## White Water Recirculation Method as a Means to Evaluate the Influence of Fines on the Properties of Handsheets

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Fines are an important factor in the papermaking industry with respect to their influence on the mechanical properties of paper. A procedure offering the possibility to produce handsheets with a constant amount of fines, as well as the determination of the fines content, is of great importance in evaluating the influences of different types of fines. In this work, a method based on a white water circulation system and fiber morphology characterization using a flow cell was evaluated. Three different wires for handsheet forming were studied (120-mesh, 325-mesh, and 500-mesh), and the 325-mesh wire was chosen for further trials. Using the 325-mesh wire, a constant amount of fines was achieved after discarding seven handsheets. This method allows reliable evaluation of the effects of primary as well as secondary fines and a cellulosic additive on handsheet properties.

Keywords: Primary fines; Secondary fines; Fines content; Fines retention; White water circulation; Handsheet forming; Paper properties

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## INTRODUCTION

Among the different components and fractions present in chemical and mechanical pulps, fines have been a focus of recent research. The effect of fines on paper formation and sheet properties has been widely analyzed (Retulainen *et al.* 1993; Seth 2003; Sirviö and Nurminen 2004). Compared to pulp fibers, fines have a large surface area (Peterson *et al.* 2001), which is also an important parameter affecting various pulp and paper properties. When it comes to the evaluation of their effects on handsheet properties, a defined amount of fines is required to allow accurate extrapolation of the results.

By definition of the Scandinavian Pulp, Paper, and Board test committee (SCAN-CM 66:05 (2005)), fines are the fraction of pulp that passes through a screen or a perforated plate with a hole diameter of 76  $\mu$ m, representing the 200-mesh screen of a fiber length classifier according to TAPPI Test Method T 261 Cm-94 (1994). These particles can be further divided into different categories, including fines from chemical pulps, fines from mechanical pulps, primary fines, and secondary fines. Mechanical pulps, for example, contain a large amount of fines (20% to 35% by weight) that have special characteristics and impart the sheet with high opacity and reasonable strength (Retulainen *et al.* 1993). For mechanical fines, Brecht and Klemm (1953) introduced the classifications 'Mehlstoff' and 'Schleimstoff,' meaning chunky particles of high lignin

content and low bonding ability and more fibrillar particles of high bonding capacity, respectively. Sundberg et al. (2003) suggested that the more fibril-like fines in mechanical pulp originate primarily from the primary and secondary fiber wall, while the more flakelike fines originate from the middle lamellae. Chemical pulps contain fewer fines than mechanical pulps. The fines content ranges from a few percent up to 10% to 12%, depending on the level of refining (Paavilainen 1992). The primary fines are formed during pulping and consist primarily of parts of the middle lamellae, ray cells, parenchyma cells, and debris from the fibers (Krogerus and Fagerholm 2002; Bäckström et al. 2008). They exhibit higher extractives and lignin contents compared to those of the rest of the pulp sample (Retulainen et al. 1993; Seth 2003). Once the pulp is refined, the newly formed fine materials produced are categorized as secondary fines. These are primarily parts of the primary and secondary walls of fibers peeled off as a result of mechanical impact. Their effect has been studied by several researchers in the past. Retulainen et al. (2002), for example, pointed out that a higher tensile strength in handsheets (kraft paper) could be obtained via the addition of 15% secondary fines, which were produced by refining the pulp for 2 h in a Valley beater. They observed changes in the sheet's density and optical properties and a higher anionic charge in the fines fraction compared to that of the long fiber fraction. The addition of secondary fines to the pulp was also shown to negatively affect the dewatering behavior, prolonging dewatering (Lindqvist et al. 2012).

The effects of secondary fines on parameters such as dewatering, tensile strength, and porosity differ from those of primary fines. Sirviö et al. (2003) and Sirviö and Niskanen (2008) demonstrated that secondary fines are usually of fibrillar nature, which leads to closer fiber contact and enhances fiber-fiber bonding, thereby increasing tensile strength. The positive impact of secondary fines on tensile properties was demonstrated to be higher compared to primary fines (Htun and Ruvo (1978)). Another study of the differences between primary and secondary fines was carried out by Xu and Pelton (2005). In this work, primary fines were presented as chunky particles acting as gaskets and increasing the contact area. They showed that the primary fines are less effective than secondary fines for this purpose. Secondary fines, however, yielded stronger adhesion than primary fines. Similar results and conclusions like those mentioned in the work of Xu and Pelton (2005) were presented by Sirviö and Nurminen (2004). Here, secondary fines were described as particles with a greater specific surface area, reducing thickness and porosity by filling voids between fibers. They also confirmed that there was a positive effect on tensile strength with the addition of fibrillar secondary fines to both chemical and mechanical pulps, as the fines brought fibers closer to each other within the fiber network. When the amount of kraft fines present in the handsheet exceeded 15%, a negative impact on the light scattering coefficient was recorded.

These previous studies show that fines have an important influence on various properties of paper, such as its thickness, tensile strength, dewatering, air permeability, and light scattering. To study the effects of different types of fines at the laboratory scale, the primary challenge is to retain a constant amount of fines in the formed handsheets. To achieve this, the following procedures can be applied:

• Forming the handsheets on filter paper using a suction filter, as is done in the preparation of laboratory sheets to measure the diffuse blue reflectance factor (ISO 3688 (1999))

- Forming the handsheets on a membrane (Sehaqui *et al.* 2010)
- Forming the handsheets using a white water recirculation system

Although the first two methods mentioned provide a defined amount of fines in each handsheet formed, based on retention of almost 100%, the third option is preferable because of two major drawbacks with the first two approaches. First, the small pore size of filter papers and membranes slows the dewatering of the pulp. Thus, sedimentation effects may occur during handsheet formation because the fines do not exhibit the same settling behavior as longer fibers. This behavior causes a distribution of fine material within the formed sheets that is not expected to occur in a handsheet former unit or in industrial processes. An example was presented by Sehaqui *et al.* (2010), who analyzed the formation of nanopaper structures using a semiautomatic sheet former. The formation of a microfibrillar cellulose (MFC) handsheet over a nitrocellulose ester filter membrane with 0.65- $\mu$ m pore size required a filtration time of around 45 min. This group also pointed out a second drawback: difficulty in separating the formed handsheet from the membrane without damaging the sheet surface. This is also a problem when using filter papers.

Before white water recirculation systems became relevant, other approaches, using a conventional handsheet former unit, were investigated. One of the first reported attempts to study fines' effect on sheet properties was carried out by Htun and de Ruvo (1978), in which a 300-mesh wire was used to form the handsheets. Some other studies, such as those presented by Sirviö *et al.* (2003), have determined the amount of fines retained in a sheet using a Britt dynamic drainage jar (BDDJ) (Sirviö and Nurminen 2004). Chen *et al.* (2013) also studied the effects of adding fines to a high-yield pulp. In this case, 5%, 10%, 15%, or 20% fines were added to the pulp before handsheets were formed using white water recirculation to retain the fines. All of these attempts have one particular problem in common. It is impossible to determine whether each formed handsheet retained the same amount of fines. This makes evaluation of certain fines effects on paper properties inaccurate.

Using a white water recirculation system can, however, be a solution for the previously mentioned drawbacks. Sedimentation effects are avoided. The fines not retained during sheet formation circulate within the white water system, allowing steady-state fines content to be reached after some handsheets are formed. A method that easily indicates whether the amount of fines in the handsheets has reached this steady-state is of great importance. Once this is accomplished, the handsheet properties can be measured. Bäckström *et al.* (2008) used a similar approach as the one presented here. They tried to achieve a constant fines content by producing and discarding 10 handsheets before sheets for mechanical testing were formed. In the study conducted by Lindqvist *et al.* (2012), the method chosen was the one suggested by the Scandinavian Pulp, Paper and Board Committee (SCAN-CM 64:00 (2000)), which also uses a white water recirculation system and links the amount of fines retained in the handsheets with the dewatering time. In this case, the dewatering time must remain constant even when preparing additional sheets.

In the present study, a method that would ensure a defined amount of fines in each handsheet formed, as well as easy and reliable determination of the fines content, was developed. Determination of the fines content is necessary for each trial, as different fiber and fines properties and furnish recipes can result in a varying number of sheets required to achieve steady-state fines content. The method should also require less effort and only a small amount of fines compared to the other options mentioned. This is crucial, especially if the effects of special types of fines on handsheet properties are to be studied, as their production could be tedious. The fines content is determined by means of L&W Fiber Tester Plus measurements, which are intended to be a substitute for BDDJ tests, as mentioned by Sirviö and Nurminen (2004). Although determination of the fines content using BDDJ tests is rather precise and simple, it is a time-consuming step. Fiber morphology measurements using a flow cell would facilitate the laboratory work and reduce the amount of pulp needed to determine the fines content (5 g when using the BDDJ as compared to 0.15 g when using the L&W Fiber Tester Plus). To reduce the effort required (*i.e.*, the amount of fines needed), a comparison of lab sheets formed with wires of three different mesh sizes (120, 325, and 500) was carried out. The influences of primary and secondary fines and a third cellulosic additive on the properties of lab handsheets are shown as exemplary results.

### EXPERIMENTAL

All tests were performed using a flash-dried, bleached softwood kraft pulp (a mixture of spruce and pine). Using this pulp, several samples were prepared for investigation. Primary fines were separated from the pulp according to SCAN-CM 66:05 (2005) using the Britt dynamic drainage jar. Originally, this standard was used to determine the amount of fines in the pulp, but it can also be used to separate fines from the pulp. The fines-free pulp was refined in a laboratory Hollander beater for 2 h to produce secondary fines. These fines were again separated from the refined pulp using the Britt dynamic drainage jar. The fines were collected in a beaker and left to settle. Afterwards, the dry solids content of the two fines suspension was determined, and the concentrated fines suspension was used on the trials. A third kind of fines material used in these trials was a commercially available cellulosic filler material.

The primary fines, secondary fines, and cellulosic filler used in the present study were analyzed using the L&W Fiber Tester Plus (Sweden) prior to their addition to the sheets. The Schopper Riegler number of each mixture was determined according to ISO 5267-1.

#### **Preparation of Handsheets using White Water Circulation**

A Rapid-Köthen (Germany) sheet former equipped with a white water recirculation system was used to produce handsheets with a grammage of  $60 \text{ g/m}^2$ . The mesh chosen was a key parameter, as it determined the amount of fines retained. Three different mesh sizes were used; 120-mesh (125-µm openings; standard mesh for sheet forming), 325-mesh (44-µm openings), and 500-mesh (25-µm openings) screens were used. The influence of the chosen mesh on the dewatering time was not an issue, as dewatering in the sheet former lasts only a few seconds and there was no considerable difference between the meshes.

In this experimental setup, the white water of each of the formed handsheet was stored in a separate tank and used during the formation of the following sheet. The fines content in the white water increased with every formed handsheet until a 'steady state' value was reached. At this point, every subsequent produced handsheet contained the amount of fines present in the feed suspension. Using this method, handsheets were formed and conditioned for 24 h. After conditioning, the thickness (DIN EN ISO 534 (2011)), air permeability (ISO 5636-3 (2013)), and breaking length (DIN EN ISO 1924-2 (2009)) were determined.

#### **Determination of Fines Content**

The amount of fines retained in each handsheet formed was determined using an L&W Fiber Tester Plus (based on morphological characterization). The L&W Fiber Tester Plus allowed for the measurement of key fiber morphological parameters such as the fines content. The fines content was defined as the arithmetic proportion of particles with lengths below 200  $\mu$ m.

### **RESULTS AND DISCUSSION**

The different fines used in this study were analyzed with the L&W Fiber Tester, and the obtained fiber length distributions are depicted in Fig. 1. From the figure, it is apparent that both kinds of fines and the cellulosic filler consisted almost exclusively of short particles below 200  $\mu$ m in size, as defined. Primary fines were larger, on average, whereas secondary fines had a lower average length. The cellulosic filler material had the highest proportion of particles ranging from a few microns to 100  $\mu$ m in size and was therefore considered the shortest portion of the studied fines.

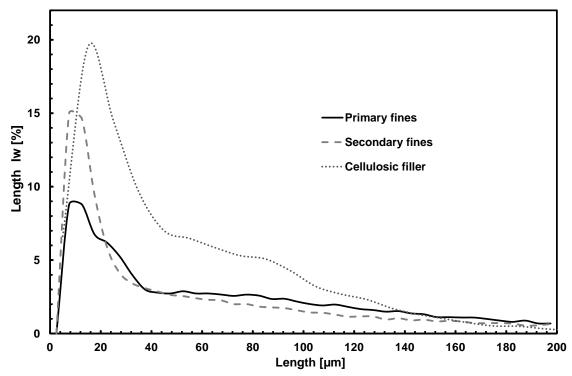


Fig. 1. Length-weighted length distribution of the fine materials used in the present study

After handsheet formation with three different meshes and subsequent L&W Fiber Tester Plus analysis, the fines content versus the number of sheets formed using white water recirculation was determined, and is represented by the arithmetic proportion of particles having lengths below 200 µm (Fig. 2). In this study, it was possible to determine the amount of fines retained in each handsheet formed, whereas in other studies no information is given. Besides, three different meshes were studied in order to evaluate the retention of fine material. As expected, the small pore size screen (500-mesh) retained more fines than the 120-mesh screen. As more handsheets were formed, the fines content increased until leveling out, achieving the so-called "steady-state". From Fig. 2, it is apparent that steady-state was achieved after three handsheets for the 500-mesh screen, five for the 325-mesh screen, and seven for the 120-mesh screen. Therefore, using a 500mesh screen would allow for the least number of handsheets to be discarded. In practice, the small openings of this wire make its application very difficult, as the high capillary forces between the sheet and mesh do not allow the sheet to be removed without damaging its surface to some extent, leaving the sheets useless for further evaluation. This was especially true for unrefined pulp samples with wet strengths lower than that of refined samples. For some refined pulp samples, the 500-mesh screen might be applicable in some cases, but one would have to complete respective trials for each sample beforehand, which makes its use impractical. For this reason, the 325-mesh screen was chosen for use in subsequent trials to determine the influence of primary fines, secondary fines, and a mechanically produced cellulosic filler material on the handsheet properties.

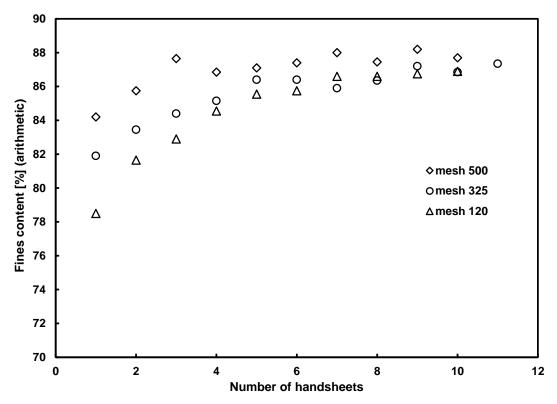


Fig. 2. Comparison between the fines retention and the mesh size

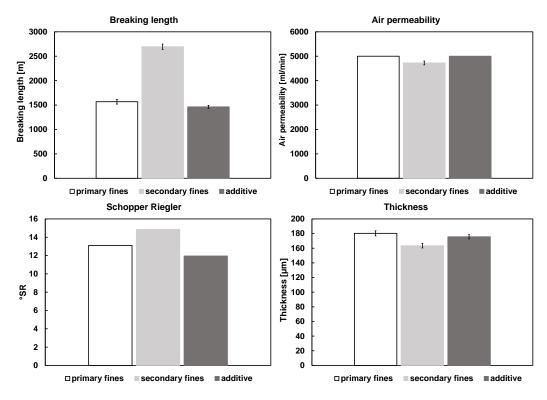
The results obtained using the 325-mesh wire are presented in Fig. 3. Three trials were performed based on the fines-free unrefined softwood kraft pulp and the addition of

primary fines, secondary fines, and the cellulosic filler material described above. The amount of fines added to the feed suspension was 4.8%, which corresponds to the amount of primary fines originally present in the pulp sample used. Handsheets were formed, and the weight, thickness, air permeability, dewatering, and breaking length were measured.

To ensure that the fines content was constant in the sheets evaluated, seven handsheets were discarded. The effects of the primary and secondary fines, as determined in this study, were in accordance with data found in literature. The addition of secondary fines resulted in higher sheet density with higher breaking length and therefore tensile strength, correspondingly lower porosity and slower dewatering (Fig. 3). This effect was explained by Bäckström et al. (2008), who claimed that the creation of higher capillary forces between the fines and the fiber surface improved the paper properties. This is in close association to the findings of Sirviö and Nurminen (2004), the presence of fines brings fibers closer together, decreasing porosity and air permeability. Compared to the primary fines and the cellulosic additive, this effect was more pronounced for secondary fines. Secondary fines are more fibrillar and have a higher charge content than primary fines, thus making them more effective in terms of sheet densification (Xu and Pelton 2005). According to Chen et al. (2013), this increases both the bonded area and the bond strength because they act as binders between long fibers. In a study conducted by Tao et al. (2007), the increase in tensile strength was approximately 5% when primary fines were added. Fines from refined pulp increased the tensile strength by almost 30%. The increase in tensile strength observed by Bäckström et al. (2008) was up to 30% when 10% secondary fines were added and 15% when primary fines were added. In the present work, the breaking length was 84% higher when secondary fines were added than when primary fines were added.

Comparison of the effects of primary fines and the cellulosic filler material revealed similar properties with slightly better dewatering achieved using the cellulosic filler. The mechanically produced cellulosic filler material was composed almost exclusively of fine particles, but it did have considerably higher width, according to the L&W morphology measurements. These more spherical particles may lead to higher porosity, which was not determinable for these samples because 5,000 mL/min is the upper limit of the measurement (Fig. 3).

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**Fig. 3.** Effect of primary fines, secondary fines, and mechanically produced cellulosic filler material on handsheet properties. The error bars represent the 95% confidence interval based on the results of 10 single measurements.

The pores were not plugged by this material as was the case with the primary and, even more so, secondary fines. This also explains the improved dewatering. From these results, one can conclude that the cellulosic filler material acted as a spacer within the sheet.

## CONCLUSIONS

- 1. The established method provides a constant amount of fine material in the handsheets and allows for easy determination of the fines content using an L&W Fiber Tester Plus. This procedure enables the evaluation of the effects of fines and other types of additives (*i.e.*, small particles comparable in size to fines) on various pulps and their properties.
- 2. Fewer handsheets must be discarded when the mechanical retention of the wire used is as high as possible. However, the 500-mesh wire could not be used for further trials because of high capillary forces, which damaged the sheet surface during removal of the wet handsheet from the wire. In practice, the 325-mesh wire was the finest wire applicable.
- 3. Secondary fines had the strongest effect on the breaking length. Primary fines and the cellulosic additive had similar influences on handsheet properties.

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