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#### THE EFFECT OF RECYCLING ON THE FINES CONTRIBUTION TO SELECTED PAPER PROPERTIES

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#### ABSTRACT

The impact of fines from various sources on selected physical and mechanical properties of paper has been examined.

In the first of two experiments, the influence of fines was determined by producing two fines free pulps from furnishes which had been refined to 600 ml and 290 ml CSF. Fines removal had a detrimental effect on most properties at a given level of densification including: formation, in-plane and out-of-plane elastic properties, and normal span tensile strength. Densification either by refining, wet pressing, or fines addition resulted in an increase in sheet roughness; this is tentatively attributed to an increase in nonuniform shrinkage in the thickness direction of the sheet. Fines removal gave a more porous sheet particularly at the higher level of refining. Zero span strength or the ultimate strength of the sheet increased with sheet densification, being largely independent of how that densification was produced.

Fines type and addition level were investigated in the second set of experiments. Fines, up to a level of 30%, were added to a fines-free furnish 740 ml CSF. Primary fines are those present in an unrefined virgin pulp, and secondary fines are those produced by refining. "Primary" fines are those fines present after repulping recycled paper, and include both primary and secondary fines. "Secondary" fines are the fines generated by refining a fines-free "primary" pulp.

It was inferred, from drainage measurements, that the secondary fines had a greater hydrodynamic surface area and were, therefore, more effective than primary fines in enhancing sheet densification and some properties. Furthermore, "secondary"(H) fines, which had been produced from handsheets which had undergone more extensive wet pressing and drying, were, surprisingly, even more effective than the control fines and "secondary" fines. The behavior of newprint fines from preconsumer waste was similar to that produced by primary fines.

It is clear that fines, defined as material passing a 200 mesh screen, are inadequate to characterize their impact on paper properties. This agrees with the findings of Hawes and Doshi (16).

## INTRODUCTION

Important technical areas related to secondary fiber utilization include repulping, deinking, bleaching, and maintaining a specific product's converting and end-use performance when these sources of fiber are included in the furnish.

Softwood and hardwood pulps can generally be subdivided into a long fiber fraction and a fines fraction. This division is somewhat arbitrary, but, nevertheless, useful in determining the relative contributions of these fractions to the making of paper and its properties.

Fines are an important furnish component whose precise function is imperfectly understood, particularly with recycled furnishes. They may comprise as little as 1% or 2%, and as much as 30% of a well-beaten furnish. It is recognized that fines can have an adverse effect on water removal, while their impact on other properties is not well understood.

Recycled fines are presently regarded as filler material, which do not generally enhance paper properties. Deliberate removal or inadvertent loss of this material as waste can have a negative impact on the landfill problem.

Can the generation and treatment of fines be improved to obtain a better papermaking and end-use performance of the paper containing them? This an important question to be answered. It could be argued that it may not be practical, or cost-effective, to separate out fines, and or to provide for their special treatment. Nevertheless, it is possible that energy savings could be an incentive if the separate treatment strategies of long fibers and fines is demonstrated to be beneficial. However, before these latter kinds of questions can be considered, we need to better understand the role of fines and how they are affected by recycling.

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#### The Characteristics of Fines

Fines have been defined as the short fiber fraction, slime, flour, fiber debris, and crill. Some researchers have described fines as the pulp fraction which passes through a 200 mesh screen, while others have used a 150 mesh screen. The relationship between fiber length and screen size has been examined by a number of researchers, for example, the relationship between weighted average fiber length and screen size for a Bauer McNett classifier has been established by Tasman (<u>1</u>). Tasman recommends that a weighted average fiber length of 0.2 mm be used for the fraction passing a 200 mesh screen.

Htun and de Ruvo (2) characterized fines from a bleached kraft pulp according to size, morphology, chemical composition, swellability, physical structure, and mechanical properties. Using a Bauer McNett classifier, they showed that the fraction passing a 200 mesh screen had the greatest effect on mechanical properties as shown in Figure 1. This figure shows that the strength of the whole pulp is comprised of two contributions. The first is due to the contribution from the fraction <200 mesh and is dependent on refining level. Whether this is due to an increase in amount of the <200 mesh fraction and/or a change in the "bonding potential" of the fines is not known. This change in "bonding potential" may simply be a change in the size and shape distribution within this fraction, or that the colloidal fraction is the portion which is truly effective. In Htun and de Ruvo's work (2) the <200 mesh fraction had a higher water swellability and resulted in handsheets with a higher density and improved mechanical properties. The second contribution, which is also dependent on the level of refining, appears to be independent of the size of the coarse fiber fraction, i.e., in the range of 16 to 200 mesh. It is speculated that the strength improvement by refining for this contribution may be due to both internal and external fibrillation, as well as improved sheet formation.

The chemical composition of the fines was similar to the long fiber fraction; however, the crystallinity was lower. Their sorption isotherms also showed that there is a difference in cellulose water interaction between the coarse fiber fraction and the fines.

According to Mancebo and Krokoska (3), fines can be classified as being either primary or secondary. Primary fines are present in the pulp prior to refining, and secondary fines are produced during refining. Primary fines are identified as fragments of parenchyma cells, vessels, and the cell wall. Primary fines exhibit a higher lignin, ash, and extractive content than secondary fines or the whole pulp, and supposedly contribute little to bonding.



Fig 1 The effect of fines addition (<200 mesh) on the strength of paper made from a bleached kraft pulp beaten to different °SR (Taken from Htun and de Ruvo [2])

The level of primary fines present in a pulp is species dependent. Mancebo and Krokoska (3) found for three softwood pulps, the range of 0.7% to 6.2%; for a mixed hardwood, 7.3%; and for a partially depithed bagasse, 12.7%.

Mancebo and Krokoska (3) found that the rate and level of secondary fines generation during refining is dependent on species and pulp type. Fines generation for bleached NSSC and sulfite pulps is much greater than for an unbleached kraft pulp. Page's (4) proposal that sulfite pulps are generally more "brittle" than kraft pulps may be one explanation for the difference in rate and level of fines generation.

Sandgren and Wahren (5) used the term "crill" instead of secondary fines. They found that the amount of crill increases approximately linearly with refining time, and has a large influence on the drainage properties of the pulp. With crill removal sheet density decreased resulting in an increase in tear and a decrease in tensile strength. It was stated that the loss in tensile strength, attributed to a loss in density, could be compensated for by an increase in wet pressing, although no evidence was provided to verify this supposition.

Kibblewhite (6) examined the quality and quantity of fines prepared from Pinus Radiata kraft pulps. He concluded that the quality and quantity of fines strongly affected pulp freeness, but had little effect on paper strength.

Lobben (7) found that the fines from chemical pulps had a significant effect on strength properties depending on fiber type and the extent of refining. The effect of fines was greater for a eucalypt kraft pulp than for a pine kraft pulp with the effects being more pronounced when the long fiber fraction was unbeaten.

In a model study, Terao et al. (8) used a microfibrillated cellulose MFC and a crystalline cellulose (Avicel) to study the impact of fines on the structure and properties of paper. The MFC consisted of fibrils whose width was of the order of a few microns or less, whereas the Avicel consisted of particles in the size range of width 10-30  $\mu$ m and length 40-70  $\mu$ m. It was found that MFC increased sheet density and tensile strength, while Avicel had the opposite effect. It would be interesting to know whether this result was simply a particle size effect or differences in structure, i.e., relative crystallinity.

From one perspective (9), the main factors controlling the tensile strength of paper are shown in Figure 2. It is noted that for a given level of bonding, i.e., R.B.A. or apparent density, strength is controlled by interfiber bond strength, fiber strength, and fiber geometry. Densification by both combined refining and wet pressing can increase strength by increasing R.B.A. or apparent density. It is suggested that refining can lead to an increase in fiber modulus, strength, and a reduction in stress concentration.

It has been shown that refining does not increase interfiber bond strength when measured on isolated bond pairs. However, one of the major differences between isolated bond pairs and the bonds in a sheet of paper is that fines are present in the latter situation. Therefore, in paper, fines are not only expected to increase bonded area, but to reduce stress concentration as shown in Figure 2.

Nanko and Ohsawa (10) investigated the structure of interfiber bonding using transmission, scanning electron, and scanning laser microscopes. The secondary fines of beaten pulps were found to reside in the spaces between fibrils on the surface of the fiber. They termed the layer between the fibers a "bonding layer" which is made of secondary fines and external fibrils. It was conjectured that the bonding layer reduced stress concentration more than the S1 layer of the fiber.

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Fig 2 Strength Development by Refining and Wet Pressing

Therefore, it is hypothesized that the level of stress concentration will be governed by the size, shape, and bonding potential of the fines, all of which may be altered by recycling.

#### General Effects of Recycling on Pulp Properties

The general effects of recycling have been the subject of a recent review by Howard (<u>11</u>). One of the major consequences of recycling is to produce hornification, which is a loss in swellability, water uptake, and surface area, of both the long and fines fraction of a pulp. Other adverse effects can include contamination by inks, surfactants, and other materials, as well as "damage" to the long fiber fraction.

According to Howard (<u>11</u>), there are four ways to recover the lost potential of recycled pulps:

- \* Beating or refining
- \* Chemical treatment
- \* Blending with virgin pulp
- \* Fractionation

Recently, Scallan and Tigerstrom (<u>12</u>) have demonstrated, using predictions of the transverse fiber modulus, that hornification of the long fiber fraction can be reversed by refining. No hornification effects were evident in pulps above a yield range of about 70%.

Bhat et al. (<u>13</u>) used several techniques to enhance the strength of secondary fibers. They examined refining, high shear field refining (HSR), and/or alkali treatment. It was found that a combination of alkali treatment followed by HSR was most effective, and in some instances, the performance of the secondary fiber almost equaled that of the virgin pulp.

Ehrnrooth et al. $(\underline{14})$  examined the use of acetylation to reverse the effects of recycling. It was found that acetylation resulted in swelling and strength properties being comparable with those obtained using a never dried pulp.

This suggests that the effects of hornification are not completely irreversible, and may be reversed by external agents. The extent to which hornification of fines can be reversed has yet to be determined.

#### The Effect of Recycling on Fines

Mancebo and Krokoska (3) considered, as most researchers now agree, that changes in pulp properties with recycling are due to changes in fiber structure, i.e., hornification. Although changes in the long fiber fraction could be reversed by refining, it was stated that this was an ineffective treatment for the fines fraction; i.e., fines hornification was irreversible. No explanation was given for why fines hornification should be irreversible. Therefore, the fines fraction of recycled pulps could only be considered as a filler material, and possibly removed for use in other product applications. Furthermore, it was stated that the fines present in an unbeaten recycled furnish labeled "Primary fines" would consist of secondary fines from the previous cycle which would be irreversibly hornified. No speculation was made as to the equivalence of virgin secondary fines and those "secondary" fines generated by refining recycled pulps.

The contribution of primary, secondary, and a mixture of "primary" and secondary fines to paper strength has been illustrated by Mancebo and Krokoska (3). At a given level of fines addition, there is a larger contribution to strength from secondary fines, while the mixture, as might be expected, falls in between the extremes of secondary and primary fines.

Whether some form of chemical treatment, e.g., caustic, amine, ozone, or enzymes, might be used to activate or reverse the hornification of primary fines remains to be determined. However, it does suggest that the production of "primary" fines should be minimized, and this would in turn require minimizing the production of secondary fines. Mancebo and Krokoska (3) also reached this conclusion. In this endeavor we clearly need to know more about the behavior of secondary fines and what constitutes their optimum characteristics, i.e., size, shape, chemical nature, etc. It might also be possible to pretreat the furnish to ensure that fines do not undergo irreversible changes.

According to Mancebo and Krokoska (3), the fines fraction of recycled pulp has a negative impact on strength since they are only acting as filler material. The fines supposedly become inert due to "hornification," and the effect is irreversible even with refining!

Szwarcsztajn and Przybysz (<u>15</u>) also found that fines and fibers become hornified with recycling, and that strength properties decrease.

On the other hand, Hawes and Doshi (<u>16</u>) found that fines from recycled paper are effective in increasing paper strength. They examined the impact of primary and secondary fines from three pulp types (a 50% yield northern softwood kraft, an 80% softwood-20% hardwood recycled kraft pulp, and a southern pine/Virginia pine TMP) on an unrefined and a refined fines-free recycled unbleached kraft bag paper.

Using the data of Hawes and Doshi (<u>16</u>), the impact of fines type is shown in Figure 3. The level of fines added to the unrefined fines-free pulp was 20%, and the level added to the refined fines-free pulp was around 8.7%. We see that a 20% fines addition to the unrefined fines-free pulp results in an increase in densification and strength. The kraft and recycled fines are about equal in performance, while the TMP fines are much less effective. The recycled bag paper originally contained about 20% fines, but strength and density figures for this paper are not given. The influence of 8.7% fines addition on the fines-free refined fiber results in a reduction of sheet density, although there is a net increase in strength, at least for kraft and recycled fines addition. The reduction in sheet density is greatest for the TMP fines, and the change in strength is not significant. The reason for the reduction in sheet density with the introduction of fines into refined pulp, as shown in Figure 1, is not immediately obvious. This finding was noted, but not commented upon by the above authors.



Fig 3 Variation of tensile strength with apparent density based on data of Hawes and Doshi (16)

Separate treatment of the long fiber and fines fraction of pulps does not yet appear to have received consideration, although it seems clear that the respective treatments required may be very different. Furthermore, it does not appear that there have been any studies concerned with the effect of contaminants on fines performance, which is another important aspect of recycling. In this and future studies, we hope to pursue some of these issues.

#### EXPERIMENTAL DESIGN

It is hypothesized that recycled "primary" fines, comprised of both virgin primary and secondary fines, will behave like primary virgin fines. Furthermore, it is proposed that the secondary fines generated from refining recycled pulps are very similar to virgin secondary fines in their level of performance. An appreciation of these issues is considered to be important in selecting the appropriate treatment, e.g., refining, fractionation, etc., of recycled furnishes and their increased usage.

#### **Experimental Plan**

As a first step toward this understanding, two experiments have been performed. The first experiment is outlined in Figure 4. Its main objective is to determine the influence of virgin fines on selected paper properties by comparing the properties of a whole pulp with a fines-free pulp. Careful accounting of fines content and addition was essential, but not an easy undertaking in this work, since fines loss could occur during sheet forming, drying, and repulping.

The second experiment is shown in Figure 5. In this experiment, the performance of fines from different sources, i.e., virgin fines removed from a pulp beaten to a freeness of 290 ml CSF (control fines), recycled "primary" fines, recycled "secondary" fines, and fines from preconsumer newsprint waste, was investigated.

#### **Repulping Conditions**

In preliminary work, the conditions for repulping both preconsumer waste papers and recycled laboratory handsheets were determined. The papers were torn into approximately 1" x 1" squares and soaked overnight in water at 20° C. These were then repulped in a British disintegrator for a specific time  $t_D$  at a consistency of 1.4%. The disintegration time was the time necessary to achieve a handsheet which was free of knots and fiber bundles. The results are shown in Table 1. As might be expected, the disintegration times and fines content, i.e., material passing through a 200 mesh screen, vary over a fairly wide range.



Fig 4 Outline of experiment 1



Fig 5 Outline of experiment 2

## TABLE 1 REPULPING TIMES, FREENESS, AND FIBER FRACTIONS FOR SOME COMMERCIAL PAPERS AND HANDSHEETS

| CONDITION                              | CSF | > 14 | 14 > 28 | 28 > 48 | 48 > 200 | > 200 |  |  |  |  |
|--|-----|------|---------|---------|----------|-------|--|--|--|--|
| SACK KRAFT                             |     |      |         |         |          |       |  |  |  |  |
| t <sub>D</sub> = 30 min                | 680 | 54.6 | 17.9    | 13.6    | 7.2      | 6.7   |  |  |  |  |
| NEWSPRINT                              |     |      |         |         |          |       |  |  |  |  |
| t <sub>D</sub> = 15 min                | 180 | 12.9 | 18.2    | 21.6    | 19.6     | 27.7  |  |  |  |  |
| t <sub>D</sub> = 30 min                | 130 | 13.4 | 18.2    | 21.2    | 18.5     | 28.7  |  |  |  |  |
| TISSUE                                 |     |      |         |         |          |       |  |  |  |  |
| t <sub>D</sub> = 2 min                 | 700 | 50.3 | 25.6    | 13.1    | 6.9      | 4.1   |  |  |  |  |
| t <sub>D</sub> = 30 min                | 650 | 44.5 | 27.6    | 12.8    | 6.7      | 8.4   |  |  |  |  |
| NOBLE AND WOOD HANDSHEETS (290 ml CSF) |     |      |         |         |          |       |  |  |  |  |
| t <sub>D</sub> = 2 min                 | -   | 57.4 | 13.9    | 13.2    | 7.0      | 8.5   |  |  |  |  |
|  |     |      |         |         |          |       |  |  |  |  |

#### **Refining and Fines Separation**

The pulps used in this study were a bleached kraft southern pine market pulp and a preconsumer newsprint made from recycled fiber. The characteristics of the repulped newsprint are those shown in Table 1. The bleached kraft pulp was refined in a valley beater, following TAPPI recommended procedures, to a freeness of 600 ml CSF and 290 ml CSF.

A Bauer-McNett classifier was used to characterize the long fiber and fines fraction of the pulp using 14, 28, 48, and 200 mesh screen sizes, according to TAPPI recommended procedures. Two determinations were made for each condition. The fines are defined as material passing through a 200 mesh screen.

To obtain a fines-free pulp and to collect fines, the method of Hawes and Doshi (16) was used. A 10g (OD) sample of pulp was placed on the vibrating screen of a Sweco

Dynoscreen Separator and washed with water until approximately 20 liters of filtrate was

collected. The fines-free pulp was collected from the screen and stored in the cold room. The filtrate was allowed to settle at room temperature for 48 hours, after which the clarified water was decanted off. The fines concentration was in the range of 0.05 to 0.2%. Using this procedure, fines-free pulp at 600 ml CSF and 290 ml CSF was produced for the experiments shown in Figure 4.

In the second experiment as shown in Figure 5, the performance of various types of fines was determined. The nomenclature used for these fines is given in Table 2.

#### TABLE 2 FINES NOMENCLATURE

| Control Fines:  | fines separated from pulp after it has been refined to 290 ml CSF.   |  |  |  |
|---|--|--|--|--|
| "Primary Fines"   | fines separated from repulped handsheets made from a pulp refined to 290 ml CSF.   |  |  |  |
| "Secondary Fines"   | fines separated from fines-free repulped handsheets refined in a PFI mill to 190 ml CSF.   |  |  |  |
| "Primary Fines"(H)  | same as "Primary Fines," but the handsheets were subjected<br>to a higher level of wet pressing and were further dried in an<br>air circulating oven at 105° C for one hour. |  |  |  |
| "Secondary Fines"(H) same as "Secondary Fines," but the handsheets were<br>subjected to a higher level of wet pressing and were further<br>dried in an air circulating oven at 105° C for one hour. |  |  |  |  |
| Newsprint   | fines removed from repulped preconsumer newsprint waste.   |  |  |  |

The control or secondary fines were the fines obtained by screening the bleached kraft softwood whole pulp which had been refined to a freeness of 290 ml CSF.

The "primary" or recycled fines were derived from handsheets made from the bleached

kraft softwood whole pulp refined to a Canadian standard freeness of 290 ml. These handsheets were produced at a low level of wet pressing and full restraint during drying.

In preliminary experiments, it was found that the amount of fines recovered during repulping was dependent on the level of wet pressing used. Presumably, as bonding increases, "primary" fines recovery diminishes, and the performance of these fines, and the recycled "secondary" fines generated, may vary. Therefore, a second set of handsheets were subjected to a high level of wet pressing and after restrained drying, were further dried in an air circulating oven at 105°C for 1 hour. The fines from this second set of repulped handsheets are designated as "primary"(H) in Table 2. Sources of "Primary" and "Primary"(H) are shown as Primary recycled in Figure 5.

After repulping and removal of the "primary" or "primary"(H) fines, the fines-free recycled pulp was refined in a PFI mill for 5,500 revolutions yielding a Canadian standard freeness of 190 ml. After screening, these fines were designated as "secondary" or "secondary"H as appropriate.

#### Handsheet Making

All handsheets in this study were made on a Noble and Wood former and had, unless otherwise stated, a nominal grammage of 60 g/m<sup>2</sup>. The handsheets were wet pressed at different levels and dried for 30 minutes at 100°C under full restraint using the IPST press and dryer combination.

In the first set of experiments, handsheets were made from the whole pulp and fines-free pulp at two levels of refining and three levels of wet pressing.

In the second series of experiments, fines performance was determined at addition levels of 10%, 20%, and 30%. These were added to the fines-free pulp having a Canadian standard freeness prior to fines removal of 600 ml CSF (720 ml CSF after fines removal). In order to maintain a grammage of 60 g/m<sup>2</sup>, an adjustment was made, through trial and error, to compensate for fines loss during sheetmaking. The effect of fines on pulp drainage was assessed by measuring the sheet mold drainage time.

The resulting handsheets were subjected to a low level of wet pressing and then dried for 30 minutes at 100°C under full restraint using the IPST press and dryer combination.

#### Handsheet Testing Procedures

Nondestructive measurements included grammage, hard and soft platen (<u>17</u>) caliper, inplane and out-of-plane elastic constants, formation, and porosity. The elastic constants were made using ultrasonic wave propagation techniques developed at IPST (<u>18</u>), (<u>19</u>). Formation measurements (optical and mass density) were made using the IPST formation tester (<u>20</u>). The Parker Print Surf tester was used to measure porosity.

Destructive tests included normal span tensile properties and zero span strength.

#### **RESULTS AND DISCUSSION**

Bauer McNett classification of the whole and fines-free pulp, the repulped wet sheet after sheetmaking, and the repulped dried handsheet are shown in Table 3.

| SCREEN SIZE  | >14 % | 14-28<br>% | 28-48<br>% | 48-200<br>% | <200<br>% | FINES<br>LOSS<br>% |  |  |  |  |
|--|-------|------------|------------|-------------|-----------|--------------------|--|--|--|--|
| WHOLE PULP C.S.F. 600 ml FINES-FREE PULP C.S.F. 740 ml |       |            |            |             |           |                    |  |  |  |  |
| WHOLE PULP   | 63.1  | 13.6       | 104        | 5.8         | 7.1       | -                  |  |  |  |  |
| FINES-FREE PULP  | 66.3  | 14.8       | 11.7       | 6.6         | 0.6       | 6.5                |  |  |  |  |
| WET SHEET  | 64.6  | 13.9       | 10.6       | 5.9         | 5.0       | 2.1                |  |  |  |  |
| REPULPED SHEET   | 66.5  | 13.1       | 11.1       | 5.5         | 3.8       | 1.2                |  |  |  |  |
| WHOLE PULP C.S.F. 290 ml FINES-FREE PULP C.S.F. 700 ml |       |            |            |             |           |                    |  |  |  |  |
| WHOLE PULP   | 56.8  | 12.6       | 10.5       | 5.9         | 14.2      | -                  |  |  |  |  |
| FINES-FREE PULP  | 67.5  | 11.9       | 12.4       | 8.0         | 0.2       | 14.0               |  |  |  |  |
| WET SHEET  | 58.9  | 12.7       | 11.1       | 7.8         | 9.5       | 4.7                |  |  |  |  |
| REPULPED SHEET   | 57.4  | 13.9       | 13.2       | 7.0         | 8.5       | 1.0                |  |  |  |  |

TABLE 3 BAUER McCNETT CLASSIFICATION RESULTS

The fines content of the whole pulp at 600 ml and 290 ml was 7.1% and 14.2%, respectively. It should be noted that these fines include a small percentage of primary fines. We note that the Sweco screening technique was quite effective in removing fines with less than 1% fines remaining, there is also a concomitant increase in C.S.F. Sheetmaking results in about a 30% loss of fines, while drying and repulping involve a further small loss.

#### **Results and Discussion of Experiment 1**

The variation of apparent density, based on soft platen caliper measurements, with press load is shown in Figure 6. For a given press load, refining increases sheet densification. Fines removal lowers sheet density presumably due to a reduction in Campbell's forces.



Fig 6 Dependence of sheet densification on wet press load and the effect of refining and fines removal

Measurements of sheet formation are shown in Figures 7 and 8. It is noted, for the measurements based on mass density, that the formation index %CV(W) (coefficient of variation of mass density) is unaffected by refining or densification by wet pressing. However, fines removal produces a significant deterioration in formation. It has been found by Waterhouse (20) that sheet formation is improved by refining and wet pressing. In that work, sheets were made on a dynamic sheet former where drainage effects are not as important, as in the present case. Nevertheless, this does not explain why densification by wet pressing should not improve formation.

Formation measurements using transmitted light show a similar trend, i.e., fines removal results in poorer formation. However, the sheets produced from the pulp beaten to 600 ml CSF show an improvement in formation with densification, while those made from the pulp beaten to 290 ml CSF show a deterioration.

The variation of sheet roughness, based on the increase in hard caliper with respect to soft caliper, as a function of sheet densification is shown in Figure 9. The increase in roughness may be similar to that found by Pikulik and McDonald (21). It appears that the sheet with more refining and fines present better replicates the wet press felt in the present case blotter stock. Part of the contribution may also simply be due to greater nonuniform shrinkage in the thicknesss direction of the sheet.

Porosity is also dependent on sheet structure. Its variation with densification by refining and wet pressing is shown in Figure 10. As one would expect, fines removal should result in a more open sheet, and this is particularly true for handsheets made from the pulp refined to 290 ml CSF. Similar changes occur at 600 ml CSF, but are less dramatic.

We now examine some of the mechanical properties to determine how they are affected by refining, wet pressing, and fines removal. The elastic properties are shown in Figures 11 and 12, and the tensile properties in Figures 13 and 14. Both the in-plane specific elastic modulus and tensile strength form an envelope with respect to densification by refining and wet pressing which has already been previously discussed (9). With fines removal, the envelope is minimized but not eliminated.

We have found previously that the variation of out-of-plane specific modulus with sheet densification is largely independent of whether the increase in density is produced by refining or wet pressing (9). A similar trend is shown in Figure 12; however, there is a small deviation for the sheets made from the pulp refined to 290 ml CSF. Interestingly, fines removal does result in a loss of out-of-plane specific modulus.

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Fig 7 Effect of refining and fines removal on formation index %CV(W) (based on mass density measurements) variation with apparent sheet density



Fig 8 Effect of refining and fines removal on formation index %CV(T) (based on transmitted light measurements) variation with apparent sheet density



Fig 9 Variation of sheet roughness with apparent density and the effect of refining and fines removal



Fig 10 Variation of sheet porosity with apparent density and the effect of refining and fines removal



Fig 11 Influence of refining and fines removal on the variation of in-plane specific elastic modulus with apparent sheet density



Fig 12 Influence of refining and fines removal on the variation of out-ot-plane specific elastic modulus with apparent sheet density



Fig 13 Influence of refining and fines removal on the variation of in-plane specific tensile strength with apparent sheet density



Fig 14 Influence of refining and fines removal on the variation of zerospan strength with apparent sheet density

Fiber strength or the ultimate strength of the sheet  $(\underline{9})$ , as inferred from zero span measurements, does increase with sheet densification independently of how it is produced as shown in Figure 14. Surprisingly, this relationship is unaffected by fines removal.

#### **Results and Discussion of Experiment 2**

The variation of drainage time with fines addition is shown in Figure 15. The drainage time at 0% fines addition is the drainage time of the 600 ml CSF fines-free pulp. It is inferred that the greater the drainage time for a specific source of fines and addition level, the larger their hydrodynamic surface area.

Above a fines addition level of about 20%, differences in the hydrodynamic surface area of the various type of fines are very evident. As expected, there is a clear difference between the primary and secondary fines.

With respect to the control fines (secondary fines plus a small fraction of primary fines), the "secondary" or recycled fines behave very similarly. Unexpectedly, the "secondary"(H) fines have an even greater hydrodynamic specific surface area. No explanation for this effect can yet be offered.

As we saw in the first set of experiments, fines have an impact on sheet densification. The variation in sheet densification with fines addition is shown in Figure 16. Again, there is a clear difference in performance level between the primary and secondary fines. The differences between the control and "secondary fines" are not as well defined, and this may be due to a greater variability in fines loss.

The properties we examined in the first set of experiments are again shown as a function of sheet densification. It was just demonstrated that sheet consolidation is controlled by the type of fines and amount added. Therefore, it may be anticipated that the structure of the sheet, at a given level of densification, will depend on how it is achieved, i.e., refining, wet pressing, or fines addition.

Figures 17, 18, and 19 illustrate how structural properties, such as formation, roughness, and porosity, vary with densification by fines addition. There is considerable scatter in the formation results as shown in Figure 17. This might be attributed to the fact, that at high levels of fines addition, increases in drainage offset gains in fiber length reduction.



Fig 15 The effect of fines type and content % on sheet mold drainage time

Interestingly, as the level of fines is increased, sheet roughness also increases and is to a large extent independent of fines type. We note that newsprint fines are not very effective in densifying the sheet; nevertheless, following a slight increase, roughness decreases with further fines addition. When the influence of primary and secondary fines on roughness is compared, the trends are approximately the same, although the secondary fines as shown in Figure 18 are more effective in densifying the sheet and, hence, producing a greater level of roughness. By comparison, the newsprint fines are inert. If the result shown in Figure 18 is compared with Figure 9, we see, at a given level of densification, that fines produce a higher level of surface roughness than refining and wet pressing.

Porosity measurements are sensitive to changes in sheet structure as demonstrated by comparing Figure 19 with Figure 10. It appears that the newsprint fines are much more effective at reducing air porosity than the chemical pulp fines. This seems to imply that the newsprint fines are located more in the interfiber void volume, while a significant proportion of the chemical fines are located at interfiber bonds. These differences might also be attributed to how the fines are distributed in the thickness direction.



Fig 16 The influence of fines type on the variation of sheet densification with level of % fines addition



Fig 17 The influence of fines type on the variation of formation index %CV(W) (based on mass density measurements) with apparent sheet density



Fig 18 The influence of fines type on the variation of sheet roughness with apparent sheet density



Fig 19 The influence of fines type on the variation of sheet porosity with apparent sheet density



Fig 20 The influence of fines type on the variation of in-plane specific elastic modulus with apparent sheet density



Fig 21 The influence of fines type on the variation of out-of-plane specific elastic modulus with apparent sheet density



Fig 22 The influence of fines type on the variation of in-plane specific tensile strength with apparent sheet density



Fig 23 The influence of fines type on the variation of zero-span tensile strength with apparent sheet density

The in-plane and out-of-plane elastic properties are shown in Figures 20 and 21. It is interesting to compare the in-plane and out-of-plane performance of the "secondary"(H) fines and the newsprint fines as two extremes. The addition of "secondary"(H) fines results in a higher level of in-plane elastic properties when compared with the control fines, while they are less effective in improving the out-of-plane modulus. By contrast the, newsprint fines have a greater impact on out-of-plane modulus while having a negligible effect on in-plane modulus.

The trends in tensile strength development, Figure 22, closely follow those shown for the in-plane elastic constant. One obvious exception is that newsprint fines appear to contribute to tensile strength but not to the in-plane elastic properties. In Figure 14, we saw an increase in zerospan strength as sheet density is increased by refining and wet pressing; however, an increase in sheet density by fines addition results in a slight downward trend in strength as shown in Figure 23. Although there is some scatter, it does appear that the primary-type fines, including the newsprint, result in an even greater loss in zerospan tensile strength.

#### CONCLUSIONS

The impact of fines on selected physical and mechanical properties of paper has been examined.

In the first of two experiments, the influence of fines was determined by producing two fines-free pulps from furnishes which had been refined to 600 ml and 290 ml CSF. Fines removal had a detrimental effect on most properties at a given level of densification including: formation, in-plane and out-of-plane elastic properties, and normal span tensile strength. Densification either by refining, wet pressing, or fines addition resulted in an increase in sheet roughness; this is tentatively attributed to an increase in nonuniform shrinkage in the thickness direction of the sheet. Fines removal gave a more porous sheet particularly at the higher level of refining. Zero span strength or the ultimate strength of the sheet increased with sheet densification, being largely independent of how that densification was produced.

Fines type and addition level were investigated in the second set of experiments. Fines, up to a level of 30%, were added to a fines-free furnish 740 ml CSF. It was inferred from drainage measurements that the secondary fines had a greater hydrodynamic surface area and were, therefore, more effective than primary fines in enhancing sheet densification and properties. Furthermore, "secondary"(H) fines, which had been

produced from handsheets which had undergone more extensive wet pressing and drying, were, surprisingly, even more effective than the control fines and "secondary" fines. The behavior of newprint fines from preconsumer waste was similar to that produced by primary fines.

It is clear that fines, defined as material passing a 200 mesh screen, are inadequate to characterize their impact on paper properties. This is essentially in agreement with the findings of Hawes and Doshi (<u>16</u>).

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## **Transcription of Discussion**

## THE EFFECT OF RECYCLED FINES ON PAPER PROPERTIES

J F Waterhouse

## A Chatterjee, University of Toronto, Canada

It wasn't quite clear whether in the first set of experiments the pulp was never dried or dried market pulp?

## **J** Waterhouse

In the first set of experiments the pulp is a never dried, unbleached kraft pulp - southern pine.

## A Chatterjee

Thank you and another question. Did you measure the turbidity of the white water to find out the specific scattering coefficient of the fines? That might be a way to look at the quality.

## J Waterhouse

We are certainly aware of the work that has been done on turbidity and we may use that approach. No we did not. We have been doing some work on critical point drying of fines as a means of characterisation.

## A Chatterjee

Thank you and the last question is, when you added 10%, 20% and 30% fines do you have any idea how much of it is translated into the actual fines content in the handsheet?

## J Waterhouse

A good question. Of course as you increase the fines content and don't do anything about it you get significant losses and so what K Omori and I decided to do was to adjust the fines so that the numbers, we hope, are fairly close to what we are indicating.

# Prof E Back, Feedback Consulting E & E Back KB, Sweden (A comment on A M Scallan's paper)

Let me summarise the existing proof for understanding the swelling restrictions introduced in drying and subsequent aging as autocrosslinking reactions in the cellulosic or ligno-cellulosic paper material. I have measured these swelling restrictions mainly as produced wet strength, wet modulus and dimensional stability.

If, before drying, a kraft pulp is oxidised with periodate to introduce some dialdehyde groups in the cellulose/hemicellulose structure, considerable wet strength is produced even during room temperature drying. Also, dialdehyde starch is a wet strength agent for paper. If instead, existing carbonyl groups in the pulp are reduced by sodium borohydride, the swelling restrictions develop much more slowly than with the untreated pulp. The reaction rate of wet strength production can also be increased by metal ions with a high redox potential in the pulp suspension, and by the existence of acidic conditions in the dry paper(1).

Over the temperature range from 70-350°C the reaction rate to produce wet strength on aging has an activation energy of 25,000 to 30,000 calories per mole, expected for covalent bond formation. The mechanism is considered to be initiated by radical formation followed by both crosslinking through ether and hemlacetatal bonds and chain scissions. Also for this chain scission an activation

energy around 25,000 calories per mole was found.

Other types of energy transfer to a dry or moist paper such as gamma-radiation, UV radiation and corona-treatment produces these swelling restrictions, as shown repeatedly in the literature. Also chemical oxidation of a paper material such as with nitric acid produces such swelling restrictions on drying at room temperature(2).

It thus appears that the proof for autocrosslinking is quite sufficient. See also an overall review(3). The aging of other organic polymer material is also explained by auto-oxidative crosslinking. It appears to me that cooperative hydrogene bonding cannot explain all these interconnected experimental data.

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## J Waterhouse

I'll comment. The only relevance I see of what you've said to my presentation concerns recyclability. Maybe we did produce some cross linking in the extended heat treatment that we performed. We didn't think of it in those terms but just wanted to see the impact on fines. To our surprise once we had generated the "secondary" fines you can see from our results that their performance was certainly equal or better than the virgin fines. It's interesting that although we are recycling, the new material that we generate is equivalent to the virgin state.

## A Scallan

I deliberately avoided mentioning auto-crosslinking which, of course Ernst, you have suggested as a mechanism in the literature several times. I don't see how crosslinking, which very possibly occurs during heat treatments, can happen at room temperature. In addition, we cannot detect the groups you are talking about in bleached pulp which only has a few acid groups as reactive groups.

## P E Wrist, PAPRICAN, Canada

It is becoming evident that many chemical and other contaminants are highly concentrated in the fines fragment of recycled pulp. Perhaps we should deliberately sacrifice the original fines component of recycled pulp as an integral part of the recycling and deinking process and recreate all the fines we need for the subsequent papermaking by refining. In an holistic approach to the environmental problems caused by paper products and their post consumer disposition, such a strategy may well prove to be a part of the optimum solution. If we were to pursue such an approach it would suggest we should give greater attention to the use of screening, and perhaps to screening research. Do you have any comments on such a strategy?

## J Waterhouse

I think that's a good point. We tried to keep an open mind because we have not tackled the problem of contaminants. We intend to look at that but part of the motivation here is to deal with the so called "primary" fines that we are generating, Mancebo and Krkoska and others have said that this hornification is irreversible. I'm not sure that I believe that or that Tony (Scallan) would. Part of our strategy might be that in some situations we can rejuvenate the fines fraction and solve the problem of water removal.