L/min at open refining gap for 15 min which therefore equals two cycles.

The disc refiner was operated at an effective power \( P_{\text{eff}} \) of 10 kW. The pulp suspension was fed to the refiner with a volumetric flow \( \dot{V} \) of 190 L/min at a consistency \( c \) of 3.9% (w/w). Thus according to Eq. 1 (Herbert and Marsh 1968), the specific energy consumption 258 (SEC) for a single pass through the refiner is 22.5 kWh/t.

\[
\text{SEC} = \frac{P_{\text{eff}}}{\dot{V} \cdot c} \tag{1}
\]

In order to achieve the targeted refining intensities of 100 kWh/t and 250 kWh/t, the refiner was operated in a closed loop as it is shown in Fig. 5. A detailed description of the sampling procedure including cycle time calculations is provided in the Appendix (Table 1).

**PFI-Mill**

Pulp samples were refined in the PFI-Mill at three different refining intensities (4000, 7000 and 10,000 revolutions) according to ISO 5264-2. Samples of 30 g\text{ad} were prepared at a concentration of 10% (w/w), according to DIN EN 20638. Dilution was done with deionised water 24 h prior to disintegration according to ISO 5263-1.

Fines separation

Pulp samples without fines were prepared by use of a bench scale pressure screen, which is depicted in Fig. 6. To ensure a very sharp separation of only fines, the device is equipped with a 150 \( \mu \text{m} \) microperforated hole screen (see Fig. 7). This separation device is able to achieve a maximum fines removal efficiency of approximately 40% in one pass with the 150 \( \mu \text{m} \) screen (Mandlez et al. 2021). In order to

**Fig. 5** Process diagram of the disc refiner process

**Fig. 6** Bench scale pressure screen (inner body diameter is 240 mm) (Mandlez et al. 2020)

**Fig. 7** Micro perforated 150\( \mu \text{m} \) hole screen installed in the bench scale pressure screen (Screen dimensions: 65.5 mm \( \times \) 30 mm) (Mandlez et al. 2020)
achieve a fines content of at least less than 1% (w/w) it was necessary to operate the system in a closed loop for approximately 10–20 cycles.

Figure 8 shows the process diagram for the pressure screen operation. Pulp samples were diluted to a concentration of 1% (w/w) and were fed to the pressure screen at volume flow rate of 20 L/min. The accept containing the fines material was discarded. The reject containing the suspension with reduced fines content returned to the feed tank. Water that was lost with the accept stream was replaced by dilution water added to the pressure screen and the reject line. After each cycle, a sample of the accept was taken and its turbidity was determined visually. A nearly clear accept sample was used as an indicator, that the remaining fines content of the reject had approached the lowest achievable level. The fines content of the reject was examined according to SCAN-CM 66:05. If the fines content was still above 1% (w/w), the separation was continued. After fines separation, the pulp was thickened by use of a centrifuge.

Pulp properties

For each sample fibre morphology, fines content and drainability were analyzed with each measurement being repeated three times. To characterize fibre morphology, the length weighted fibre length distribution was determined according to ISO 16065-2 using a L &W Fiber Tester+ having a resolution of 3.3 μm/pixel. The measurement was carried out with a minimum particle detection number of 100,000 particles per sample. Furthermore, the total fines content was determined for each sample using a BDDJ according to SCAN-CM 66:05. Drainability of the pulp suspensions in terms of the Schopper-Riegler (SR) number was determined according to EN ISO 5267-1.

Handsheat preparation and properties

To test the resulting physical paper properties, 80 g/m² laboratory handsheets sheets were formed on a Rapid–Köthen sheet former using a closed water system according to ISO 5269-3. Especially when working with fines it is important to use white water recirculation in order to assure that a retention equilibrium of fines is attained in the formed handsheets (Giner Tovar et al. 2015). Therefore, the first four sheets were discarded.

Two sets of handsheets were prepared from each pulp sample, a standard set produced according to ISO 5269-2 and a second set including an additional wet-pressing step between sheet forming and vacuum drying in the Rapid Köthen sheet forming system. In order to produce the additional wet pressed handsheets, the formed sheets were couched with the topside onto a blotting paper (240 g/m²) and covered with another blotter (65 g/m²) on the wire side. Two rectangular shaped felts were prepared with the size of the blotter (blotter diameter equals felt edge length). The handsheet between the two blotting papers was pressed between the felts using a static hydraulic press. The sample was pressed 120 s at a pressure of 3010 kN/m² corresponding to 150 bar hydraulic pressure on the used pressing system. After this additional wet pressing step, the handsheets were again dried in the vacuum dryers of the Rapid Köthen sheet former according to ISO 5269-2.

Prior to physical testing, all handsheets were conditioned in accordance with ISO 187 (23 °C and 50% relative humidity) for at least 24 h. The apparent sheet density was measured according to ISO 534. The tensile index respectively the breaking length BL in km was determined according to ISO 1924-2. The breaking length BL is defined according to Eq. 2 (Töppel 1993) as the fraction between the breaking force \( F_B \) [N] and the product of sample width \( b \) [mm], grammage \( m_A \) [g/m²] and the gravitation \( g = 9.81 \text{ m/s}^2 \). Additionally the light scattering coefficient was determined according to ISO 9416.

\[
BL = \frac{F_B}{b \cdot m_A \cdot g} \cdot 10^3
\]  

(2)
Results and discussion

Following the results are presented and discussed, beginning with the change in fibre morphology due to refining and fines separation. Further on, the relation between apparent density and breaking length is shown. The main part is the quantification of the fines contribution on the strength development due to refining. Finally the dewatering behaviour of the different pulp compositions resulting from refining and fines separation is discussed.

Due to the large variety of sample configurations it is necessary to explain the color coding, symbols and naming conventions used within the presentation and discussion of the results (see Fig. 9). Not wet pressed samples (index: \( WP_0 \)) are presented in blue shades and the wet-pressed (index: \( WP_1 \)) ones in red shades. The lighter colours represents samples were the fines were removed (index: \( f_0 \)) and the darker colours samples including fines (index: \( f_1 \)). The symbol shape indicates the used refining equipment as well as the unrefined samples. The unrefined sampled (index: \( 0 \)) is marked with an circle, disc refiner with a diamond and the PFI-mill with a square. When addressing beaten samples in general (PFI and refiner) index \( R \) is used.

Morphological changes due to refining

Refining causes a change in the morphological properties of the pulp. Figure 10 depicts the cumulative length weighted fibre length distribution of the unrefined reference sample in comparison with the PFI-mill and the disc refiner at an equivalent breaking length of 7.1 km, each with and without fines. As expected the disc refiner results in more fibre shortening compared to the PFI-mill, which is expressed by the higher slope in the range between 1 mm and 3 mm in case of the disc refiner. The PFI-mill produces slightly less secondary fines compared to the disc refiner at the given breaking length. The fibre length distribution for the PFI-mill and the reference sample, each without fines, is nearly congruent. Therefore, it can be concluded that for the PFI-mill fibre shortening can be neglected. Both results for the PFI-mill were expected, as this
device is known for internal fibrillation being the predominant refining effect (Yasumura et al. 2008; Kerekes 2005; Wang et al. 2007; González et al. 2012; Gharehkhani et al. 2015).

The fibre length distribution for samples without fines are indicating a successful separation of the fines by the pressure screen. This is confirmed by the microscope images shown in Fig. 11. Image (Fig. 11a) shows the untreated reference pulp, where the fibre walls are undamaged and primary fines mainly consist of flake like material. After PFI-mill treatment with 10,000 revolutions the disruption of the fibre wall structure and the secondary fines produced are clearly visible in the microscope image (Fig. 11b). Moreover, no fines can be detected in the images of the samples after fines removal (Fig. 11c, d). Furthermore, images in Fig. 11c, d show the external fibrillation as effect of refining on the fibre wall. fibrillation due to refining with the PFI-mill can be clearly seen. This supports the earlier conclusion that the high amount of secondary fines in the PFI mill is a result of external fibrillation.

**Apparent density**

Apart from the morphological properties of the pulp suspension, the resulting paper properties are of further interest. Figure 12 shows the relation between apparent density and breaking length for the entire dataset. As expected, a strong linear relationship between these two properties is shown. However, this correlation is only valid for separate observation of not wet-pressed and wet-pressed samples. The removal of fines does not have an impact on the correlation in both cases.

It is obvious, that wet-pressed samples generally show a higher apparent density compared to the not wet-pressed samples. This is confirmed by the relation between apparent density and light scattering coefficient of the sheets, Fig. 13.

It can be concluded, that sheet densification due to refining basically leads to an increase in fibre RBA which then creates a higher breaking length (Rennel 1969; Ingmanson and Thode 1959). However, there are other mechanisms of densification involved when wet-pressing of sheets is applied. Therefore, it is necessary to discuss the relation between densification and breaking length in more detail.

To do so, the relation between apparent density $\rho$ and breaking length $\text{BL}$ is investigated separately for refining and wet-pressing. The development of the breaking length due to densification is calculated for both cases. The change in breaking length due to the change in density $\Delta\text{BL}/\Delta\rho$ is calculated according the Eqs. 3 and 4. Figure 14 is used to describe how they are derived.

Equation 3 represents the changes $\Delta\text{BL}/\Delta\rho$ due to refining (index: $0 \rightarrow R$) for not wet-pressed samples (index: $\text{WP}_0$). Therefore, all refining points (index: $R$) are referenced to the unrefined sample (index: $0$). The calculations are done separately for samples with and without fines.
The effect of wet-pressing (index: \( WP_0 \rightarrow WP_1 \)) on the increase in breaking length due to densification \( \Delta BL/\Delta \rho \) is derived by Eq. 4. In this case the changes are calculated for the corresponding data points for each refining configuration between the not wet-pressed (index: \( WP_0 \)) and the wet-pressed (index: \( WP_1 \)) sample.

\[
\left( \frac{\Delta BL}{\Delta \rho} \right)_{0 \rightarrow R, WP_0} = \frac{BL_{R, WP_1} - BL_{R, WP_0}}{\rho_{R, WP_1} - \rho_{R, WP_0}}
\]

The results for the breaking length increase due to densification are depicted in Fig. 15. The left part shows the influences of refining and the right part that of wet-pressing. One can clearly see, that the effect due to wet pressing on average is only half as much as that due to refining. This indicates, that densification by wet-pressing seems not to lead to the same amount of RBA as refining.

The fibre flexibilization is increasing with higher refining intensity. Hence, the conformability of the fibres increases and the fibre network is able to achieve higher density. Depending on the conformability a certain degree of RBA is generated in the sheet forming process, still with remaining voids in the fibre network. When it comes to wet-pressing, the sheet density increases as the void volume is reduced and therefore also the bonding area increases. However it seems that the increased conformability due to internal fibrillation in refining is more efficient in creating bonded area than wet pressing. Wet pressing also reduces the pore volume but apparently it is not...
creating much more contact area (see Fig. 16). The results are demonstrating that there are two different mechanisms active when it comes to creating network strength due to densification. The first one is densification due to fibre flexibilization (internal fibrillation in refining), which effectively increases bonding area and network strength. The second one is densification by network compression (wet pressing), which is less effective in terms of bonded area and network strength. This is in good accordance to Vainio and Paulapuro (2007). Note that fibre flexibilization is even promoting network strength due to wet pressing, as shown in the right part of Fig. 15 with refining the effectivity of network densification is much better than without refining, i.e. $\Delta BL/\Delta p$ is at least twice as large for refined pulp than for unrefined pulp. However, this effect seems reverse with higher refining intensities in case of the PFI mill. A possible reason might be, that the maximum density is approached.

Breaking length development

It has been shown in the literature multiple times, that fines play an important role in relation to strength properties (Herbert and Marsh 1968; Kibblewhite 1975; Retulainen et al. 1993; Töppel 1993; Ferreira et al. 1999, 2000; Garcia et al. 2002; Pruden 2005; Mayr et al. 2017). Therefore, Fig. 17 shows the breaking length over the mass based fines content for all samples. The unrefined pulp sample has a primary fines content of 5% (w/w). The fines content and breaking length increase with refining energy, for both refiners, as expected. The disc refiner shows up to approximately 8% (w/w) fines content for the highest used refining intensity of 250 kWh/t. In comparison, the PFI-mill produced approximately 2% (w/w) less secondary fines at an equivalent breaking length of 7.1 km. The PFI-mill however, has been investigated also at higher refining intensities, where a total fines content of 10% (w/w) was reached at 10,000 rev and a resulting breaking length of 8.9 km.

In order to quantify the influence of fines on the breaking length, fines were removed by the pressure screen described above. The separation results are depicted in Fig. 17 on the left side, where all samples show a fines content of less than 1 %wt. It already can be seen, that the breaking length decreases due to the fines removal. The quantification of the fines influence was determined in two ways which will be discussed in the following section.

Breaking length increase by secondary fines

The first approach to quantify the fines influence on strength properties is to calculate the breaking length increase by secondary fines formed during refining. For that, the results depicted in Fig. 17 are used for further analysis. Figure 18 is used as an example, in which one set of data points is highlighted, while all others are greyed out.

As a first step, the total change in breaking length due to refining $\Delta BL_R$ in km is calculated according to Eq. 5, i.e. the difference between the breaking length
Further on, the difference in breaking length of the corresponding refining points with fines $BL_{R,f_1}$ and without fines $BL_{R,f_0}$ corresponds to the change in breaking length by fines $\Delta BL_{f_0 \rightarrow f_1}$ in km which includes both primary and secondary fines (Eq. 6).

$$\Delta BL_{f_0 \rightarrow f_1} = BL_{R,f_1} - BL_{R,f_0}$$  

(6)

The change of breaking length due to primary fines $\Delta BL_{\text{prim}}$ in km is determined according to Eq. 7 by calculating the difference in breaking length of the unrefined samples with fines $BL_{0,f_1}$ and without fines $BL_{0,f_0}$.

$$\Delta BL_{\text{prim}} = BL_{0,f_1} - BL_{0,f_0}$$  

(7)

Based on the values obtained from Eqs. 5 to 7 we can calculate $\uparrow BL_{f_{\text{sec}}}$, the key parameter for the influence of fines on strength development, Eq. 8.

$$\uparrow BL_{f_{\text{sec}}} = \frac{\Delta BL_{f_0 \rightarrow f_1} - \Delta BL_{\text{prim}}}{\Delta BL_R}$$  

(8)

The idea of this parameter is that it describes how much of the total strength increase due to refining ($\Delta BL_R$) can be attributed to the presence of secondary fines (given by $\Delta BL_{f_0 \rightarrow f_1} - \Delta BL_{\text{prim}}$). Figure 19 depicts the results for breaking length increase by secondary fines for all refining configurations with and without wet-pressing. It is shown, that up to 25% of the breaking length increase is related to secondary fines in case of the disc refiner, while the remaining 75% are obtained by the fibre modification including internal/external fibrillation as well as primary fines. This is well above the result for the PFI-mill, where secondary fines contribute not even more than 12% to the breaking length increase at a very high refining intensity of 10,000 rev. This tendency was expected, as the PFI-mill, contrary to the disc refiner, is known for a good fibre flexibilization result. The high flexibilization leads to a good conformability in the fibre network and therefore to an already high RBA. The secondary fines are not able to improve the RBA as much as in the case of disc refining. However, it can be seen that breaking length increase obtained by secondary fines increases with refining intensity for both refining equipment. Therefore, it can be stated that fines formation becomes more relevant for increasing refining intensities. This is clearly more pronounced for the disc refiner. In conclusion it is shown that secondary fines are important for the breaking length increase, especially in case of the disc refiner.

The effect of wet-pressing on the importance of fines differs for the two refining equipment. In case of disc refining, the breaking length increase by secondary fines decreases due to wet pressing, while for the PFI-mill the opposite is true. However, for both refining types, the difference in breaking length increase between not wet-pressed and wet-pressed sheets, seems to become lower with higher refining intensities.

Fines efficiency on breaking length increase

Figure 20 shows again breaking length over fines content. Two corresponding points—one including fines (index: $f_1$) and the second without fines (index: $f_0$)—are highlighted, while the others are greyed out. The slope between these two points according to Eq. 9 defines the efficiency of total fines on the breaking length increase $\eta_f$. This value indicates how much km of breaking length BL are gained per mass percentage of fines $w$ including primary and secondary fines. In case of the unrefined reference
sample only the primary fines are included, as there are no secondary fines formed.

\[ \eta_f = \frac{BL_{f1} - BL_{f0}}{w_{f1} - w_{f0}} \]  

(9)

Figure 21 shows the fines efficiency breaking length increase over fines content. The fines impact on breaking length shown for the reference sample is based on the primary fines content, while for all other samples it is the total fines content including primary and secondary fines. The first mass percent of secondary fines formed show a clear and high increase for the fines efficiency for both refining equipment. However, there is a strictly different behaviour between disc refiner and PFI-mill at increasing refining intensities. In Fig. 21 we find a fines efficiency between 0.05 and 0.23 km/% (w/w). This means that for one weight percent of fines in the furnish an increase of breaking length between 0.05 km and 0.23 km has been found. While the total fines efficiency stays nearly constant at approximately 0.12 km/% (w/w) for the PFI-mill, the fines efficiency for disc refiner fines increases rapidly up to more than 0.20 km/% (w/w). As it was shown before in Fig. 19, the breaking length increase by secondary fines for the disc refiner is higher compared to the PFI-mill. As the amount of secondary fines formed in the disc refiner is comparably low in the investigated cases (see Fig. 21), it is straightforward, that the fines efficiency for creating breaking length is also higher for the disc refiner. Furthermore, the PFI-mill shows nearly no increase in the efficiency of the fines with rising refining intensity. This results from the comparably high amount of fines formed in combination with a low increase in breaking length obtained by secondary fines (see Fig. 19).

It is very interesting, that wet-pressing does not affect the fines efficiency significantly in case of the disc refiner. The PFI-mill shows a completely opposite behaviour when wet-pressing is used. The fines efficiency declines for rising refining intensities and approaches the same efficiency compared to not wet-pressed handsheets at 10,000 revolution.

It can be concluded, that independent of the secondary fines amount produced in the disc refiner, they have a drastically higher importance for the breaking length increase, compared to the PFI-mill. We think that this effect can not be attributed to the quality of fines (Kibblewhite 1975) itself, in our opinion the nature of PFI created fines is similar to the nature of disc refiner created fines. Instead we are inclined to think that the higher fines efficiency in disc refining can be attributed to the lower fibre flexibility, which leaves more potential for RBA increase due to the fines. However, the disc refiner creates a considerable amount of shortened fibres,
which is a known reason for reduced tensile strength properties (d’A Clark 1987).

Influence on dewatering resistance

Finally the behaviour of the dewatering resistance due to fines and refining intensity is investigated. Figure 22 depicts the dewatering resistance over the mass based fines content. The dewatering resistance experiences a progressive correlation to the fines formed in the refining process and reaches 30 SR for 10,000 rev in the PFI-mill. By removing the fines from the samples, the dewatering resistance is only influenced by the treated fibres. Without fines the dewatering resistance varies just between 10 and 16 SR, depending on the refining intensity. Therefore, it can be concluded that the fines produced in refining are the main reason for increased dewatering resistance (compare Cole et al. 2008) due to refining, while fibre flexibilization shows only a minor effect.

Conclusion

In this work we studied the effects of refining on the paper strength development, with focus on the differences between secondary fines production and fibre modification (i.e internal fibrillation, external fibrillation and fibre shortening). We have removed fines from refined pulp and compared the resulting loss in breaking length for different levels of refining and wet pressing.

For all data we found a strong relationship between breaking length and sheet density, reflecting an increase in bonding area and a corresponding rise in breaking length. However, a key result of our investigation was the finding that a sheet density increase due to refining creates two times more increase in breaking length than the same density increase created by wet pressing. The higher effect of sheet density on breaking length due to refining can be explained by the flexibilization of the fibres which are more conformable and thus create more bonding area than a sheet wet pressed to the same density.

Furthermore, the effect of fines formed in the refining process was investigated for disc- and PFI refining. It was shown that secondary fines resulting from the refining process are responsible for up to 25% of the breaking length increase in case of the disc refiner at 250 kWh/t, while it is only a maximum 12% for the PFI-mill at a comparably high refining intensity of 10,000 rev. This leads to the conclusion, that fines have a lower contribution to the paper strength, if the fibres are more flexible after refining as it is the case for the PFI-mill in comparison to the disc refiner. The disc refiner showed a considerable amount of shortened fibres which might influence the tensile properties. This might be a further reason for the higher

![Fig. 22 Fines content versus Dewatering resistance. Error bars indicate a 95% confidence interval](image)

Table 1 Parameters used for the disc refiner operation

|                        | $m_{\text{Pulp}}$ | $V_{\text{Tank}}$ | $V_{\text{Sample}}$ | $V$ | $t_{\text{Cycle}}$ | $\text{SEC}_\text{Target}$ | $\text{SEC}/\text{Cycle}$ | Cycles | $\tau_{\text{Op}}$ |
|------------------------|-------------------|-------------------|---------------------|-----|-------------------|-----------------------------|---------------------------|--------|-----------------|
| Disintegration         | 55.8              | 1430              |                     |     |                   |                             |                           |        | 30              |
| Dellaking              | 55.8              | 1430              | 190                 | 7.5 |                   |                             |                           |        | 2               |
| Refining               | 55.8              | 1430              | 190                 | 7.5 | 77.5              | 22.5                        |                           | 3.4    | 25.9            |
| Sampling at 100 kWh/t  | 7                 | 180               | 190                 | 7.5 | 100               | 22.5                        |                           | 0.1    | 0.9             |
| Refining               | 48.8              | 1250              | 190                 | 6.6 | 227.5             | 22.5                        |                           | 6.7    | 44.1            |
| Sampling at 250 kWh/t  | 7                 | 180               | 190                 | 6.6 | 250               | 22.5                        |                           | 0.1    | 0.9             |
attribution on strength development of secondary fines produced in disc refining.

Independent of the secondary fines amount formed, they have a drastically higher importance for the breaking length increase in disc refining, compared to the PFI-mill. For each additional mass percent of fines in the furnish an increase in breaking length between 0.1 and 0.23 km is achieved. It was shown, that the fines efficiency on the breaking length increase is approximately twice as high for the disc refiner. Therefore, secondary fines are of high importance for the strength development especially in case of the disc refiner. However, secondary fines are the main reason for increasing dewatering resistance due to refining, while fibre flexibilization plays only a minor role.

Acknowledgments We are grateful for the support by our industry partners in the frame of the FLIPPR project, Mondi, Sappi, Zellstoff Pöls AG, a member of Heinzle pulp, and Papierholz Austria. The K-Project FLIPPR is funded as part of COMET—Competence Centers for Excellent Technologies promoted by BMVIT, BMWFW, Styria and Carinthia. The COMET program is managed by FFG.

Author Contributions The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Funding Open access funding provided by Graz University of Technology. The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. Open access funding provided by Graz University of Technology. This study was funded by the Austrian Research Promotion Agency (FFG).

Data Availability The datasets generated and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Human participants This chapter does not contain any studies with human participants or animals performed by any of the authors.

Code availability Commercial software was used.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article’s Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article’s Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit http://creativecommons.org/licenses/by/4.0/.

Appendix

Refiner sampling procedure and calculation of cycle times

The pilot scale 12-inch single disc refiner was operated at an effective power $P_{\text{eff}}$ of 10 kW. The pulp suspension was fed under neutral pH conditions to the refiner with a volumetric flow $\dot{V}$ of 190 L/min at a consistency $c$ of 3.9% (w/w). According to Eq. 10 (Herbert and Marsh 1968), the SEC for a single pass through the refiner is 22.5 kWh/t.

$$\text{SEC} = \frac{P_{\text{eff}} \cdot \dot{V} \cdot c}{V}$$

In order to achieve the targeted refining intensities of 100 kWh/t and 250 kWh/t, the refiner is operated in a closed loop (see Fig. 5 in the main document). The necessary cycle time $t_{\text{Cycle}}$ for a single pass is calculated according to Eq. 11. The suspension volume in the feed tank $V_{\text{Tank}}$ at the beginning is 1430 l, which results in a cycle time $t_{\text{Cycle}}$ of 7.5 min.

$$t_{\text{Cycle}} = \frac{V_{\text{Tank}}}{\dot{V}}$$

Refining was carried out for a calculated operation time $t_{\text{Op}}$ in minutes (Eq. 13) based on the necessary amount of cycles according to Eq. 12. A summary of all calculated values e.g. cycle times for all process steps are shown in Table 1.

$$\text{Cycles} = \frac{\text{SEC}_{\text{Target}}}{\text{SEC}}$$

$$t_{\text{Op}} = t_{\text{Cycle}} \cdot \text{Cycles}$$
The first sample representing a refining intensity of 100 kWh/t is taken after 25.9 min (equally 3.4 cycles). At this time the pulp in the tank has is refined with an intensity of 77.5 kWh/t. As the sampling happens continuously at the refiner outlet the missing 22.5 kWh/t will be achieved during sampling. The sample size is 180 L and takes 0.9 min. After sampling the refining is repeated in in closed circle to achieve a refining intensity of 250 kWh/t with a reduced volume of 1250 L at the feed tank. Note, that the pulp in the feed tank actually experienced a refining intensity of 77.5 kWh/t. The procedure follows the rules, described for the first sample taken at 100 kWh/t. A summary of all parameters are shown in Table 1.

References

Cole CA, Hubbe MA, Heitmann JA (2008) Water release from fractionated stock suspensions. 1. Effects of the amounts and types of fiber fines. In: TAPPI Press-paper conference and trade show, PaperCon’08 3, pp 1412–1419

d’A Clark J (1978) Pulp technology and treatment for paper. Miller Freeman Publications, San Francisco. 0-87930-066-3

Eibinger K (2005) Entwicklung eines neuen Verfahrens zur energiesparenden und faserschonenden Mahlung cellulosischer Fasern. Ph.D. thesis, Graz University of Technology

El-Sharkawy K, Haavisto S, Koskenhely K, Paulapuro H (2006) Effect of fiber flocculation and filling design on refiner loadability and refining characteristics. BioResources 3(2):403–424. https://doi.org/10.15376/biores.3.2.403.424

Ferreira PJ, Matos S, Figueiredo MM (1999) Size characterization of fibres and fines in hardwood kraft pulps. Part Part Syst Char 16(1):20–24

Ferreira PJ, Martins A, Figueiredo M (2000) Primary and secondary fines from Eucalyptus globulus Kraft pulps. Characterization and influence. Paperi Puu-Pap Tim 82(6):403–408

García O, Torres AL, Comol JF, Pastor FI, Díaz P, Vidal T (2002) Effect of cellulase-assisted refining on the properties of dried and never-dried eucalyptus pulp. Cellulose 9(2):115–125. https://doi.org/10.1023/A:1020191622764

Genco JM (1999) Fundamental processes in stock preparation and refining. In: Tappi pulping conference, Tappi, Orlando, pp 57–96

Gharekhani S, Sadeghinezhad E, Kazi SN, Yarmand H, Badarudin A, Safaei MR, Zubir MNM (2015) Basic effects of pulp refining on fiber properties—a review. Carbohydr Polym 115:785–803. https://doi.org/10.1016/j.carbpol.2014.08.047

Giner Tovar R, Fischer WJ, Eckhart R, Bauer W (2015) White water recirculation method as a means to evaluate the influence of fines on the properties of handsheets. BioResources 10(4):7242–7251. https://doi.org/10.15376/biores.10.4.7242-7251

González I, Boufi S, Pélach MA, Alcalà M, Vilaseca F, Mutjé P (2012) Nanofibrillated cellulose as paper additive in eucalyptus pulps. BioResources 7(4):5167–5180. https://doi.org/10.15376/biores.7.4.5167-5180

Hartman RR (1985) Mechanical treatment of pulp fibres for paper property development. In: Papermaking raw materials, transaction of the VIIIth funding Res. symposia, Oxford, pp 413–442. https://doi.org/10.15376/frc.1985.1.413.

Herbert W, Marsh PG (1968) Mechanics and fluid dynamics in disc refiner. Tappi J 51(5):235–239

Higgins HG, de Yong J (1961) The beating process—primary effects and their influence on pulp and paper properties. In: The formation and structure of paper—transactions of the 2nd fundamental research symposium held in Oxford, September 1961, 1, pp 651–690

Ingmanson WL, Thode EF (1959) Factors contributing to the strength of a sheet of paper. II. Relative bonded area. Tappi J 42(1):83–93

Kang T, Paulapuro H (2006) Effect of external fibrillation on paper strength. Pulp Pap Canada 107(8):51–54

Kerekes R (2005) Characterizing refining action in PFI mills. Tappi J 4(3):9–14

Kerekes RJ, Schell CJ (1992) Characterization of fibre flocculation regimes by a crowding factor. J Pulp Pap Sci 18(1):32–38

Kibblewhite R (1975) Interrelation between pulp refining treatments, fibre and fines quality, and pulp freeness. Paperi Puu-Pap Tim 8:519–526

Koskenhely K, Ammalä A, Jokinen H, Paulapuro H (2005) Refining characteristics of softwood fibre fractions. In: The pulp and paper fundamental research society, pp 427–456

Luukko K, Paulapuro H (1999) Mechanical pulp fines: effect of particle size and shape. Tappi J 82(2):95–101

Mandl D, Zangl-Jagiello L, Eckhart R, Bauer W (2020) Softwood kraft pulp fines: application and impact on specific refining energy and strength properties. Cellulose 27(17):10359–10367. https://doi.org/10.1007/s10570-020-03467-1

Mandl D, Eckhart R, Bauer W (2021) Evaluation of fines separation from unbleached softwood kraft pulp using microperforated hole screens. Nord Pulp Pap Res J. https://doi.org/10.1515/nprrj-2020-0110

Mayr M, Eckhart R, Bauer W (2017a) Improved microscopy method for morphological characterisation of pulp fines. Nord Pulp Pap Res J 32(02):244–252. https://doi.org/10.3183/NPPRJ-2017-32-02-p244-252

Mayr M, Eckhart R, Thaller A, Bauer W (2017b) Characterization of fines quality and their independent effect on sheet properties. In: Transactions of the 16th fundamental research symposium held in Oxford, pp 299–322

Mutamedian HR, Haliholic AE, Kulachenko A (2019) Mechanisms of strength and stiffness improvement of paper after PFI refining with a focus on the effect of fines.
Nordström B (2016) Densification by wet pressing versus refining of never-dried high-yield softwood kraft pulp—effects on compression strength, tensile stiffness, and tensile strength. Nord Pulp Pap Res J 31(3):422–431. https://doi.org/10.3183/npprj-2016-31-03-p422-431

Odabas N, Henniges U, Potthast A, Rosenau T (2016) Cellulosic fines: properties and effects. Prog Mater Sci 83:574–594. https://doi.org/10.1016/j.pmatsci.2016.07.006

Osong SH, Norgren S, Engstrand P, Lundberg M, Reza M, Tapani V (2016) Qualitative evaluation of microfibrillated cellulose using the crill method and some aspects of microscopy. Cellulose 23(6):3611–3624. https://doi.org/10.1007/s10570-016-1068-x

Park S, Venditti RA, Jameel H, Pawlak JJ (2006) Hard to remove water in cellulose fibers characterized by high resolution thermogravimetric analysis—methods development. Cellulose 13(1):23–30. https://doi.org/10.1007/s10570-005-9009-0

Pruden B (2005) The effect of fines on paper properties. Pap Technol 46(4):19–26

Rennel J (1969) Opacity in relation to strength properties of pulps-3. Tappi J 52(10):1943–1947

Retulainen E, Moss P, Nieminen K (1993) Effect of fines on the properties of fibre networks. In: Products of papermaking 10th fundamental research symposium, pp 727–769

Sandgren B, Wahren D (1960) Part 3 influence of Crill on some properties of pulp and paper. Svensk Papperstid 63(24):879–883

Steenberg B, Sandgren B, Wahren D (1960) Part 1. Studies on pulp Crill suspended fibrils in paper pulp fines. Svensk Papperstid 63(12):395–397

Töppel O (1993) Physikalisch-technologische Prüfung. No. 3 in Prüfung von Papier, Pappe und Zellstoff, Springer, Berlin Heidelberg. ISBN: 3-540-55896-9

Vainio A, Paulapuro H (2007) The effect of wet pressing and drying on bonding and activation in paper. Nord Pulp Pap Res J 22(4):403–408. https://doi.org/10.3183/npprj-2007-22-04-p403-408

Wang X, Maloney TC, Paulapuro H (2007) Fibre fibrillation and its impact on sheet properties. Paperi Puu-Pap Tim 89(3):148–153

Yasumura P, D’Almeida M, Park S (2008) Refining actions in PFI mill and in industrial disc refiners. O Papel (Brazil) 69(8):63–72

Publisher’s Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.