

FRACTIONAL PULPING OF TONER AND PIGMENT-BASED INKJET INK PRINTED PAPERS - INK AND DIRT BEHAVIOR

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Large visible dirt specks and microscopic ink particles are released from the surface of recycled paper when pulping toner and pigment-based inkjet ink printed papers. The applicability of pulp fractionation during laboratory scale pulping was investigated without chemicals to prevent the redeposition of small ink particles to fibers, and furthermore to break down the large toner fragments. Reference pulping was performed without fractionation stages, and the two pulping procedures were compared in terms of bound ink and dirt speck content. The results show that while the microscopic ink particles redeposit to the fibers at the very beginning of conventional pulping, the redeposition can be minimized by removing the detached ink particles from the vicinity of the fibers as soon as they have been detached. Thus, it is recommended to remove the ink from the pulp before a substantial defiberization level is achieved. In addition, the dirt specks broke down more efficiently in fractional pulping than in reference pulping at the same pulping consistency.

Keywords: Recycled paper; Pulping; Fractionation; Ink detachment; Ink redeposition; Dirt specks

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INTRODUCTION

Digital printing processes, such as electrophotography (toner printing) and inkjet printing, are gaining a higher and higher market share relative to conventional printing methods. In particular, the use of inkjet presses has grown steadily due to their exceptional print quality, flexibility, and ability to personalize documents (Ben and Dorris 2010). Digitally printed papers represent a valuable raw material source for deinking processes, but their large-scale utilization may cause quality issues in deinked pulp production.

In order to produce high quality deinked pulp out of digitally printed papers, the deinking treatments required for toner printed papers vary from those required for inkjet prints, due to the differences in ink compositions and printing methods between the two. Toner particles mainly consist of pigments to impart color, and thermoplastic resins to bind the pigment to the paper surface during fusing (Oittinen and Saarelma 2000). Due to the fusing step in printers, toner particles strongly adhere to the surface of paper. When toner-printed paper is disintegrated in the pulping process, relatively large toner fragments (up to 700 μm , some having fibers entrapped), easily remain in the pulp suspension (Johnson and Thompson 1995; Berg et al. 1997; Dorris and Page 1997). To remove these dirt specks in flotation efficiently, the entrapped fibers need to be released

from the toner particles, and a suitable size range for toner particles should be obtained (Johnson and Thompson 1995; Dorris and Page 1997). These requirements are more or less accomplished by utilizing a sufficiently long pulping time, high pulping energy, and/or costly dispersing stages (Borchardt and Lott 1995; Dorris and Sayegh 1997; Fabry et al. 2005).

Pigment-based inkjet ink, mainly consisting of very finely divided dispersed pigments in a vehicle (water, alcohol additives, and sometimes binders) (Oittinen and Saarelma 2000), appears to be the formulation used most in today's commercial inkjet solutions (Carré and Magnin 2002; Ben and Dorris 2010). When subjected to pulping, these inkjet printed papers do not create dirt speck problems (Faul and Oberndorfer 2010), but merely release tiny ink particles (as small as 0.1-0.3 μm in diameter) to the surrounding water phase, where they have a high tendency to redeposit inside the lumen of fibers (Ben and Dorris 2010). The redeposited ink cannot be removed in subsequent flotation or washing stages without substantial fiber losses, presenting a clear yield loss or quality problems for the processing of deinked pulp. In general, to prevent the excessive redeposition of the very small ink particles to fibers during pulping, a short pulping time, gentle pulping conditions, and highly alkaline conditions are required (Ben et al. 2000; Bennington and Wang 2001, Fabry et al. 2001; Ben and Dorris 2010). These are, however, contrary to the requirements set by toner prints if recycled papers are pulped, as they usually are, as a mixture.

A different approach, known as fractional pulping, could overcome the opposing pulping requirements set by toner and inkjet printed papers. According to recent studies (Kemppainen et al. 2010, K rkk  et al. 2011), fractionation of insufficiently defibered pulp after a very short pulping time, and continued pulping of the fiber fraction in the absence of a high ink load prevents the excessive redeposition of small coldset offset and flexo ink particles to fibers. Even if the benefits of fractionation during pulping have been shown earlier, it remains unclear whether this approach is advantageous with toner and inkjet printed papers. In particular, the degrees of ink detachment and dirt speck fragmentation are still ambiguous when fractional pulping is performed with toner and inkjet prints.

The aim of this study was to find out whether efficient ink detachment, prevention of excessive ink redeposition, and efficient dirt speck fragmentation can be achieved with toner and inkjet printed papers in non-chemical pulping conditions by fractionating the pulp slurry after a very short pulping period and continuing the pulping of the fiber fraction that is obtained. The pulping procedures were performed with strictly controlled, custom-made toner and inkjet prints. The results were compared with those obtained using a conventional pulping procedure without fractionation stages.

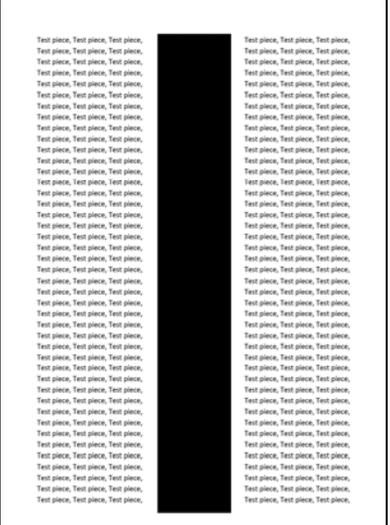
EXPERIMENTAL

Unprinted and Printed Papers

Uncoated wood free copy paper (Table 1) was used as the base paper in the experiments. A custom-made print (illustration in Table 1), including text in two columns and a rectangle between the columns, was applied on a single side of the base paper,

either with a black chemical toner (XEROX Black Toner 006R01317) or with pigment-based black inkjet ink (Epson DurabriteUltra waterbased ink T0711) using a Xerox WorkCentre 7132 or Epson Stylus SX218 printer, respectively. The area covered by the black ink was estimated to be 19.3% (found by counting the black-white pixel ratio) for the single-sided print. The toner printed papers were aged for 10 weeks (at ~22°C) prior to the pulping experiments, whereas the inkjet printed papers were not more than one week old. The experiments were performed with 100% toner printed papers and with a blend consisting of 80% toner and 20% inkjet papers, and also with unprinted papers.

Table 1. Specifications of Base Paper and Custom-made Print

Specifications of the base paper		Custom-made print	
Grade	Uncoated wood free copy paper		
Grammage (g/m ²)*	80		
Dry matter content (%)**	95.8		
Ash content at 525°C (%)***	24.3		
Ash content at 900°C (%)****	14.0		
* According to manufacturer's specifications ** SFS-EN 20638 *** ISO 1762 **** ISO 2144 Dry matter content and ash content were obtained using a Precisan PrepASH 129.			

Pulping and Fractionation Methods

Printed papers (50 printed papers representing approximately 233 g of oven dry (o.d.) paper) were shredded into pieces approximately 4 cm x 4 cm and put into a laboratory pulping vessel equipped with a flat beater (Kenwood laboratory pulper, whose operational principle is similar to the Hobart lab pulper used in the deinkability test in INGEDE Method 11). Deionized water (temperature adjusted to room temperature) was added among the paper pieces to adjust the pulp consistency to 16% (on o.d. paper basis). Pulping was performed with a constant rotational speed (speed 1 of the device) at room temperature (ca. 22°C), and without any chemicals.

Pulp fractionation was performed with a Sommerville device equipped with a 150-mesh screen. The procedure consisted of washing the pulp on a 150-mesh screen using 8 L/min water flow for 12 to 13 min for every 30 g of o.d. pulp. This fractionation procedure is commonly known as hyperwashing (HW), and it practically removes all the free ink, fiber fines, and inorganic particles from the pulp (see Fig. 1 for typical ash content decrease in the HW process), and can thus be considered an ideal fractionation method. The fibers (and flakes if present) that remained on the screen after washing were collected and thickened to 28-34% consistency in a Büchner funnel. The fiber fraction

(including flakes if present) was then diluted to 16% consistency with deionized water and pulped further for a given time period.

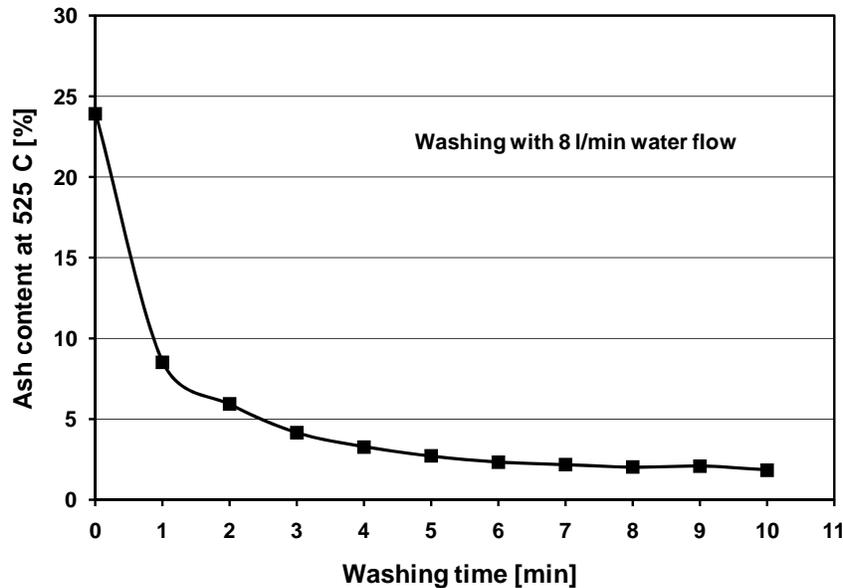


Fig. 1. Ash content of pulp made of unprinted paper as a function of washing time (water flow 8 l/min) in a Sommerville device equipped with 150-mesh wire screen. Residual ash (approximately 2%) is considered to be bound to fibers and thus not removable by the washing treatment.

Fractional Pulping and Reference Pulping Procedures

In fractional pulping, two parallel one-minute pulping stages were performed, after which the pulps were combined and fractionated. After the fractionation step (and thickening and dilution to 16% consistency), 230 g of o.d. pulp was directed to a subsequent one-minute pulping phase. Altogether eight one-minute pulping stages were performed to reach a total pulping time of 8 minutes. Fractionation was performed after each one-minute pulping stage (Fig. 2).

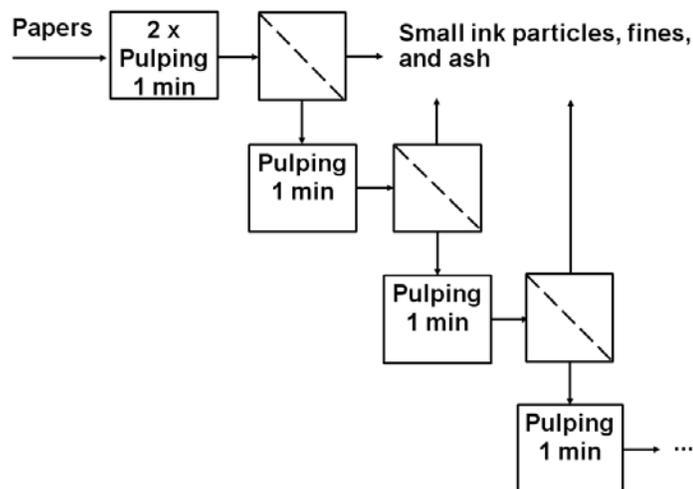


Fig. 2. A schematic drawing of the fractional pulping procedure

Fine material was released during the pulping stages as the defibering proceeded. In the fractional pulping, the fractionation stages diverted approximately 35 to 38% cumulatively of the released dry fine material into the fine fraction.

Reference pulping was performed without any fractionation stages and taking pulp samples after fixed time intervals. To facilitate the comparison between the reference pulping and fractional pulping, the amount of pulp in the reference pulping was adjusted after one-minute pulping periods to reach approximately the same pulping batch weight (o.d. pulp), as in the fractional pulping.

Determination of Residual Ink and Dirt Speck Contents

After each pulping stage (or after fixed time intervals in the reference pulping), an aliquot of the pulp (15 g o.d.) was taken and disintegrated in a milkshake blender (Waring GWB blender, V = 1 L) in three equal parts at 0.5% consistency, for 10 seconds, and using the “lo” rotational speed of the device. This procedure was necessary in order to make uniform handsheets from insufficiently defibered pulp. Part of the disintegrated sample (10 g o.d.) was hyperwashed on a 150-mesh screen using 8 L/min water flow for 10 min. Low grammage sheets were prepared in a sheet mold from disintegrated pulp and from hyperwashed pulp by filtering the diluted pulp samples through high retention filter paper (Munktell 00H, porosity 1-2 μm). The grammage of the sheets was adjusted (on a bone dry basis) to approximately 30 g/m^2 (disintegrated samples) or 40 g/m^2 (hyperwashed samples), to avoid exceeding the opacity limitation (<97%) required for light scattering coefficient determination. Two sheets were prepared from disintegrated pulp and three from hyperwashed pulp. A more detailed description of the sheet preparation procedures used is given elsewhere (Körkkö et al. 2010).

The scattering coefficient of the sheets was determined by measuring the reflectance of a single sheet against a black cavity and stack with a Lorentzen & Wettre Elrepho 070 spectrophotometer, and using $C/2^\circ$ illumination and an operating wavelength of 700 nm. The Residual Ink (RI, at 700 nm) content of the prepared sheets was calculated using the determined scattering coefficient and measured reflectance as described in TAPPI T 567 om-09 and ISO 22754. The RI content determined from the disintegrated (but not hyperwashed) pulp represents the total ink content (free and bound ink), whereas the RI content determined from the hyperwashed pulp represents the ink bound to fibers. It should be noted that the bound ink content is the sum of undetached and redeposited ink if ink redeposition is not prevented during pulping.

Residual ink measurement is sensitive both to the amount of ink particles in a sheet and also to the size of the ink particles, a higher amount and/or decreasing particle size (with constant ink load) resulting in a higher residual ink value (Haynes 2000). Since the mass of ink did not increase here during pulping for a given print type, the higher total ink content indicates a greater number of small ink particles in the pulp (i.e. ink fragmentation into smaller particles or release of tiny ink particles). Due to the ability of fiber material to reflect and scatter light, even a paper sheet that does not contain any ink particles gives a residual ink value in RI measurement. Thus, the RI content determined from sheets prepared after pulping unprinted papers represents the level where there is no ink present in the handsheet.

Optical inhomogeneities that are detectable with the naked eye, i.e. dirt specks, were determined with a DOMAS image analysis system equipped with an Epson 1680Pro scanner on the same three 40 g/m² sheets that were used for the bound ink content determination. Both sides of the sheets were scanned once per 225-cm² area using a scanning resolution of 1680 dpi. Objects that were 15% darker than the mean gray level of the background were identified as dirt specks. Since large ink particles >50 µm are visible to the naked eye as black or colored spots in the paper (Lassus 2000), the lower limit for dirt recognition was adjusted to 50 µm. Since the hyperwashing procedure, performed on a 150-mesh screen prior to sheet making, efficiently removes free particles smaller than 100 µm (open area in a hole is ca. 0.01 mm²), dirt specks from 50 µm to 100 µm (in the equivalent circle diameter) were considered to be bound to fibers. Dirt specks larger than 100 µm are either free particles too large to be removed by the washing treatment and/or bound to fibers, therefore the contribution of each is not discernible by this method.

The RI and dirt speck results are presented as mean values for two or three sheets, and the confidence interval for a 95% confidence level was assessed as ± 2.77 multiplied by the standard deviation of the test results (TAPPI T 1200 sp-00).

Determination of Flake Content

Separate reference pulping procedures without fractionation stages were performed with 100% toner printed papers and with unprinted papers. Samples (ca. 20 g o.d.) were taken after 1, 3, and 5 minutes of pulping, and the flake content of each pulp sample was determined with a Sommerville device equipped with a screen plate with 0.15 mm slits (TAPPI T 275 sp-02).

RESULTS AND DISCUSSION

Defiberization

When the 100% toner printed papers were pulped, the flake content was still 51.5% after 1 min of pulping, indicating insufficient defiberizing, in terms of conventional processing. As one of the aims of pulping in deinking is to produce flake-free pulp slurry so as to avoid fiber losses in the subsequent screening stages, pulping had to be continued to obtain a sufficient defiberizing level. After 3 and 5 min, the flake content was reduced to 3.9% and 1.3%, respectively, which can be considered close to full defiberizing. A somewhat lower flake content was attained with unprinted papers, namely 47.7%, 3.2%, and 0.1% after 1, 3, and 5 min of pulping, respectively, in agreement with earlier knowledge (Dorris and Sayegh 1997).

As will be shown later, the pulping time required for good defiberizing in conventional pulping was detrimental for optical properties when the recycled paper contained inkjet printed papers. However, impaired pulp quality was prevented by fractionating the pulp, before it was sufficiently defibered, into the fiber fraction (flakes and fibers) and the fine fraction (fines, fillers, and detached ink particles).

Behavior of Microscopic Ink

A percentage of 10% inkjet printed paper in a recycled paper mixture is considered the maximum that a deinking process can handle without problems in stock and process water quality (Carré and Magnin 2002; Ben and Dorris 2010). Thus the proportion of 20% inkjet papers used in this work represented a relatively strong challenge for the pulping stage operation.

As shown in Fig. 3, pulping of the 80% toner/20% inkjet mixture led to a significantly higher total ink content than pulping of 100% toner printed papers after the first minute of pulping, indicating a substantial release of small inkjet ink particles from fiber surfaces into the surrounding water phase. Further pulping with the 80/20 mixture released even more tiny ink particles, and the final total ink content became uniform at 1000 ppm after approximately 6 min of pulping. When the 100% toner printed papers were pulped, a relatively low and almost constant total ink content (304 to 349 ppm) as a function of pulping time was observed (Fig. 3). As residual ink measurement is considered to be most sensitive to ink particles smaller than 10 μm in diameter (Haynes 2000), some microscopic ink particles formed from the 100% toner printed papers (in agreement with Borchardt and Lott 1995) during the early stages of pulping, but not excessively when the pulping was continued up to 15 minutes. The result was expected, as the problems with toner printed papers are related to the release of relatively large ink particles rather than to the formation of microscopic ink (Dorris and Sayegh 1997).

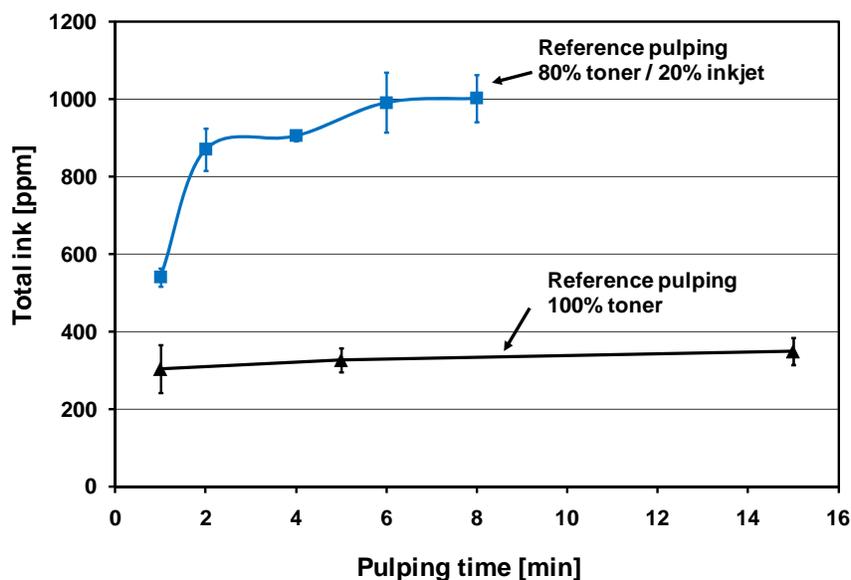


Fig. 3. Total ink content as a function of pulping time in conventional pulping when pulping 100% toner printed papers and an 80/20 mixture of toner/inkjet printed papers. The error bars represent the confidence interval for a 95% confidence level.

The presence of microscopic free ink particles will result in ink redeposition into fibers during conventional pulping (Ben et al. 2000; Bennington et al. 2001; Ben and Dorris 2010). Furthermore, the higher the load of free ink particles in the pulp suspension, the stronger the ink redeposition (Ben et al. 2000). Despite the fact that our

pulping equipment differs from that used in industry, our results are in agreement with earlier knowledge obtained with industry-type vat pulping equipment.

As shown in Fig. 4, a much higher bound ink content was observed with the 80% toner/20% inkjet mixture than with the 100% toner prints when the pulping was performed in a conventional way. The slight increase in bound ink content with the 80% toner/20% inkjet mixture would indicate that more ink was redeposited to the fibers than was detached from the fibers when pulping was continued from 2 min to 8 min. No increase in bound ink content as a function of pulping time was observed with 100% toner printed papers. Perhaps the reason behind this is that the quantity of microscopic ink particles present in the pulp suspension was not enough to show a clear sign of ink redeposition, and that throughout more ink was detached from 100% toner printed fibers than was redeposited to fibers.

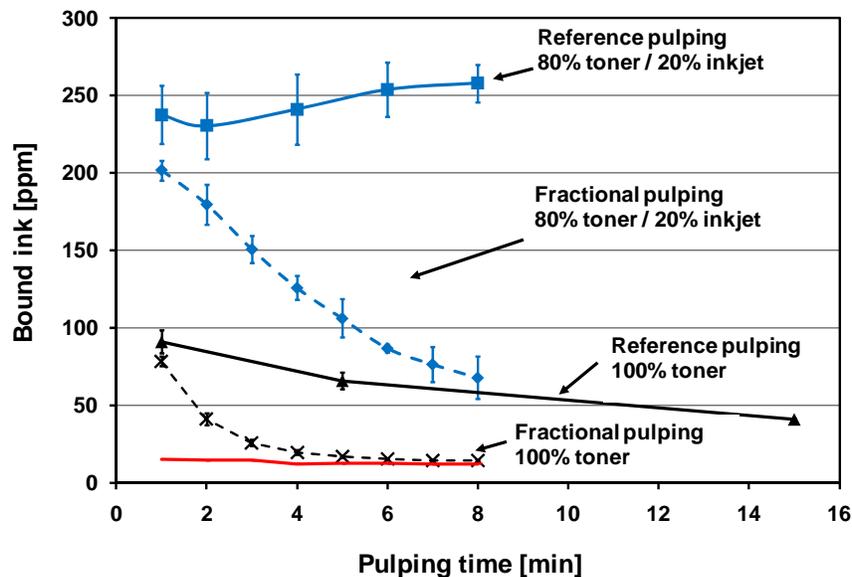


Fig. 4. Ink bound to fibers as a function of pulping time when pulping 100% toner printed papers, and an 80/20 mixture of toner/inkjet printed papers in a conventionally and with the fractional pulping approach. The red solid line on the bottom represents the hyperwashed residual ink content of unprinted paper which is free of ink. The error bars represent the confidence interval for a 95% confidence level.

The very detrimental ink redeposition to fibers can be avoided simply by removing the ink particles from the vicinity of the fibers as soon as they have been detached. This is shown with fractional pulping both with the 100% toner prints and the 80% toner/20% inkjet mixture (Fig. 4). The fractional pulping was based on semi-continuous ink removal during pulping (the detached ink particles were removed after each one-minute pulping period), which minimized ink redeposition to fibers, and allowed the attainment of a lower bound ink concentration than with a conventional pulping operation. The small difference in bound ink content after 1 min pulping between the pulping procedures with the toner/inkjet mixture is a consequence of the variation that naturally occurs when sampling very heterogeneous material.

The fractional pulping approach was beneficial not only with pigment-based inkjet printed papers but also with toner printed papers, as the bound ink content with the 100% toner printed paper quickly approached the level of that of unprinted paper (Fig. 4), which implies that all the ink was detached from the 100% toner printed papers in fractional pulping. Since the fractional pulping was continued only to reach a total pulping time of 8 minutes, it remains unclear whether perfect ink detachment could be achieved with the 80% toner/20% inkjet mixture when utilizing the fractional pulping strategy. Nonetheless, a significantly lower bound ink content was achieved with the fractional pulping approach than with conventional pulping when the raw material consisted of toner and inkjet printed papers (Fig. 4).

Based on the results presented above, the most important issue seems to be that the microscopic ink particles should be removed from the vicinity of the fibers as soon as they are detached, as proposed earlier by Kemppainen et al. (2010). Bearing in mind that the pulp can be in an insufficiently defibered form after a very short mixing time in the pulper (here the flake content was still around 50% after 1 min pulping, after which the first fractionation stage was performed), and that small ink particles are released into the pulp suspension as soon as the paper covering the ink matrix is broken down, the results presented here highlight the importance of ink removal from the vicinity of fibers before a sufficient defibering level is achieved. Furthermore, by preventing ink redeposition, a very low bound ink content can be achieved with the fractional pulping strategy even in the absence of deinking chemicals.

Dirt Specks

Figure 5 presents the area of dirt specks in the 50 to 100 μm range (considered to be bound to fibers) as a function of pulping time for conventional pulping and for fractional pulping with the two raw materials. A significantly lower dirt content was attained with the fractional pulping strategy, implying that the dirt specks were either more efficiently released from the fibers and/or more efficiently broken down into particles smaller than 50 μm with the fractional pulping strategy compared to the conventional pulping procedure. Similar results were also attained for larger dirt specks (Fig. 6). These results imply that the fractional pulping strategy was more efficient in breaking down small and large dirt specks and/or releasing fibers from the dirt specks than conventional pulping, when operating at the same pulp consistency.

To find out the reason behind the better dirt breakdown in fractional pulping, two significant disparities between the pulping procedures must be noted. Firstly, the presence of fractionation stages (washing stages) might have broken down dirt specks or weakened the cohesive strength of the toner particles between the pulping stages in fractional pulping. Secondly, even if the pulp consistency was kept constant (at 16%) during the pulping procedures, the fiber consistency was higher in fractional pulping (16%) than in conventional pulping (ca. 11%) due to the removal of fine material in the fractionation stages in the fractional pulping procedure. The fine material (fines and fillers) could have reduced the internal friction of the pulp (had a lubricating effect), leading to more gentle pulping conditions than pulping in the absence of fine material. Thus, the higher frictional forces present in pulping without the fine material could have broken down the dirt particles more efficiently.

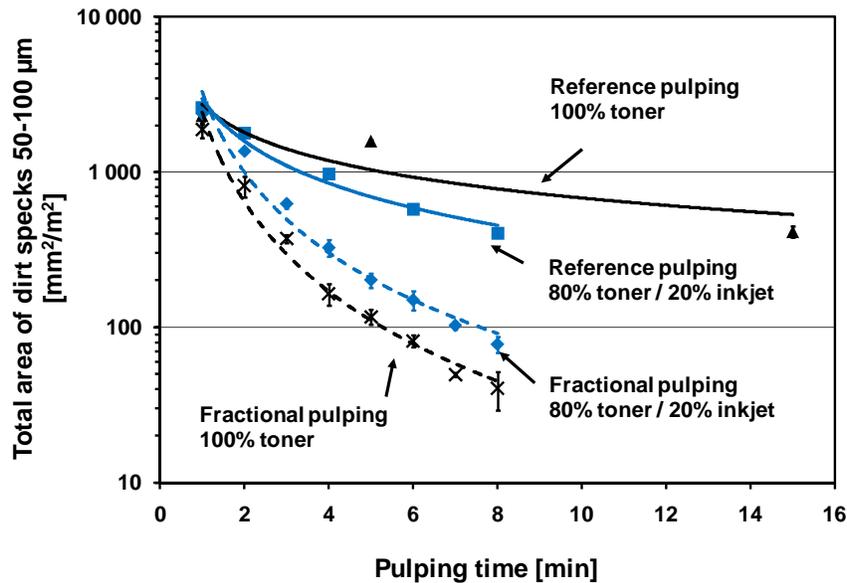


Fig. 5. The total area of small dirt specks (50-100 μm) as a function of pulping time when pulping 100% toner printed papers, and an 80/20 mixture of toner/inkjet papers conventionally and with a fractional pulping approach. The error bars represent the confidence interval for a 95% confidence level.

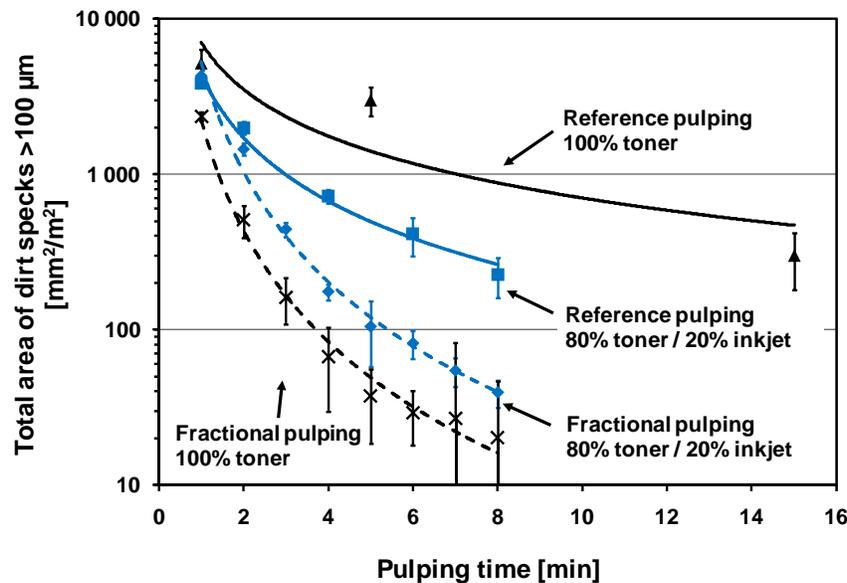


Fig. 6. The total area of large dirt specks (>100 μm) as a function of pulping time when pulping 100% toner printed papers, and an 80/20 mixture of toner/inkjet papers conventionally and with a fractional pulping approach. The error bars represent the confidence interval for a 95% confidence level.

In order to determine the capability of the washing stages to break down dirt specks, a short washing procedure (12 min washing representing the effect of a single fractionation stage in fractional pulping) or a long and harsh washing procedure (90 min

washing representing the effect of 7 fractionation stages) was performed on insufficiently or fully defibered pulp after 1 min or 8 min of pulping with 100% toner printed papers, respectively.

While both the short (12 min) and long (90 min) washing treatments broke up the dirt specks easily (especially large $>100\ \mu\text{m}$ dirt specks) from the insufficiently defibered pulp (Table 2), the dirt breakdown in both washing operations diminished significantly (down to non-existent for $>100\ \mu\text{m}$ dirt specks) when performed after the 8 min pulping period for fully defibered pulp. This result was to be expected, since dirt disintegration becomes more difficult the fewer dirt particles there are present in a pulp suspension.

However, the dirt speck contents after these pulping and washing combinations were significantly higher than those observed with the fractional pulping strategy (Figs 5 and 6), which implies that the harsh washing procedure does not satisfactorily explain the superior dirt breakdown observed in fractional pulping. Thus, a simple experiment was performed (consisting of 1 min pulping, 90 min washing, and continued pulping for 7 min) to reveal the effect of pulping in the absence of fine material. When the 1 min pulped and 90 min washed pulp was pulped further for 7 min (in the absence of most fine material, to reach a total pulping time of 8 min), the dirt breakdown was more efficient compared with 8 min pulping (in the presence of fine material) followed by 90 min washing (Table 2). Thus, it is likely that both the washing stages used and pulping in the absence of fine material affected dirt breakdown in fractional pulping, the effect of the washing being greater when performed in the early stages rather than in the late stages of fractional pulping.

Table 2. Total Area of Dirt Specks when Pulping and Washing 100% Toner Printed Papers

Procedure	Total area of dirt specks 50-100 μm [mm^2/m^2]	Total area of dirt specks >100 μm [mm^2/m^2]
Pulping 1 min	2914 (± 138)	4487 (± 647)
Pulping 1 min + washing 12 min	2210 (± 29)	2696 (± 561)
Pulping 1 min + washing 90 min	2251 (± 68)	2253 (± 532)
Pulping 1 min + washing 90 min, and continued pulping for 7 min	601 (± 19)	305 (± 75)
Pulping 8 min	940 (± 30)	633 (± 357)
Pulping 8 min + washing 12 min	849 (± 56)	545 (± 212)
Pulping 8 min + washing 90 min	814 (± 30)	451 (± 39)
The number in parentheses presents the \pm error at 95% confidence level.		

However, the dirt breakdown was not as efficient with the pulping and washing combinations studied as it was with the fractional pulping, which consisted of short pulping and washing cycles. Indeed, in fractional pulping the areas of both 50-100 μm and $>100\ \mu\text{m}$ dirt specks were very low ($<100\ \text{mm}^2/\text{m}^2$) after the 8-min pulping period

(including the 7 washing stages) (Figs. 5 and 6). Thus, the cyclic pulping and washing stages in fractional pulping may have had a stronger effect on dirt breakdown than a single long and harsh washing treatment in the middle of the pulping process (Table 2).

CONCLUSIONS

1. Fractionation stages embedded in the pulping process remove detached ink particles from the vicinity of fibers, and thus prevent the redeposition of small ink particles to the fibers, which occurs during the course of conventional pulping. Since the ink redeposition occurs as soon as the paper covering the ink matrix is broken down, the detached ink particles should be removed from the vicinity of the fibers before a sufficient defibering level is achieved. The fractional pulping procedure was seen to be beneficial especially when pulping a mixture of 80% toner/20% pigment-based inkjet printed papers.
2. By preventing ink redeposition, a low bound ink content can be attained with the fractional pulping strategy even in the absence of deinking chemicals. Within the pulping time range studied, 65 to 70% lower bound ink content was attained with the fractional pulping approach in comparison with conventional pulping.
3. Visible dirt specks generated by toner printed papers are more efficiently broken down in fractional pulping than in conventional pulping with the same pulp consistency. The reason for the superior dirt fragmentation was proposed, but not proven, to be partly due to the higher friction between fibers and dirt specks as a result of the absence of fine material in fractional pulping.

ACKNOWLEDGMENTS

The authors wish to acknowledge the financial support of the international GSCE graduate school. Also, the enthusiastic assistance during practical work, by Mr. Jani Österlund and Mr. Ari Pulkkinen, is greatly appreciated. Additionally, the authors wish to express their gratitude to Mr. Pasi Karinkanta for his technical assistance and Dr. Antti Haapala for helpful discussions concerning the manuscript.

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Article submitted: April 15, 2011; Peer review completed: May 26, 2011; Revised version received and accepted: June 20, 2011; Published: June 22, 2011.