INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

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# UTILIZATION OF RECYCLED FIBERS

## Improved Utilization of Recycled Fines

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Report 1

A Progress Report

to the

MEMBER COMPANIES OF THE INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

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## TABLE OF CONTENTS

## SUMMARY

This progress report is concerned with increasing the utilization of recycled fiber and more specifically with improving the utilization of recycled fines. Currently, recycled fines are considered to have an adverse effect on both paper machine and product performance. Consequently, fines may be burnt or sent to landfill solutions which are becoming less viable due to environmental concerns and the steeply rising cost of landfilling.

It is presumed that with suitable screening techniques a recycled furnish can be fractionated to produce long fiber and fines fractions for which appropriate treatments to improve their papermaking performance can be sought.

This progress report contains a review of the literature concerning recycling, the role of fines in papermaking, and how paper properties are affected by fines. The loss in fines performance due to recycling and ways to recover it are also considered.

Fractionation trials of OCC have been conducted using both commercial and laboratory scale equipment, and exploratory studies to reverse the effects of drying have included sodium hydroxide, ozone, and hydrogen peroxide. Sodium hydroxide treatment in the range of 0% to 10% has so far shown the most promise.

## INTRODUCTION

Recycling is making an important contribution to protecting our environment and improving raw material utilization. Recycling has been considered since the advent of modern papermaking, however; the driving forces to significantly increase our utilization of recycled or secondary fiber have resulted in the need for us to address a number of technical issues.

Technical areas of concern identified by technical managers of IPST member companies for IPST are, in order of priority, listed below.

* FIBER AND PRODUCT STRENGTH
* WET END CHEMISTRY
* DEINKING
* PRINTABILITY
* REFINING
* COATABILITY
* BLEACHING
* FIBER FRACTIONATION
* RECYCLED FIBER CONTENT ANALYSIS
* TECHNICAL EDUCATION OF THE PUBLIC
* ENVIRONMENTAL ISSUES

Some of these concerns are being addressed by IPST's recycling team of which this author is a member. Prior to the formation of the recycling team and identification of the above areas of concern, the issue of fines and recycling was proposed by this author as an important area for investigation. Fines are important to a number of the above areas including product strength, fiber fractionation, refining, printability, and coatability.

Although the emphasis will be on fines as related to recycling, it is recognized that fines cannot be studied in isolation, and must be related to other furnish and papermaking performance areas.

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% of a never dried furnish, and as much as 30% or more of a never dried well-beaten furnish. It is generally agreed that fines have an adverse effect on water removal, while their impact on properties, at least in some areas, remains controversial.

Can the generation and treatment of fines be improved to obtain a better papermaking and end-use performance of the paper containing them, is an important question to be answered. The argument that it might not be practical or cost-effective to separate out fines and provide for their special treatment is premature. Energy savings and the improvement of product properties is possibly sufficient incentive, if the separate treatment of the long fiber and fines fraction is both viable and beneficial. There is also the need to increase recycled fiber utilization and minimize the amount of that material going to landfill. The fines content of recycled fiber is presently one of the prime candidates for landfill, and therefore, treatments to upgrade this material would help to reduce this burden.

Solid waste reduction is being driven by strong economic and environmental factors. The hierarchy of solid waste strategies consists of:

a) reduction

b) recycle and alternative uses

c) burn

d) landfill

Solutions c) and d) are no longer acceptable, although it can be argued, in some cases, that the burning of solid waste is an economically viable energy source if environmental concerns can be reconciled.

Currently, the disposal of solid waste is as follows (1):

* Landfill 70%
* Burning 21%
* Land use 8%
* Alternative uses 1%

Very few mills, if any, are totally "closed," although there has been considerable progress toward achieving this goal, particularly to reduce fiber solid waste, since it has been realized that a valuable resource is being wasted. The need to increase recycled fiber usage and reduce solid waste levels places an even greater emphasis on the need to implement mill closure and better strategies for improving the utilization of recovered fiber.

The literature review which follows is comprised of the following subsections: General literature on Recycling and Fines, Fines Characterization, Impact of Fines on the Papermaking Process, Impact of Fines on Paper Properties, Recycled Fines, and the Chemical and Mechanical Processing of Recycled Fines.

# LITERATURE REVIEW

The role of fines in papermaking has always been, to some extent, controversial, and even less is known about the impact of recycling. It is generally accepted that fines have an adverse effect on the drainage resistance of a pulp, but can have a positive benefit on some paper properties, e.g., the mechanical properties of paper. How these papermaking variables are affected by different recycling strategies has yet to be determined. Even with virgin pulps, good control of pulping and bleaching is essential in optimizing properties, for example, strength and drainage performance. Similar strategies will also be needed with recycled furnishes.

## GENERAL LITERATURE ON RECYCLING AND FINES

A considerable effort is now being expended to increase the utilization of recycled or secondary fibers in a variety of grades of paper and board. Recycled fiber is, without some form of mechanical and/or chemical treatment, inferior in performance to virgin fiber. One manifestation of this is the difference in performance of never dried and market pulps, although one does not regard the latter as recycled fiber. A market pulp has experienced little or no refining, but when subjected to refining can develop properties comparable with a never-dried beaten pulp. In addition to the common refining effects, i.e., internal and external fibrillation, fines generation, etc., an important refining action for a market pulp is the removal of kinks and curl, which are responsible for the initial increase in sheet strength according to Page (2). The kinks and curl arise from high consistency processing, and unrestrained drying on the paper machine or in flash drying processes.

The major difference between a market pulp and recycled fiber is that prior to recycling the fibers have generally been subjected to a significant level of refining. The more refining the fiber has received the greater the effects of drying or hornification (3). Furthermore, in addition to drying or hornification, the furnish may be contaminated by stickies, surfactants, starch, wet strength agents, printing inks, and adhesives. It is presumed that ineffective strategies for removal of these contaminants can lead to a further degradation of the pulp's papermaking potential. There is little published data in this area, and the focus of attention has been mainly on deinking of various grades of paper and the achievement of satisfactory optical properties.

A recycled furnish also differs from a virgin or market pulp furnish in as much as there may be a high level of fines present. In many operations using recycled fiber, the fines which are considered to be only an inert filler material are removed to become a component of sludge or landfill. Fines utilization and management (4) have become important issues due to increasing demands for recycled fiber usage and mill closure.

There is extensive literature on the subject of recycling. This literature review is not intended to be all-inclusive, but where relevant some general sources are referenced. The Institute of

Paper Science and Technology has produced two useful Bibliographic Series on "Reclaimed Fibers," the last of which was issued in 1983 (5). More recently, TAPPI Press 1990 has published two volumes of collected papers on the subject of "Recycling Paper" (6), and part of Volume 3, in the TAPPI/CPPA series devoted to Pulp and Paper Manufacture, deals with secondary fiber usage (7). A recent publication by TAPPI "Secondary Fiber Recycling" (8) also deals with various recycling topics.

Some of the technical issues of recycling have been reviewed by de Ruvo and Htun (9) and more recently by Howard (10). These issues include changes in properties due to repeated cycling, factors affecting the recycle potential of a pulp, papermaking variables, i.e., refining, wet pressing, drying, calendering, and chemical additives. The reviews also considered strategies for recovery of the pulp's papermaking potential.

A number of researchers McKee (11), Bovin, Hartler, and Teder (12), Bobalek and Charturvedi (13), and Howard and Bichard (14) have examined property changes with repeated cycling. The general findings are well-known, and one can conclude that there is a general loss in strength properties with the exception of tear which usually increases.

It is important to know what the likely level of recycling is within a furnish, and this will obviously depend on the level of recycled fiber utilization one wants to achieve. This question has been addressed by Howarth and Rogers (15) and by Cardwell and Alexander (16) whose equation is given below.

$$EPn = (100 - P)(P/100)^n \qquad (1)$$

where EPn is the equilibrium percentage of fiber recycled n times, and P is the target percentage of fiber to be utilized. Using equation (1) the percentage of the total furnish which has been recycled 1, 2, 3, 4, and 5 times for different utilization rates is given in Table 1.

TABLE 1 PERCENTAGES OF FURNISH WHICH HAS BEEN RECYCLED FOR 1, 2, 3, 4, AND 5 TIMES FOR DIFFERENT UTILIZATION RATES.

| **Number of Recycles** | **% RECYCLED FIBER UTILIZATION** | | | | | |
|---|---|---|---|---|---|---|
| | **20** | **40** | **50** | **60** | **70** | **80** |
| **0 (virgin)** | 80 | 60 | 50 | 40 | 30 | 20 |
| **1** | 16.0 | 24.0 | 25.0 | 24.0 | 21.0 | 16.0 |
| **2** | 3.2 | 9.6 | 12.5 | 14.4 | 14.7 | 12.8 |
| **3** | 0.64 | 3.84 | 6.25 | 8.64 | 10.3 | 10.2 |
| **4** | 0.13 | 1.54 | 3.13 | 5.18 | 7.20 | 8.19 |
| **5** | 0.03 | 0.61 | 1.56 | 3.11 | 5.04 | 6.55 |
| **TOTAL %** | 99.87 | 99.59 | 98.44 | 95.33 | 88.23 | 73.74 |

Achieving a target of 50% recycled fiber utilization by the year 2000 (17) means that approximately 25% of the total furnish will have been recycled on average more than once.

Papermaking is generally a balance between property development and runnability. Cardwell and Alexander (16) have expressed this for recycling by how the number of recycles affects the strength - freeness envelope which is shown in Figure 1. For a target strength level the bad news is that a reduction in freeness seems to be inevitable when compared with the virgin fiber. Ways to circumvent this might include refining a fines free recycled furnish, fractionation and separate treatment, e.g., refining, of the long fiber and fines fraction, additives, chemical treatment, or increasing basis weight.

Machine speed sensitivity and hence productivity to drainage has been emphasized by de Ruvo and Htun (9). Their relationship for a furnish containing 25% recycled furnish is shown in Figure 2.

As we will discuss in more detail later on, the loss of strength due to recycling is mainly attributed, in a simple system, to drying (hornification) which results in a loss in swelling of both the long fiber and fines fraction. de Ruvo and Htun (9) have shown, for a given furnish and within a limited range of fines addition, that the linear relationship between tensile strength and swelling as measured by water retention value (WRV), see Figure 3, is independent of composition. Thus, a loss in swelling produces a concomitant loss in strength. The greatest loss in swelling, according to their results and those of other researchers, is associated with the fines fraction.

There are a number of reviews focusing on virgin and recycled fines (18), (19), (20), and (21), aspects of which we shall briefly consider. We will next review the characteristics of fines with the main emphasis on chemical pulps; however, reference will be made to mechanical pulp fines as appropriate.

## FINES CHARACTERIZATION

Fines have been defined as the short fiber fraction, fiber debris, crill, slime, and flour. The latter two are usually associated with mechanical pulps. 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 established by Tasman (22) is shown in Figure 4. Tasman recommends that a weighted average fiber length of 0.2 mm be used for the fraction passing a 200 mesh screen.

Pelton, Jordan, and Allen (23) have used image analysis techniques to look at size distributions in mechanical pulp fines. Their main determination was ALLD (average longest length dimension). They rejected fines having a length greater than 200 μm and an area greater than 25 $\mu m^2$. Great care was required in sample preparation and dyeing to avoid large errors. Typically, 25 slides/pulp were prepared to measure a total of around 5000 fines. An optical microscope was used in conjunction with a video camera and image analyzer to determine the distribution of ALLD. Averages were in the range of 16-25 μm with standard deviations of the same order.

Gavelin, Kolmodin, and Treiber (24) have used a critical point drying (cpd) technique to exam the fines from TMP and stone gwd pulps. They found the crill to consist of filaments, ribbons and threads, lamellae, coarser fragments, and material lacking a fibrillar structure, i.e., pitch, lignin, and contaminant materials. The filaments had a mean width of 0.3 μm for the TMP and 0.6 μm for the GWD. The fines when air dried from water appeared to form an amorphous mass; however, in cpd of the same fines, the fibrillar structure was preserved. The authors state that when air-dried fines from water are rewet they can easily regain their fibrillar structure. This implies that in air drying, surface tension forces operate and bring the filaments into close contact followed by hydrogen bonding. However, since the fibrillar structure is recovered upon rewetting, there does not appear to be irreversible hornification of these fines. This is consistent with Scallan and Tigerstrom's (25) results that hornification does not occur above a pulp yield of 72% as discussed in more detail below.

Htun and de Ruvo (26) 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 5. 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 it is seen that this increment is dependent on refining level. Whether this is due to an increase in the amount of the <200 mesh fraction and or a change in the "bonding potential" of the fines is not known. The 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. Little work has been devoted to assessing the contribution of the colloidal fraction. This fraction may be rich in hemicelluloses and contribute to sheet strength (26), as we will discuss shortly.

In Htun and de Ruvo's work (26), 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 second 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, as shown in Figure 6, the crystallinity was lower. Figure 7, which shows the sorption isotherms for the whole pulp and the fines fraction, illustrates that there is a difference in cellulose-water interaction.

According to Mancebo and Krokoska (19), 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 extractives content than secondary fines or the whole pulp. It is generally found that primary fines contribute little to bonding (20).

The level of primary fines present in a pulp is species dependent. Mancebo and Krokoska (19) found for three softwood pulps that fines content was in the range of 0.7% to 6.2%, for a mixed hardwood 7.3%, and for a partially depithed bagasse 12.7%.

The rate and level of secondary fines generation during refining will be dependent on species and pulp type as illustrated in Figure 8. We see that fines generation for the bleached NSSC and sulfite pulps is much greater than the unbleached kraft pulp. Page's (27) 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 (28) 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 support this supposition.

## IMPACT OF FINES ON THE PAPERMAKING PROCESS

The major difference between laboratory refining and production refining is, according to Danforth (29), the uniformity of stock treatment, i.e., homogeneous versus hetrogeneous.

Homogeneous refining produces strength development without excessive fines production and fiber cutting. In terms of the specific edge load concept (30), this means a low specific edge load. How the rate of fines generation and their behavior varies with pulp type, and the kind and level of refining has yet to be determined.

There are many fines-related challenges at the wet end of the paper machine. These include fines management and retention (4), which was mentioned earlier, and the conflicting demands of drainage and formation. Fines in moderation appear to be a necessity. Although they can retard drainage, their role in sheet consolidation through Campbell's forces is a positive effect. The majority of workers have found that fines, as we shall see shortly, increase elastic and strength properties, and usually a reduction in tear at least for chemical pulps. Stone, Scallan, and Abrahamson (31) have shown, using the solute exclusion technique, that the water associated with fines <100 mesh is much greater than the long fiber fractions. Furthermore, they also found that bleached kraft fines have more water associated with them than the bleached sulfite fines. A number of other researchers, for example, Rhys (32), Giertz (33), Szwarcsztajn and Przybysz (34), confirm the finding that fines have more water associated with them.

It is interesting to note that when fines are removed and then added back to the long fiber fraction the drainage differs from the whole pulp having the same level of fines (Rhys (32)). Giertz (33) states that if there is bound water sharing between the fibrillated fibers and the fines, then the sum of the bound waters of these components should be greater than the whole pulp. That this is so is shown in Table 2 where the values are determined using the solute exclusion technique of Stone and Scallan (35).

TABLE 2 INACCESSIBLE WATER g/g FOR VARIOUS PULP FRACTIONS - Data of Giertz (33).

| Pulp Type | Crill Content % | Whole Pulp | Decrilled Fibers | Calc. for Crill in Whole Pulp | Isolated Crill |
|---|---|---|---|---|---|
| Spruce Sulfite | 2.9 | 1.24 | 1.15 | 4.0 | 7.51 |
| | 6.1 | 1.87 | 1.67 | 4.9 | 9.69 |
| Birch Sulfate | 5.5 | 1.40 | 1.35 | 2.2 | 5.9 |
| | 10.1 | 2.17 | 1.78 | 5.6 | 9.2 |

Fines also play a dominant role in sizing and the addition of other wet end additives because of their large surface area. Sizing can also negatively impact the recycling potential of fines Marton (36). Whether the charge behavior of the fines is representative of the long fiber fraction has long been a contentious issue (37).

As with dry strength, fines can have a positive effect on wet web strength (38). The effect of fines on wet strength, however, does not appear to have been extensively investigated, although the work of Stratton (39) is an important contribution.

Because of the large amount of water associated with fines, the ease with which water is removed will be dependent on their characteristics. The washing out or migration of fines during wet pressing is also in dispute (40). Carlsson and Lindstrom have also shown that hornification can begin with wet pressing (41).

Generally, the higher the WRV the more energy will be required to dry the paper. This author is not aware of any detailed study to determine how fines and their characteristics affect drying performance. With regard to high intensity drying, for example, impulse drying, its effectiveness may be limited at high levels of hydrodynamic surface area, i.e., too high a level can lead to sheet delamination problems as shown by Orloff and Lindsay (42).

Therefore, alternate strategies to produce sheets with a high fines content may be necessary with impulse drying methods. The strength properties of a sheet containing 100% fines are certainly impressive (43).

Drying and internal stresses will also be strongly affected by the amount and type of fines present.

We have seen that the influence of fines on paper machine performance is pervasive, and this situation is not expected to be any less challenging with increased recycled fiber utilization.

## IMPACT OF FINES ON PAPER PROPERTIES

The main factors controlling the tensile strength of paper are shown in Figure 9. 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 wet pressing and refining can increase strength by increasing R.B.A. or apparent density. Refining can lead to an increase in fiber modulus and strength, and a reduction in stress concentration and or improved stress transfer.

It has been shown by Stratton and Colson (45) 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 and/or improve stress transfer as depicted in Figure 9. Stress concentration will be governed by the size, shape, and bonding potential of the fines, all of which may be changed during recycling.

One of the major consequences of recycling is 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 include contamination by inks, sizing, surfactants and other materials, as well as "damage" to the long fiber fraction.

To better understand the role of fines, particularly with respect to recycling, we will, for the time being, focus our attention on the fines fraction of a recycled pulp, its separate treatment, and subsequent impact on papermaking and properties.

Kibblewhite (46) 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 (47) 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 were 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, Murakmi, and Katsura (48) used a micro-fibrillated cellulose and a crystalline cellulose (Avicel) to study the impact of fines on the structure and properties of paper. The microfibrillated cellulose 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 μm and length 40-70 μm. It was found that MFC increased sheet density and tensile strength, while Avicel had the opposite affect. It would be interesting to know whether this result was simply a particle size effect or differences in structure, i.e., relative crystallinity.

Nanko and Ohsawa (49) 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. This model is similar to the concepts proposed by Giertz (33).

There is general agreement that recycling causes a decrease in strength properties of paper, and it has been shown by a number of researchers that this decrease is related to a reduced bonding ability of the fibers termed "hornification." Hornification results in a loss of swelling and surface area, and a reduction in fiber flexibility. McKee (11) observed that the water retention value (WRV) decreases with recycling. Lundberg and de Ruvo (50) found a direct relationship between WRV and tensile strength.

According to Mancebo and Krokoska (19), the fines fraction of a 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 it is claimed that the effect is irreversible even with refining! No chemical treatments were reported in their work.

Swarcsztajn and Przybysz (51) also found that fines and fibers become hornified with recycling, and that strength properties decrease.

On the other hand, Hawes and Doshi (21) 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 refined fines-free recycled unbleached kraft bag paper.

The results of Hawes and Doshi (21) are shown in Figure 10. 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 10, is not immediately obvious. This finding was noted, but not commented upon by the above authors.

## RECYCLED FINES

Mancebo and Krokoska (19) 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, they considered the fines fraction of recycled pulps as a filler material to be 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 during the refining of recycled pulps.

In recent work, Scallan and Tigerstrom (25) have demonstrated, using predictions of transverse fiber modulus, that hornification of the long fiber fraction can be reversed by refining. Furthermore, they found no evidence of hornification in pulps above a yield of about 70%. Therefore, fines from mechanical pulps should not hornify. As stated earlier, this was the finding of Gavelin, Kolmodin, and Treiber (24).

The contribution of primary, secondary, and a mixture of "primary" and secondary fines to paper strength has been demonstrated by Mancebo and Krokoska (19) and is shown in Figure 11. We note that, at a given level of fines addition, secondary fines improve strength more than primary fines, while the mixture of primary and secondary fines falls in between these extremes.

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, a conclusion also reached by Mancebo and Krokoska (19). 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 if that is established.

## CHEMICAL AND MECHANICAL PROCESSING OF RECYCLED FINES

Howard (10) has suggested four ways to recover the lost property potential of recycled pulps namely:

a. Beating or refining

b. Chemical treatment

c. Blending with virgin pulp

d. Fractionation

As we have already hypothesized, there might be advantages in a separate treatment of the long fiber and fines fraction of the pulp.

By fractionation, virgin and recycled pulps can be divided into long fiber and fines fractions. We use the generally accepted definition of fines as the pulp fraction which passes through a 200 mesh screen.

Fines material can be further categorized depending on pulp type, i.e., mechanical, chemical, virgin, or recycled. The properties of fines will also depend on where they originate as discussed earlier. We will use the definitions given in Table 3 in our discussion of fines. The terms primary, "primary," secondary, and "secondary," although useful, are not sufficiently precise.

TABLE 3 FINES NOMENCLATURE.

| CYCLE | PRIMARY | "PRIMARY" | "SECONDARY" | SECONDARY |
|---|---|---|---|---|
| | | "Unrefined" | | Refined |
| VIRGIN FINES $F_0$ | $P_0$ | - | - | $S_0$ |
| ONCE DRIED $F_1$ | - | $P_{0d1}$ | $S_{0d1}$ | $S_1$ |
| TWICE DRIED $F_2$ | - | $P_{0d2}$ | $S_{0d2} + S_{1d1}$ | $S_2$ |
| THRICE DRIED $F_3$ | - | $P_{0d3}$ | $S_{0d3} + S_{1d2} + S_{2d1}$ | $S_3$ |

The above table demonstrates that keeping track of fines can be a complex undertaking. As an example, the fines present in twice dried refined pulp $F_2$ consist of:

$$F_2 = P_{0d2} + S_{0d2} + S_{1d1} + S_2 \tag{2}$$

The primary fines $P_0$ present in a virgin pulp prior to refining consist mainly of ray and parenchyma cells, and represent around one to eight percent of the furnish. They are regarded as filler material and do not contribute significantly to paper properties (19). On the other hand, secondary fines $S_0$ do contribute positively to many paper properties, although they do have an adverse effect on drainage and water removal (19), (20).

The fines present in a once dried pulp consist of a portion of dried primary fines $P_{0d1}$, and dried secondary fines $S_{0d1}$. These fines, which are the main subject of our proposal, tend to perform like primary P fines, i.e., as a filler material. On the other hand, the secondary fines $S_1$, produced by refining a once dried pulp, tend to equal or better the performance of virgin secondary fines $S_0$ (20), (21).

We assume that fractionation of a recycled pulp will enable a more appropriate treatment for the long fiber and fines fractions. Treatment of the long fiber fraction will probably involve some level of refining and possibly chemical treatment to reverse the effects of drying and remove defects, while minimizing fiber damage and fines production. This strategy has been suggested by Musselmann (52) as a way to reduced energy consumption.

In many instances, recycling does involve fractionation, but the fines fraction ($P_{0d1}$ + $S_{0d1}$) of the pulp is disposed off, and a portion of the long fiber fraction is also lost. When deinking is involved, in addition to fines, filler, ink particles, and other contaminants are disposed off as solid waste. **However, our main goal is to recover the papermaking potential of the fines fraction $S_{0d1}$ in order to make fractionation a more efficient process and reduce**

**material currently going to landfill.**

One of the main factors contributing to the loss in papermaking potential of a recycled pulp is drying. The effect is known as hornification and results in a loss of swelling of both the long fiber and fines fractions (51) after they have been dried and rewet. Scallan and Laivins (53) have recently shown, using infrared analysis and deuterium exchange, that hornification of a fines-free pulp is the result of irreversible hydrogen bond cross links formed between microfibrils during drying, i.e., these bonds are not broken upon rewetting.

The mechanism proposed by Scallan and Laivins (53) for hornification of the long fiber fraction might also be presumed to hold for the fines fraction when the pulp yield is less than 70%. As already noted, Mancebo and Krokoska (19) found that refining does not reverse fines hornification even though refining does reverse fiber hornification. No reasons were given for why this was so, yet, this difference is crucial for understanding the loss in fines performance. Their finding might be an indication that other factors, in addition to hornification, e.g., agglomeration, changes in wetting behavior, and ineffective communication of mechanical stresses to the fines fraction, might be involved.

An important part of our research program is to determine why the papermaking performance of fines is adversely affected by drying, and why refining does not restore it. This knowledge should enable us to design specific chemimechanical treatments to either prevent or reverse the adverse effects that drying has on fines.

It is anticipated that mechanical action to impart stresses to the fines will be a necessary part of the treatment, for example, to reduce agglomeration and reswell the fines. Solute exclusion (31), water retention values (51), and drainage resistance measurements (21) have shown that there is a much higher association of water with the fines than the long fiber fraction. Owing to the smaller dimensions of the fines and surface tension effects, caution has to be exercised when interpreting the extent of fines-water interaction.

It is interesting to note that fines $S_1$, produced by refining a once dried pulp, are comparable in performance to virgin secondary fines $S_0$ (20). This implies that drying does not alter the cellulose-water interaction of new surfaces created by refining. However, if changes in crystallinity due to drying occur, as found by Marton et al. (54), this finding may be modified.

There are no theories which satisfactorily account for the contribution of fines in a network. In broad terms, we can say that fines contribute positively to the mechanical properties of paper by reducing stress concentration and effectively increasing interfiber bonding (44). There are, according to Page (55), many effects associated with the refining of a pulp, and fines production is one of them. However, we are now considering the treatment of fines themselves which have been dried. As with refining, we will assume that internal and external fibrillation of the fines will be necessary to reswell them.

Possible approaches for mechanically treating the fines fraction include: homogenizing equipment (56), and abrasive minidisk refining (57). Conditions favorable to reswelling and possible chemical treatments are next reviewed.

The effects of cations, pH, and electrolyte concentration on refining and paper properties have recently been investigated by Scallan and Grignon (58) and Lindstrom and Kolman (59).

Scallan and Grignon have proposed that swelling produced by refining in the presence of electrolytes is due to both mechanical and osmotic stresses. Using fiber saturation point (solute exclusion) as a measure of fiber swelling, monovalent sodium was shown to be the most effective cation in their studies. Furthermore, when kraft and sulfite pulps were acid washed to remove the metal ions present in the pulp and replaced with sodium ions, the osmotic refining effect was found to be more effective than refining in a PFI mill, i.e., the fibers were internally fibrillated without fines production.

Whether cationic exchange procedures would be effective in reswelling the long fiber and fines fractions of a recycled pulp remains to be determined. In any case, beating under alkaline conditions in the presence of sodium ions should enhance refining. Lindstrom and Kolman (59) investigated the effects of pH and electrolyte concentration on swelling (water retention value) and paper properties. They were careful to separate out the effects of chemical environment differences during beating and sheetmaking. They found for an unbleached kraft pulp pH=10 is optimum for maximizing swelling, and that the addition of 0.1M NaCl results in a lower wrv at the same beating level of 4000 revs. No similar findings were found for a bleached kraft pulp.

Centola and Borruso (60) in earlier work demonstrated that Congo red has a very significant effect on refining rate and strength development. The mechanism is that suggested by Scallan and Grignon (58), and the osmotic stress is created by the counterions of the sulfonic acid groups. According to Page (55), other additives which could produce this effect include:

| |
|---|
| Sodium carboxy-methyl cellulose |
| Oxidized Starch |
| Lignosulfonates and modified lignins |
| Napthalenē, benzenē, and stilbene-based dyes |
| Hydrolysed polyacrylonitrile |

Page (55) also cautions that fiber-fiber and metal-fiber friction may also be modified by additives. Again, we do not know if such additives would be effective in treating recycled fibers.

Sodium hydroxide is frequently used to treat recycled fiber furnishes, particularly where deinking is involved; however, little information of its effect on refining and strength development has been reported.

An important exception is the study by Freeland and Hrufiord (61), where an OCC furnish (whole) was treated with 2% NaOH for 4 hours. Their data show that there is a rapid rise in compressive strength (which is used as a measure of the effectiveness of the NaOH treatment) over the first hour and then a more gradual rise over the next 15 hours. The authors suggest that the strength improvement is due to straightening of the fibers with beating. Surprisingly, they found that freeness increases at the same time as sheet density and strength increase.

A more relaxed (no definition given) fiber was suggested as the reason for the higher freeness since the fibers would be less susceptible to damage in this state. We expect that the freeness would be lower with straighter fibers; however, the osmotic stress contribution as proposed by Scallan and Grignon (58) may have resulted in less fines production.

Bovin, Hartler, and Teder (12) found that tensile strength was increased when a kraft pulp was disintegrated in an alkaline solution, but no explanation of this finding was given.

Most of the work which has been done with high levels of caustic treatment of cellulose has been done with cotton. When one examines the changes in cotton fiber properties with caustic treatment level, the large changes occur close to and beyond 10% NaOH (62). In our situation, it is presumed that higher levels of caustic (0% to 10%) will improve swelling, but the effects of mechanical action also need to be determined. Large losses in hemicellulose and yield are not acceptable, and treatments have to be examined in this light. Freeland and Hrufiord (61) found that the biological oxygen demand increased significantly at 4% NaOH treatment, but was still considerably less than that produced in a Kraft or Sulfite process.

## AREAS FOR INVESTIGATION

The performance of virgin and recycled furnishes can be compared by considering the relative contributions of their long fiber and fines fractions (material passing through a 200 mesh screen). Specific issues associated with recycled furnishes include: the effects of drying and other degradative effects, stickies and contamination, deinking, solid waste reduction, and energy. In our review of the literature, we have noted that fines are an important furnish component (approximately 5% to 30% of the furnish) in spite of their negative impact on water removal. The fines present in a recycled furnish (where the yield is less than 70%) have a greatly impaired performance which may not be easily reversed. The loss in fines performance is not well understood, but is mainly attributed to drying (hornification), with curl, contamination, loss of hemicelluloses, etc., also being contributing factors. The long fiber fraction, as shown in Figure 12, is similarly affected. However, our current focus is on the fines component. If they can be effectively treated, i.e., the effects of drying reversed, then strategies for treatment of the long fiber fraction can be modified accordingly.

The immediate goals of our program are to:

1. Determine the mechanism(s) responsible for the impaired performance of different sources of recycled fines.

2. Determine if chemical and/or chemimechanical treatments can restore or enhance the papermaking performance of recycled fines.

3. Seek external funding to compliment and leverage the Institute's member company dues for the recycling research program.

In what follows, we report on our initial studies of fractionation and chemical treatment to reverse the adverse effects of drying.

A student-related research study titled, "The Effect of Recycling on the Fines Contribution to Selected Paper Properties," is included in Appendix I.

* TREATMENT OF LONG FIBER FRACTION

Defects Present - Hornification, Microcompressions, Curl, Contaminants, Loss of Hemicelluloses.

* TREATMENT OF FINES FRACTION

Defects Present - Hornification, Curl, Contaminants, Loss of Hemicelluloses, Agglomeration, Loss of adhesion.

| **Chemical Treatment** | **Mechanical Treatment** |
| --- | --- |
| Sodium Hydroxide | Refining |
| Ozone | Ultrasonic |
| Peroxide | Homogenizer |
| Enzyme | Colloid mill |
| Mild Kraft Pulping | Small particle impact refining |

**RESWELLING MECHANISMS**

**Mechanical Stress + Osmotic Stress**

**Figure 12 REVERSING THE EFFECTS OF DRYING**

# EXPERIMENTAL

The recycled fiber used in this study was a commercial source of old corrugated containers (OCC). This was fractionated using commercial fraction equipment. The Canadian Standard Freeness (CSF) tester was used primarily as an indicator of hydrodynamic specific surface, and because it is a well-known pulp drainage test. In addition, the pulp pads from the CSF were wet pressed, and after drying and conditioning used for nondestructive and destructive property measurements.

## Long Fiber Fraction Refining and Fines Separation

Secondary fines, i.e., $S_1$, were generated from the long fiber fraction of the OCC in a PFI mill using 24 gms of dried fiber, and a consistency of 10%, over the range of 0 to 10,000 revolutions. The fines, i.e., $S_1$, were separated from the long fiber fraction using the TAPPI Britt Jar recommended procedure T261 cm-90.

## Sodium Hydroxide Treatment of Fines Fraction

The level of caustic treatment was 0%, 1%, 2%, 4%, 6%, 8%, and 10%. Fines were treated at room temperature for 45 minutes, and neutralized to a pH of 7 using $H_2SO_4$.

## Canadian Standard Freeness and Property Measurements

In order to conserve on the amount of fines used and treated, it was decided to try and get more "mileage" out of the CSF measurement. The basic idea is to carefully remove the wet pad from the tester, couch and wet press it, and then dry the pad under full restraint.

The nominal grammage of the CSF pad is 370 $g/m^2$, and is removed from the tester using a "piston," consisting of a circular metal disk attached to a rod. Prior to removal the pad is gently dewatered by increasing the pressure on the disk. Depending on the freeness, the pad will most times adhere to the metal disk facilitating its removal. After couching the pad is placed between blotters, i.e., two below and one above the pad, and wet pressed for 5 minutes at 50 psi. Prior to couching and wet pressing a synthetic filter disk is placed above and below the disk to avoid sticking to the blotter stock.

The pad is weighed prior to drying to determine its consistency after wet pressing. It is dried between two dryer felts for 15 minutes at a platen temperature of 300°F. Sufficient pressure is applied to the pad during drying to ensure that it is dried under full restraint.

Physical property measurements on the conditioned pads included: grammage, soft platen caliper measurements (63), and in-plane and out-of-plane elastic constant measurements using techniques developed at IPST (64) (65). Compressive strength measurements (STFI) were made following TAPPI recommended procedure T826.

# RESULTS AND DISCUSSION

## FRACTIONATION TRIALS AT BLACK CLAWSON

Fractionation trials were conducted at Black Clawson by Dr. Jack Firkins (prior to leaving IPST Jack was leader of the recycling team). The furnish consisted of 90% corrugated clippings and 10% OCC. Fractionation trials were run at 2000 ft/min and 5000 ft/min using a double-nip thickener; however, because of inconsistencies in freeness with the 2000 ft/min trial, our subsequent work has been done with material fractionated at 5000 ft/min. The main results of the fractionation trials at 5000 ft/min (DNT 771) are summarized in Table 4 under these conditions, the fines fraction was 12.4%.

TABLE 4 BLACK CLAWSON DOUBLE NIP THICKENER FRACTIONATION RESULTS.

| TYPE | CONSISTENCY % | C.S.F. ml |
|---|---|---|
| FEEDSTOCK | 1.15 | 517 |
| LONG FIBER | 12.9 | 731 |
| FINES (12.4%) | 0.16 | - |

Approximately 1.9 lbs, 11.6 lbs, and 4.6 lbs O.D. of the feed, long fiber, and fines fraction, respectively, were sent to IPST. The fines were thickened using a laboratory centrifuge to a consistency of about 15%.

## CHEMICAL TREATMENT OF FINES

One of the initial objectives of our program was to screen chemical treatments for their ability to rejuvenate the fines fraction of the pulp. We believe that as a result of drying the papermaking potential of fines can be adversely affected by hornification (loss of swelling and flexibility), agglomeration, curl, adhesion, etc.

The experimental design included the following chemical treatments: sodium hydroxide, ozone, and hydrogen peroxide. Treated fines were added at 15% and 30% levels to the unrefined long fiber fraction, and two levels of wet pressing, 50 psi and 100 psi, were used. The nominal basis weight of the handsheets was 150 $g/m^2$. The performance of the chemically treated fines is also to be compared with secondary fines generated by refining the long fiber fraction.

The design is summarized in Table 5, and the properties measured include: C.S.F., grammage, calipers, elastic properties, tensile, and compressive strength properties.

TABLE 5 EXPERIMENTAL DESIGN FOR CHEMICAL TREATMENT OF FINES.

| FURNISH | FINES ADDITION LEVEL | FINES TREATMENT | WET PRESSING |
|---|---|---|---|
| WHOLE PULP | (12.5%) | None | High & Low |
| LONG FIBER | 0% | None | High & Low |
| LONG FIBER | 15% & 30% | 2% & 10% NaOH | High & Low |
| LONG FIBER | 15% & 30% | 3% $O_3$ | High & Low |
| LONG FIBER | 15% & 30% | 3% $H_2O_2$ | High & Low |

* secondary fines $S_1 + S_2 + ...$ are generated by refining the long fiber fraction in a PFI mill.

**PFI REFINING OF LONG FIBER FRACTION**

The long fiber fraction was refined in a PFI mill over the range of 0 to 10,000 revolutions. The variation of CSF with PFI revs. is shown in Figure 13 together with the amount of fines produced. There is close to a linear variation of fines production with PFI revs. Also shown in Figure 13 is the CSF of pulp which has been fractionated at each beating interval. The drop in CSF of the fines-free pulp with PFI revs. is attributed to internal and external fibrillation.

Figure 14 shows the variation of CSF with fines content for both secondary $S_1$ and "primary" ($P_{0d1} + S_{0d1}$ +..) fines. With regard to the secondary fines, we have taken the fines produced at each refining interval shown in Figure 13, i.e., 3000, 5000, and 10,000 revs., and added various percentages of them to the long fiber fraction. The resulting CSF curve is in close agreement with the original curve indicating, at least for this case, that the fines produced at say 3000 revs. are no different in character from 10,000 revs. This is in agreement with the findings of Retulainen, Moss, and Nieminen (18).

The curve denoted "primary" ($P_{0d1}$ + $S_{0d1}$) in Figure 14 is derived from the feedstock to the DNP which has a CSF of 517 ml as shown in Table IV. Values of CSF were also measured for 15% and 30% addition of the untreated fractionated fines ($P_{0d1}$ + $S_{0d1}$). We note that there is a large difference in the freeness performance of the "primary" and secondary fines, i.e, for a given level of fines addition, the "primary" fines result in a higher freeness than the secondary fines. CSF is, in spite of its various critics, a sensitive indicator of hydrodynamic specific surface. The difference in freeness level is, therefore, attributed to a loss in surface area due to drying. From one perspective, we might expect that successful treatment of the "primary" fines will result in an increase in hydrodynamic specific area and a concomitant drop in freeness at a given level of fines addition.

The CSF of fines which have been chemically treated with sodium hydroxide, ozone, and peroxide are shown in Figure 15. These values should be compared with the "primary" and secondary curves reproduced from Figure 14. With the exception of the sodium hydroxide data points, deviations from the "primary" curve for ozone- and peroxide-treated fines are not large. One has to exercise caution in interpreting changes in CSF with respect to changes in drainage and strength. Treatment with 2% sodium hydroxide does show a reduction in freeness, while at 10% the freeness is almost equal to the fines-free pulp. This dramatic change in freeness has also been found by Giertz (33) who treated a rayon grade spruce sulfite in 10% NaOH for 3 hours at 120°C. The control pulp and the extracted pulp were refined ultrasonically, and both showed significant external fibrillation. The properties of Giertz's (33) pulps are summarized in Table 6.

TABLE 6. EFFECT OF HOT ALKALI EXTRACTION ON PULP PROPERTIES-GIERTZ (33).

| PROPERTY | CONTROL | 10% NaOH EXTRACTED 3hrs @ 120°C |
|---|---|---|
| YIELD | 40 | 31 |
| % HEMICELLULOSE | 6.9 | 2.1 |
| °SR | 90 | 19 |
| TENSILE Nm/g | 69 | 19 |

Pulp: Rayon grade spruce sulfite.

Giertz (33) argues that hemicellulose losses are responsible for the above changes. No specific mention is made of changes from cellulose I to cellulose II or possible changes in geometry which might also be a factor.

## HANDSHEET PROPERTY RESULTS

Handsheets were made according to the experimental design shown in Table 5 and then conditioned following TAPPI procedures. Figures 16 through 18 show, respectively, the variation of modulus, tensile and compressive strength with fines content for control, ozone- and peroxide-treated fines at two levels of wet pressing. We note that for the control handsheets fines addition and wet pressing improves the strength properties. However, for the chemically treated fines, the change with wet pressing is much smaller, and furthermore, the properties do not differ significantly from the controls.

For clarity, the 10% sodium hydroxide and ozone results are compared in Figures 19 through 21. We see that there is a large loss in properties with this level of treatment. These findings are in agreement with Giertz's results (33), see also Table 6. In contrast to the chemical treatments, we see that wet pressing is effective in improving strength properties. This may be due to there being less water associated with the 10% NaOH treated fines. Despite the improvement due to wet pressing, the performance of the 10% NaOH treated fines is still below the control fines.

**TREATMENT OF FINES WITH SODIUM HYDROXIDE 0% TO 10%**

For each NaOH treatment level, CSF measurements were made on blends containing 15% and 30% fines. The variation of CSF with caustic treatment level is shown in Figure 22. We note that freeness drops initially and then rises to approach the freeness level of the fines-free pulp at 10% NaOH.

This dramatic change in freeness has also been found by Giertz (33) who treated a rayon grade spruce sulfite in 10% NaOH for 3 hours at 120°C. The control pulp and the extracted pulp were refined ultrasonically, and both showed significant external fibrillation. The properties of Giertz's (33) pulps are summarized in Table 6.

Giertz (33) suggests that hemicellulose losses are responsible for the above changes. No specific mention is made of changes from cellulose I to cellulose II, or possible changes in fiber cross-sectional geometry, i.e., fibers becoming more circular, which might also be a factor in such a treatment (66).

Using light microscopy, photographs of the untreated OCC fines, and treatment with sodium hydroxide at 2%, 6%, and 10% are shown in Figures 23, through 26. We note that with increasing sodium hydroxide concentration the fines appear to form more compact flocculated structures. It was not possible to break down these structures with a gentle stirring action. Increasing flocculation of the fines with increasing sodium hydroxide concentration might also be responsible for the increase in CSF and other property changes to be discussed.

From a careful accounting of the fines lost to the filtrate in running the freeness test, an estimate of the fines lost from those added is shown in Figure 27. Interestingly, fines loss passes through a maximum at around 1% NaOH treatment level. It is not clear why the fines loss should be the highest at this point, but in view of the minimum in freeness, this might imply that the effective viscosity of the filtrate is increased due to fines loss.

Property measurements on the conditioned CSF pads are shown in Figures 28, through 31.

Fines at 15% and 30% addition levels increase apparent density; however, as shown in Figure 28, sodium hydroxide treatment does not appear to significantly affect densification. Furthermore, the dryness of the pad after wet pressing was 44% and unaffected by fines level and sodium hydroxide treatment.

The out-of-plane elastic constant $Ez/\rho$ increases with both fines addition and caustic treatment as shown in Figure 29, whereas the in-plane elastic constant $C/\rho$ rises initially and then decreases with increasing sodium hydroxide level as shown in Figure 30. The changes in elastic properties shown in Figures 29 and 30 may be an indication of changes in cellulose structure, for example, conversion of cellulose I to II and/or changes in fines geometry as illustrated by the flocculated structures shown in Figures 24, through 26. The three-dimensional structure of the flocculated fines is consistent with the increase in out-of-plane elastic constant and the concomitant drop in in-plane elastic constant at high levels of sodium hydroxide treatment.

The variation of short span compressive strength is shown in Figure 31, and the variation is consistent with the elastic property behavior shown in Figures 29 and 30, according to the simplified model of Habeger and Whitsitt (67).

# CONCLUSIONS

The Black Clawson double-nip thickener is an effective means of fractionating large quantities of pulp, in this case, OCC.

The secondary fines $S_1$ generated by refining the long fiber fraction of OCC have a much larger hydrodynamic surface area than the so-called "primary" fines ($P_{0d1}$ + $S_{0d1}$) as evidenced by changes in CSF at different levels of fines addition to the unrefined long fiber fraction.

Initial screening of peroxide and ozone to rejuvenate the fines has not resulted in any dramatic changes in properties, i.e., freeness and strength.

Property measurements made on CSF pads which have been wet pressed and dried show promise as a technique for initial pulp characterization studies.

The treatment of fines with sodium hydroxide in the range of 0% to 10% has a significant effect on CSF, elastic, and strength properties. Freeness passes through a minimum at around 1% NaOH, and then rises to almost equal the freeness of the unrefined long fiber fraction at 10% NaOH. Water removal by wet pressing also appears to be independent of fines and sodium hydroxide levels. For both 15% and 30% fines addition, the in-plane elastic constant increases initially, reaching a maximum around 1% to 2% NaOH, after which it decreases to just below the level of 0% NaOH treatment. Both cellulose structure and changes in fines geometry may be responsible for this behavior.

In future studies, we hope to examine chemimechanical treatment of fines, as well as treatment of the long fiber fraction.

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## LIST OF FIGURES

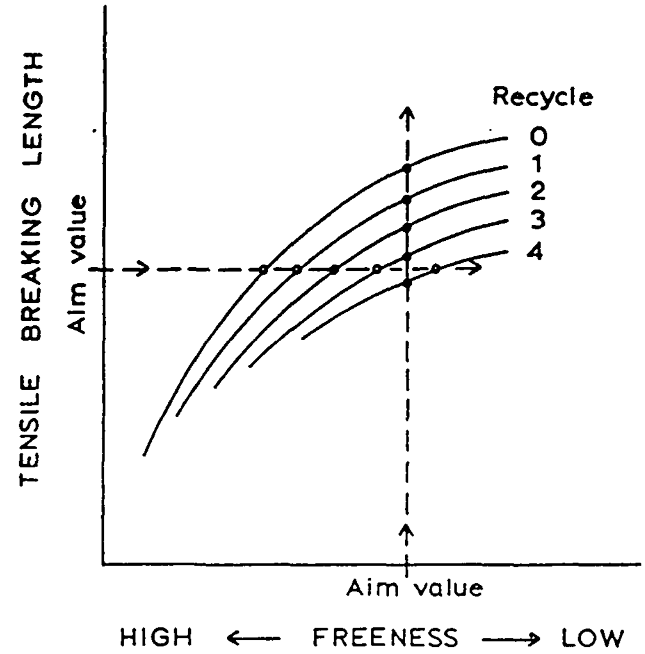


Fig. 5 — Breaking length versus freeness of recycled pulps.

**Figure 1** Variation of Tensile Strength with Freeness with Recycling - Cardwell, R.D. and Alexander, S.D. (16).

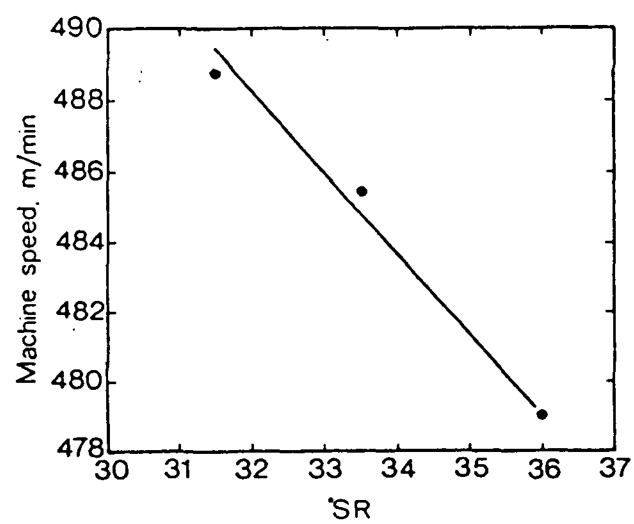


**Figure 2** Machine Speed Versus Drainage - de Ruvo, A. and Htun, M. (9).

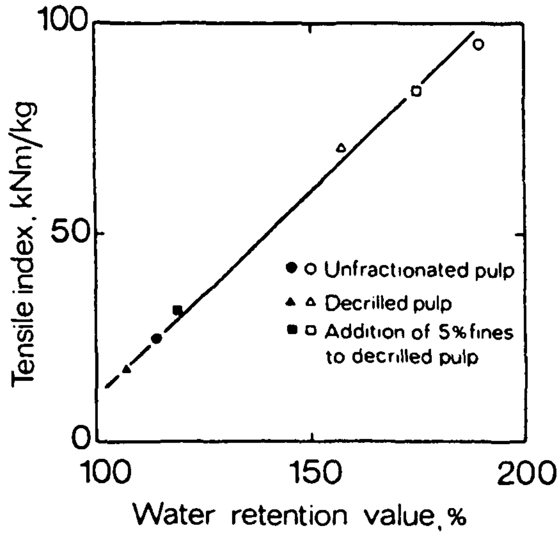


Fig 2—The relation between tensile index and WRV for handsheets made from unbeaten and beaten pulps containing fines or without fines
Symbol key: unbeaten = filled symbols, beaten = unfilled symbols

**Figure 3** Variation of Tensile Index with Water Retention Value with and without Fines for Unbeaten and Beaten Pulps - de Ruvo, A. and Htun, M. (9).

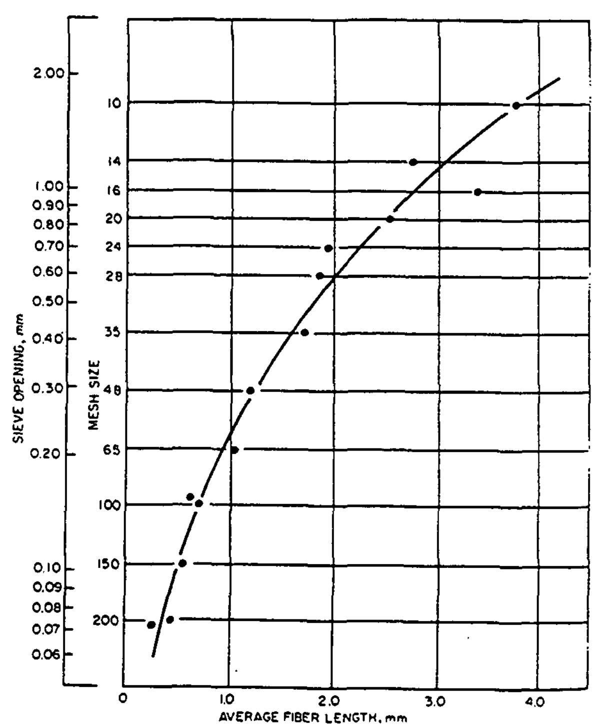


**Figure 4** Variation of Sieve Opening with Average Fiber Length - Tasman, J.E. (22).

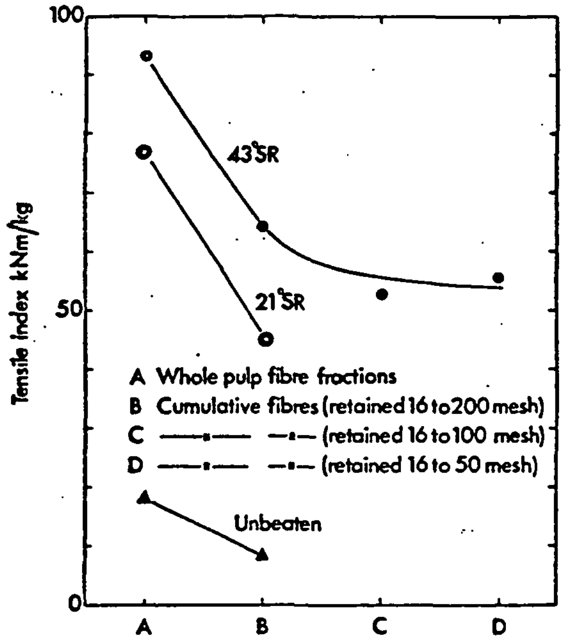


*Fig. 4.* The effect of fines (<200 mesh) on the strength of sheets made from bleached kraft beaten to different °SR. The fiber fractions retained on 50—200 mesh screens show very little effect on the tensile index.

**Figure 5** Effect of Fines Addition (< 200 mesh) on Strength of Paper Made from a Bleached Kraft Pulp Beaten to Different Levels of °SR - Htun, M. and de Ruvo, A. (26).

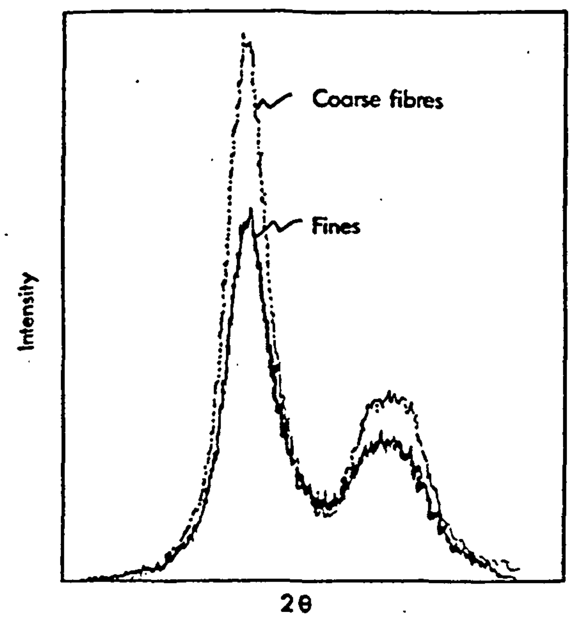


*Fig. 3.* X-ray diffractograms for fines and coarse fibers of a bleached kraft pulp.

**Figure 6** X-Ray Diffraction Spectra of Coarse Fibers and Fines - Htun, M. and de Ruvo, A. (26).

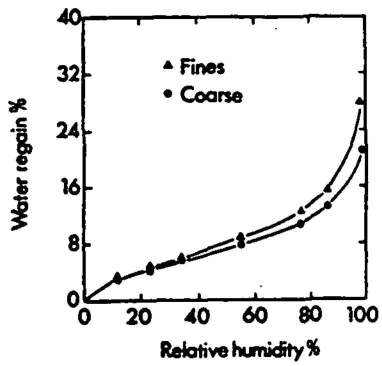


*Fig. 2.* Water sorption isotherms at 25°C for the fines and coarse fiber fraction of bleached kraft pulp.

**Figure 7** Sorption Isotherms for Coarse Fibers and Fines - Htun, M. and de Ruvo, A. (26).

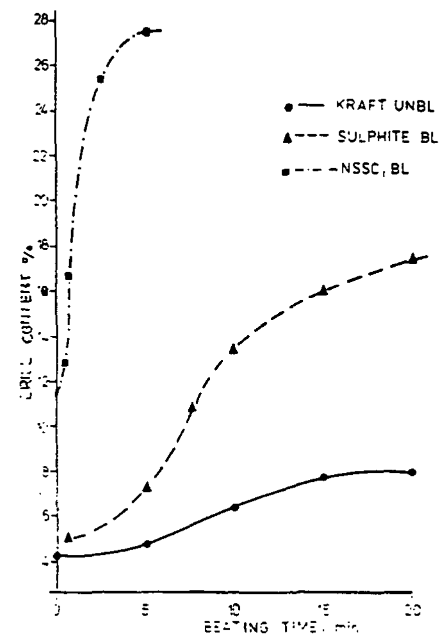


Fig. 1. Crill formation during beating of various pulps in a PFI mill

**Figure 8** Rate of Fines Generation - Mancebo, R. and Krokoska, P. (19).

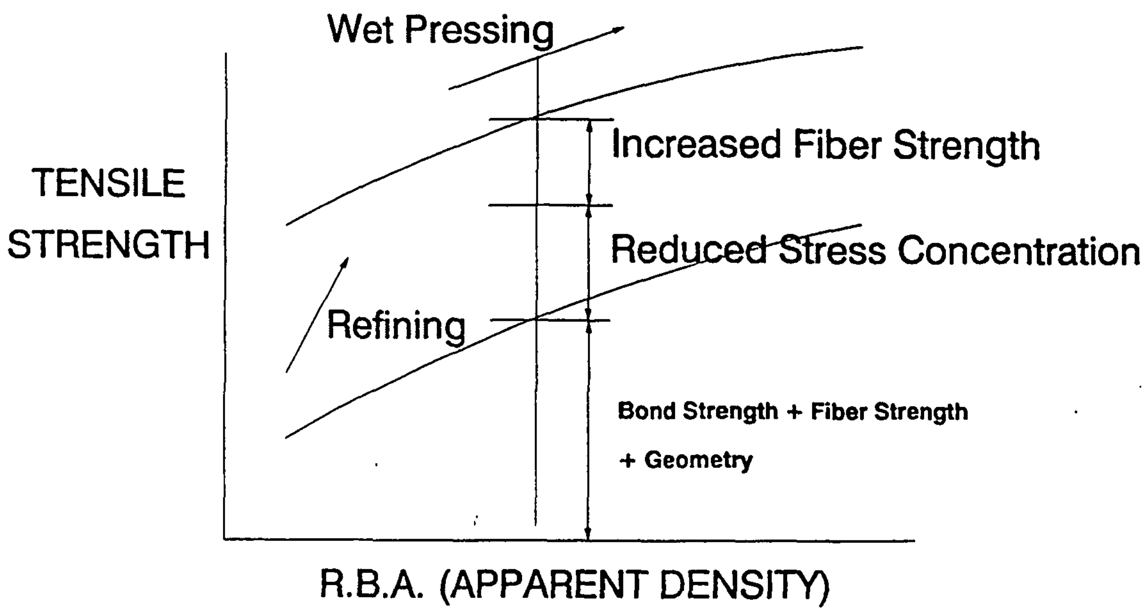


**Figure 9** The Effect of Refining and Wet Pressing on the Tensile Strength Variation with Apparent Density or RBA.

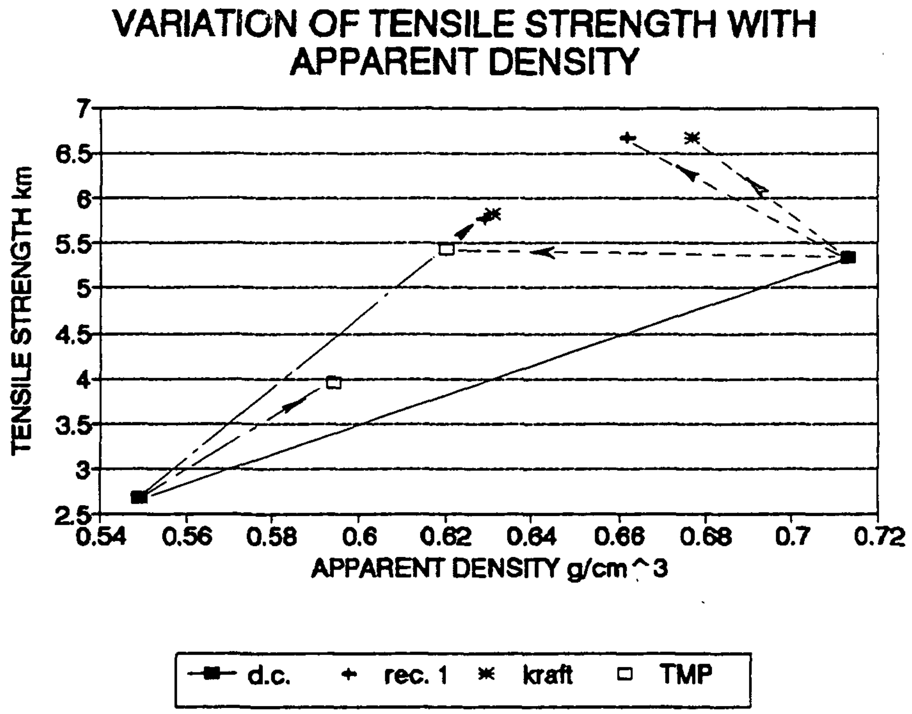


**Figure 10** Variation of Tensile Strength with Apparent Density using Data of Hawes, J.M. and Doshi, M.R. (21).

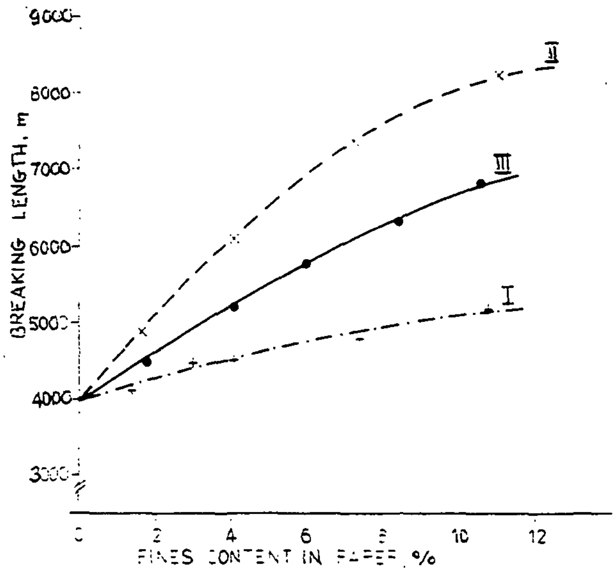


Fig. 5. Influence of the addition of waste paper fines on paper strength
I - Primary fines, II - Secondary fines, III - Whole fines from highly beaten waste paper (70 SR)

**Figure 11** Tensile Strength of Handsheets Containing Different Types of Fines - Mancebo, R. and Krokoska, P. (19).

**Figure 12** Reversing the Effects of Drying (already in text).

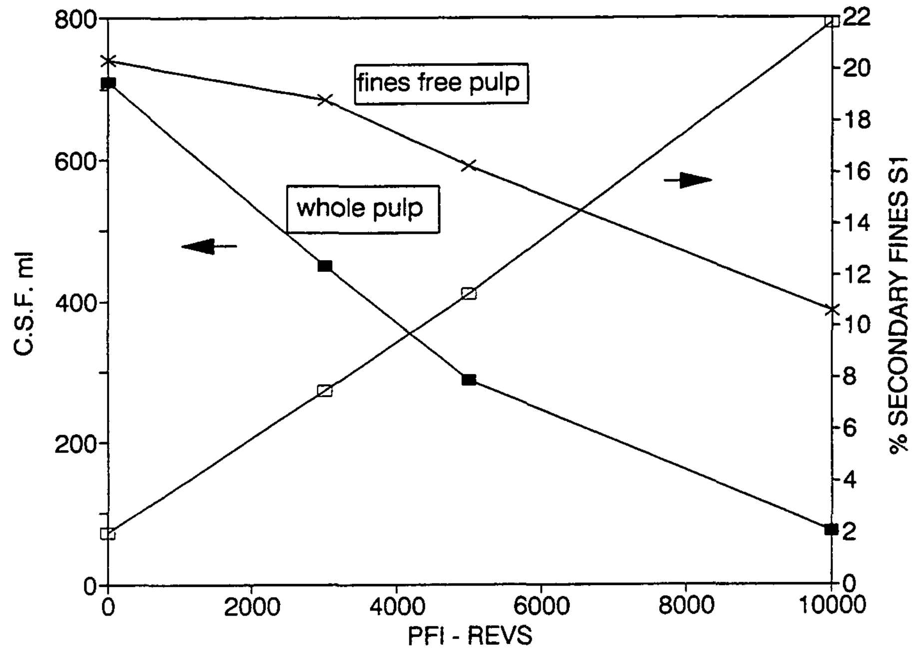


**Figure 13** Variation of Canadian Standard Freeness and Fines Content with PFI Revolutions for Refining of Fines-free OCC.

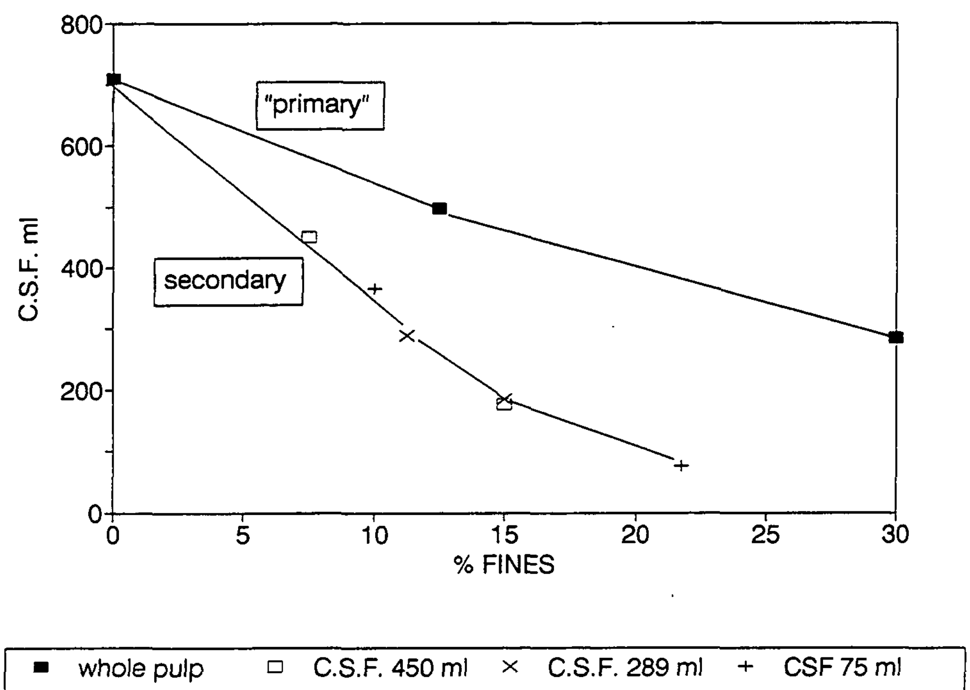


**Figure 14** Variation of Canadian Standard Freeness with Fines Content for "Primary" and Secondary Fines.

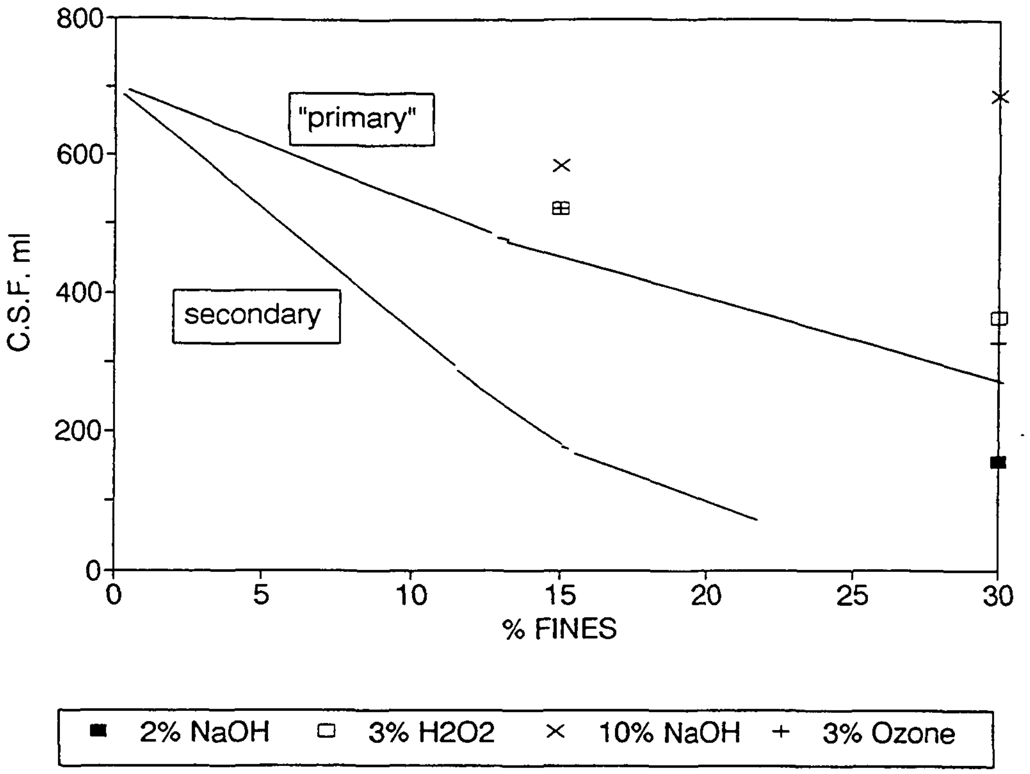


**Figure 15** The Effect of Chemical Treatments on the Variation of Canadian Standard Freeness with Fines Content.

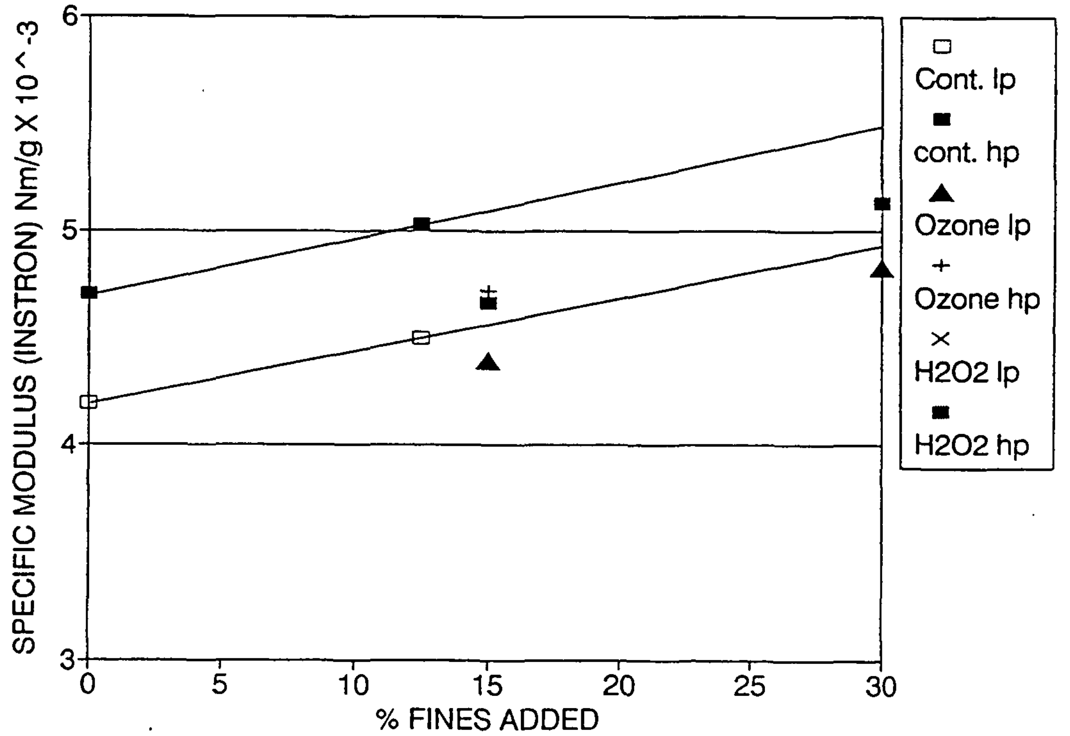


**Figure 16** The Effect of Ozone and Peroxide Treatment of Fines on the Variation of In-Plane Specific Modulus (Instron) with Fines Content.

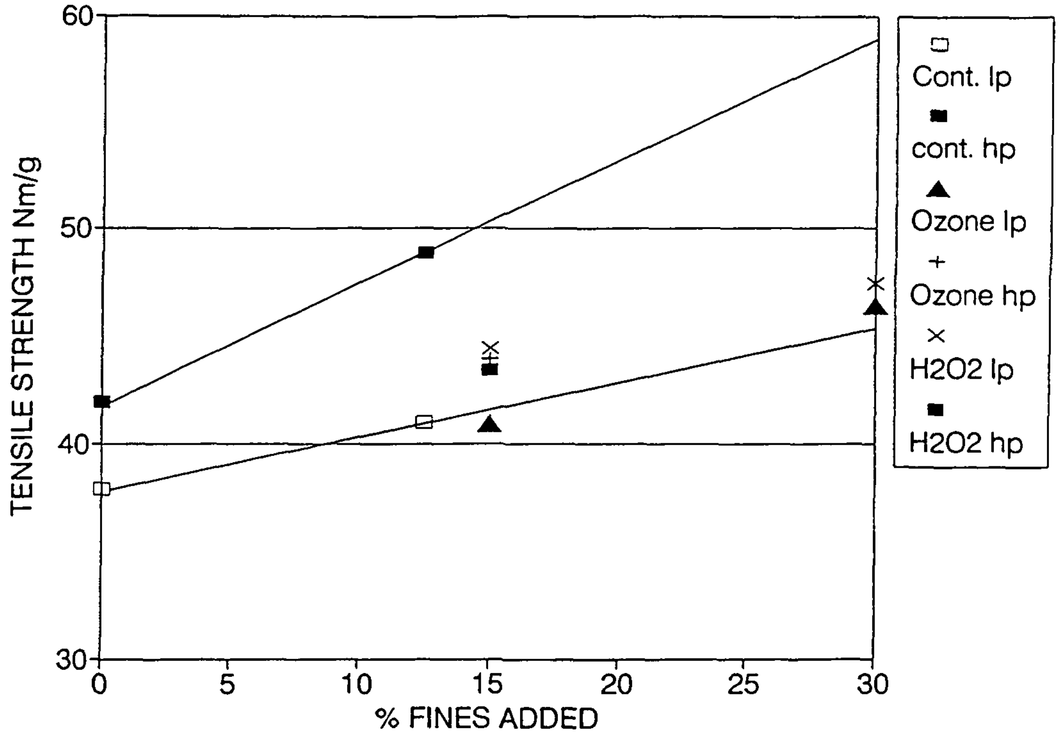


**Figure 17** The Effect of Ozone and Peroxide Treatment of Fines on the Variation of Tensile Strength with Fines Content.

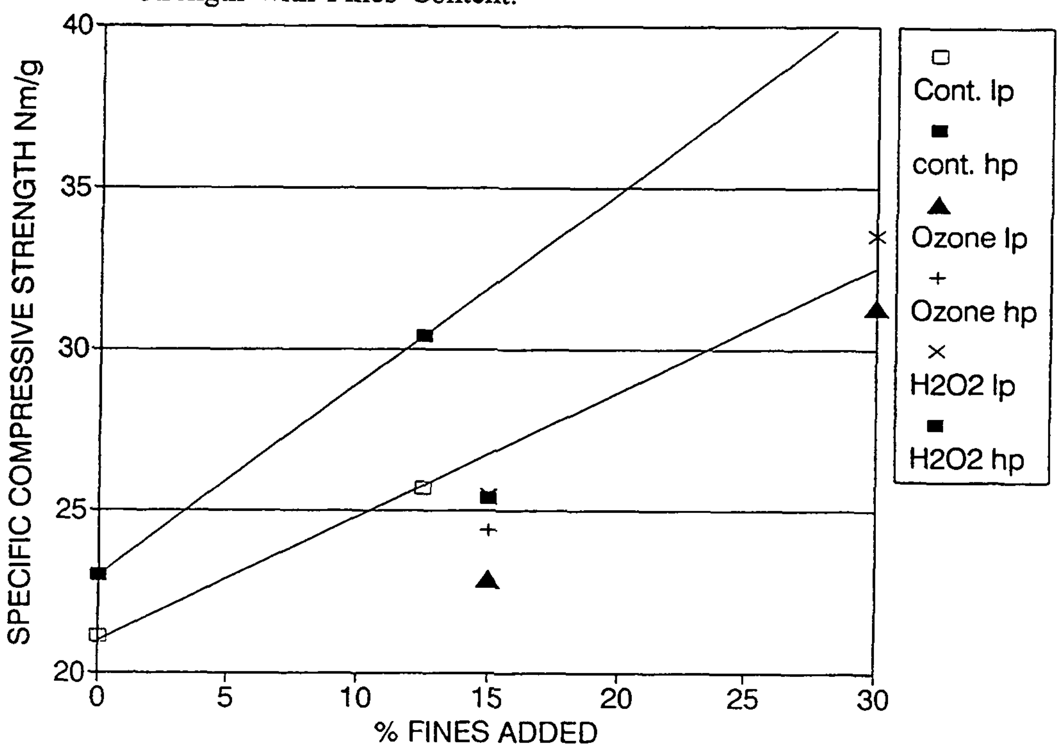


**Figure 18** The Effect of Ozone and Peroxide Treatment of Fines on the Variation of Compressive Strength with Fines Content.

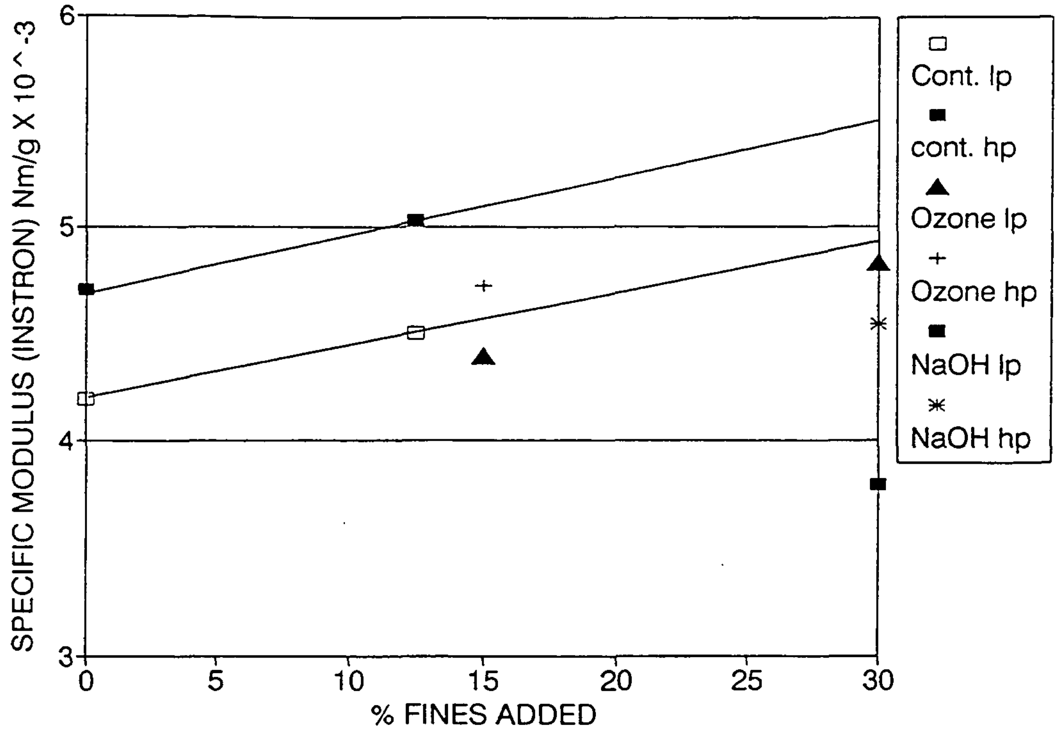


**Figure 19** The Effect of Ozone and Sodium Hydroxide Treatment of Fines on the Variation of In-Plane Specific Modulus (Instron) with Fines Content.

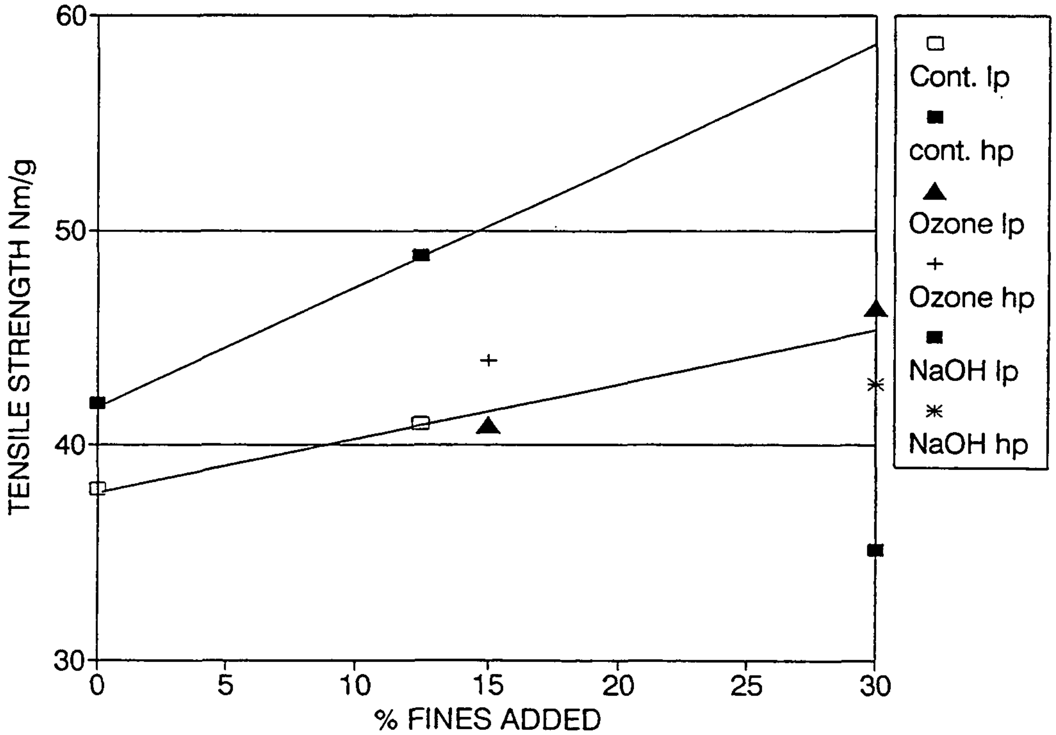


**Figure 20** The Effect of Ozone and Sodium Hydroxide Treatment of Fines on the Variation of Tensile Strength with Fines Content.

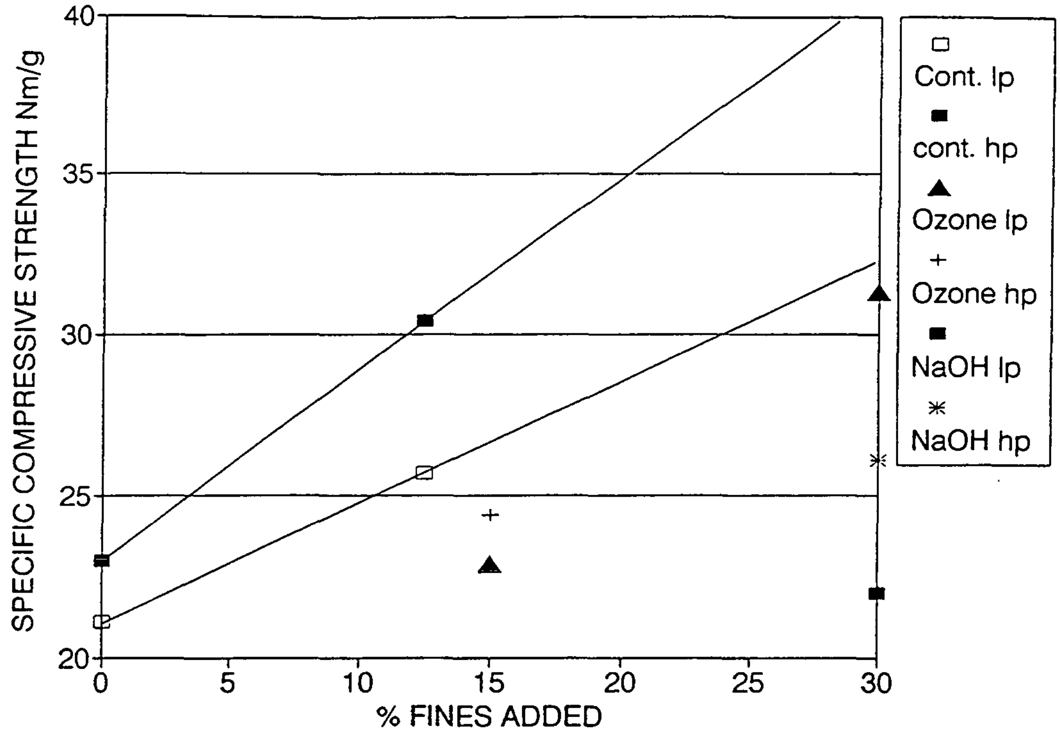


**Figure 21** The Effect of Ozone and Sodium Hydroxide Treatment of Fines on the variation of Compressive Strength with Fines Content.

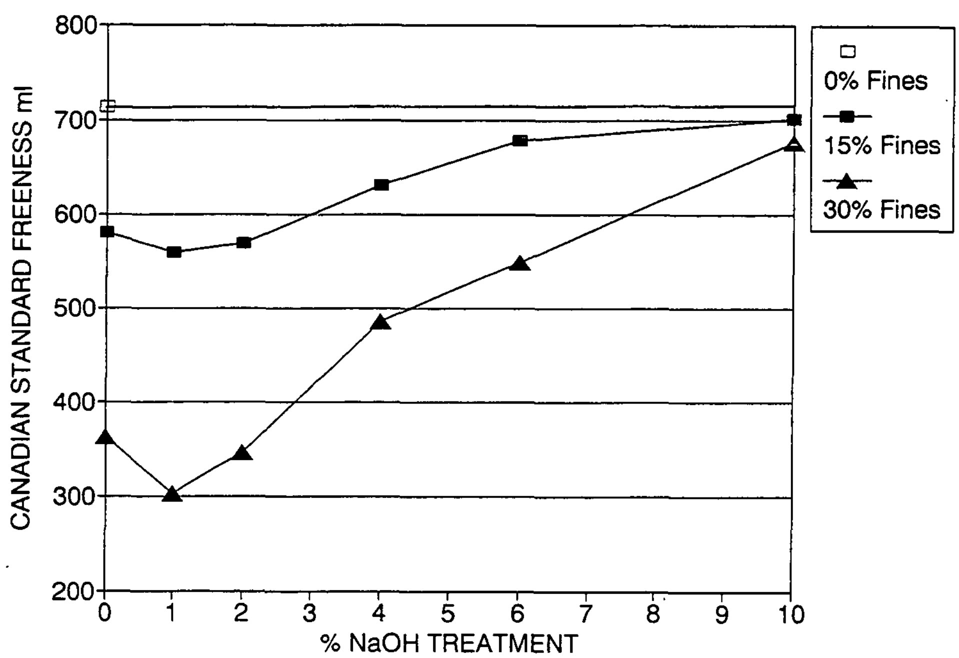


**Figure 22** Variation of Canadian Standard Freeness with Sodium Hydroxide Treatment of OCC Fines.

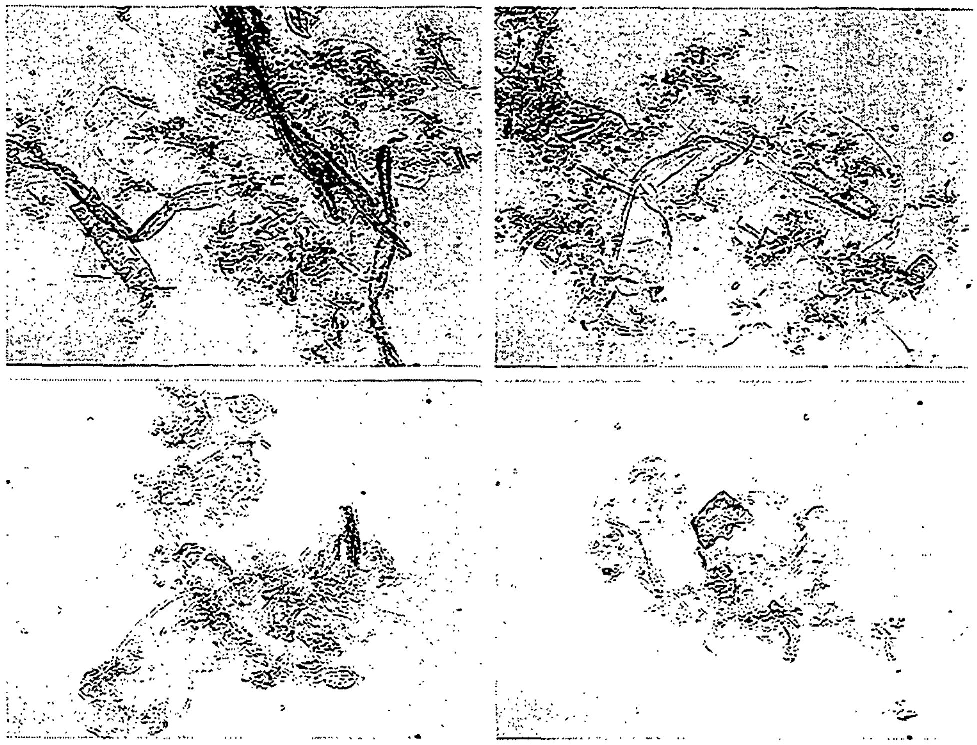

**Figure 23** OCC Fines - Control.

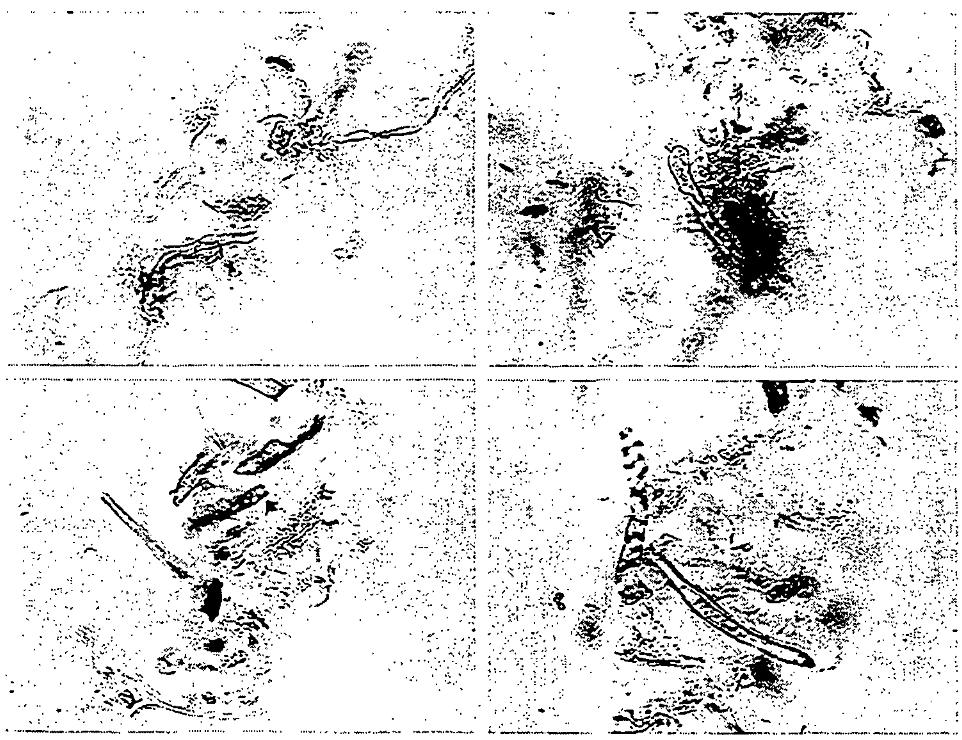

**Figure 24** OCC Fines - 2% NaOH Treatment.

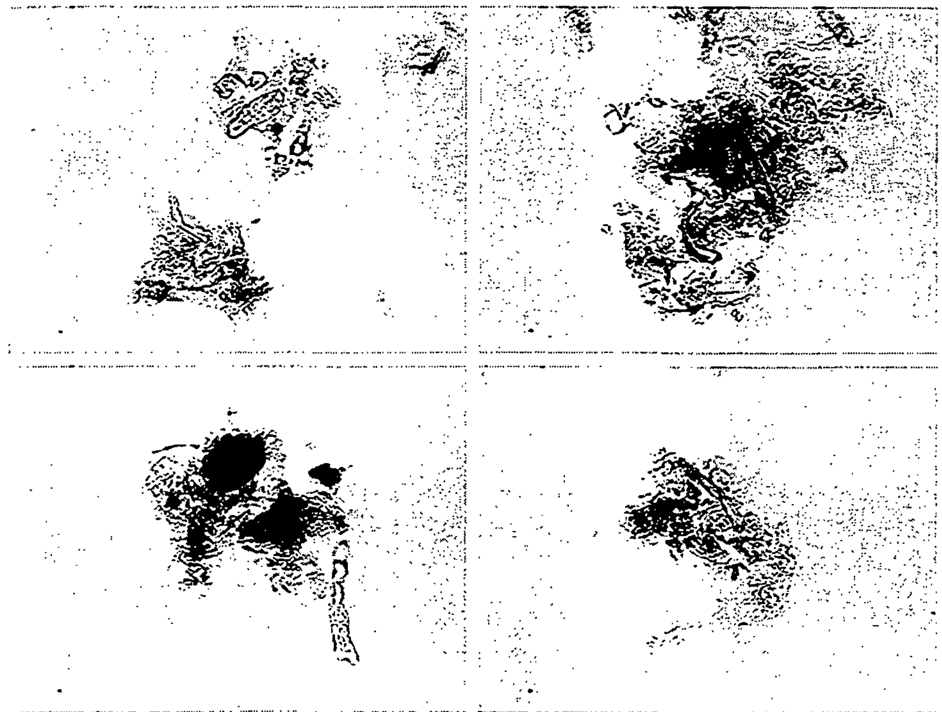

**Figure 25** OCC Fines - 6% NaOH Treatment.

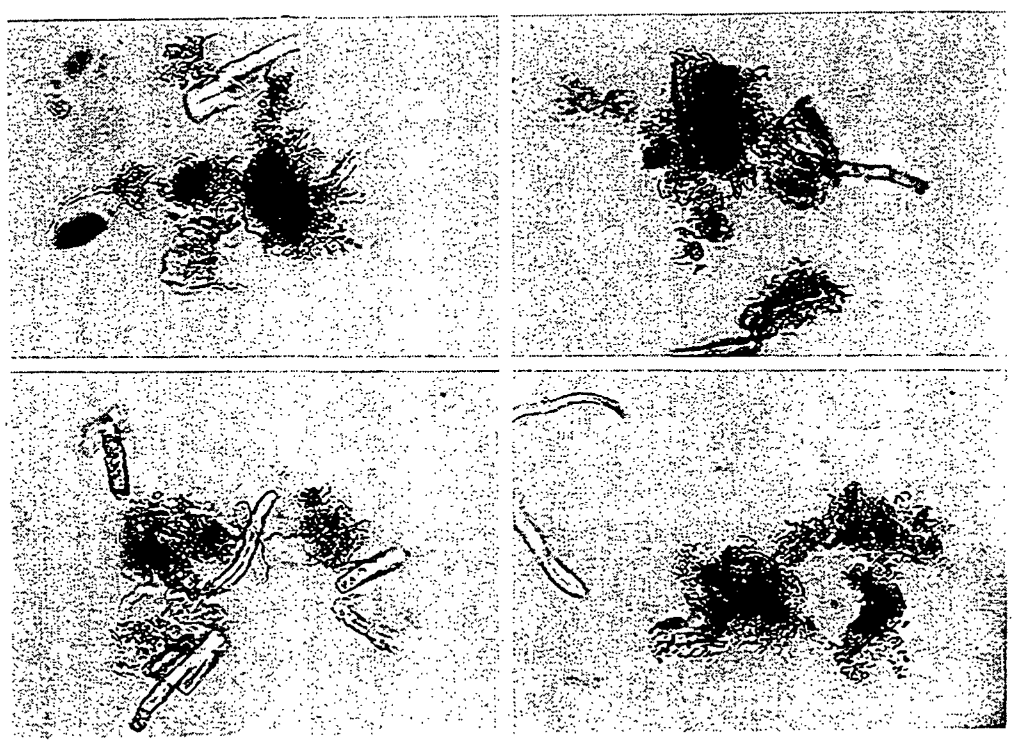

**Figure 26** OCC Fines - 10% NaOH Treatment.

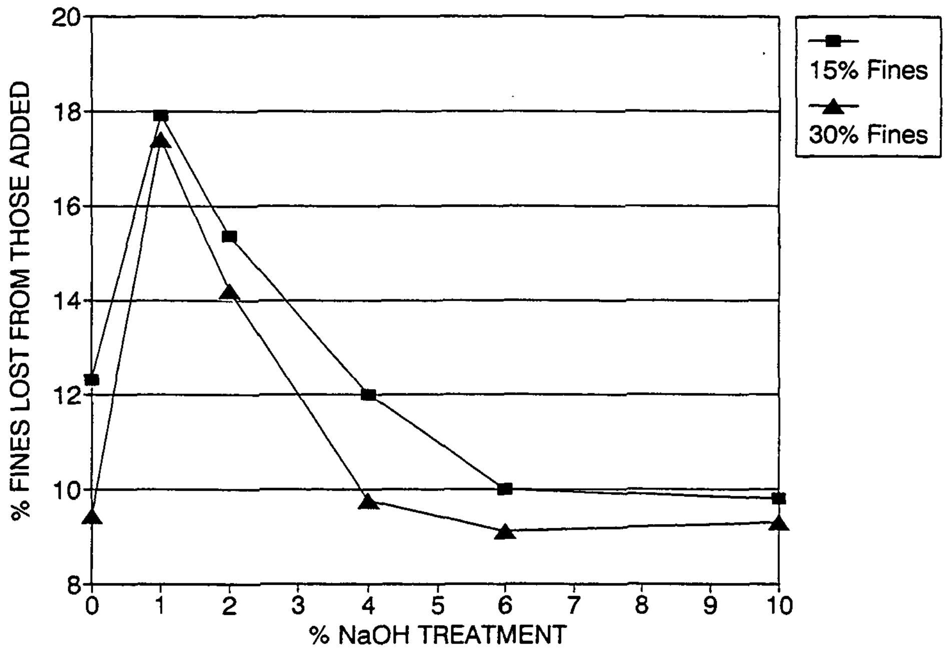


**Figure 27** Variation of Fines Lost During Canadian Standard Freeness Test with Sodium Hydroxide Treatment of OCC Fines.

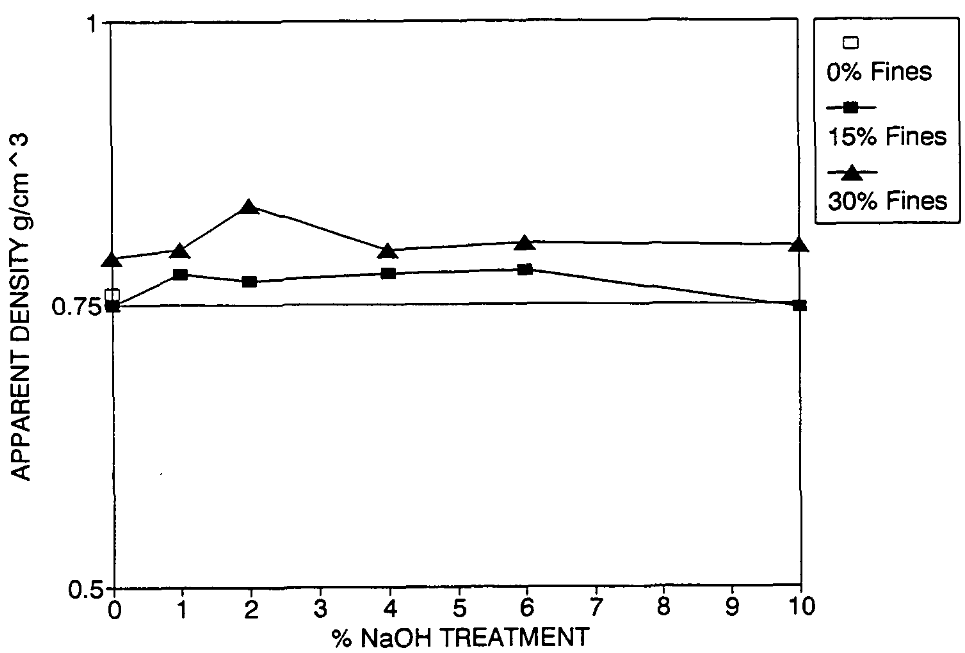


**Figure 28** Variation of Apparent Density with Sodium Hydroxide Treatment of OCC Fines.

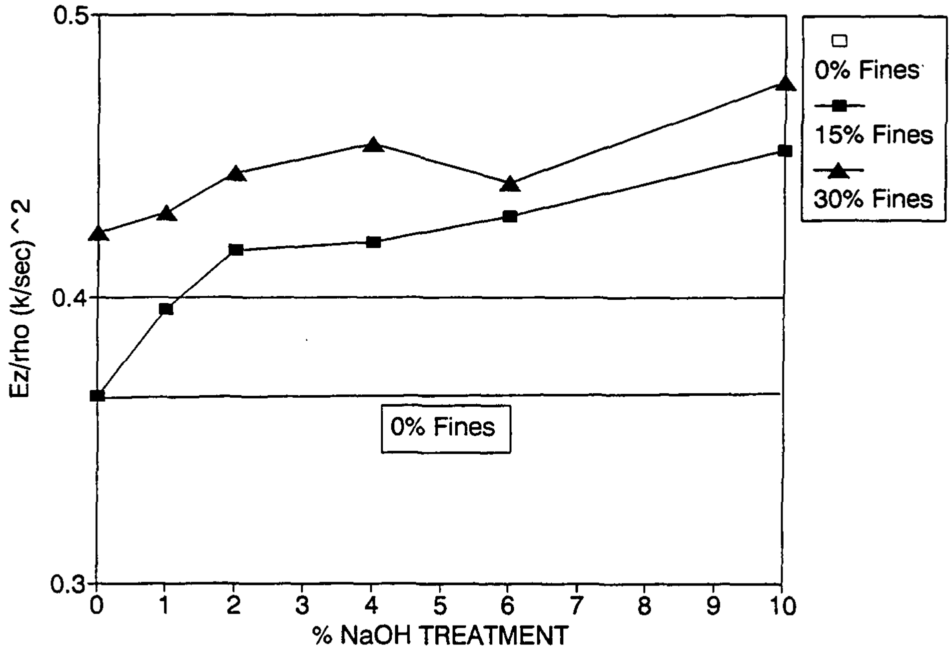


**Figure 29** Variation of Out-of-Plane Longitudinal Specific Modulus with Sodium Hydroxide Treatment of OCC Fines.

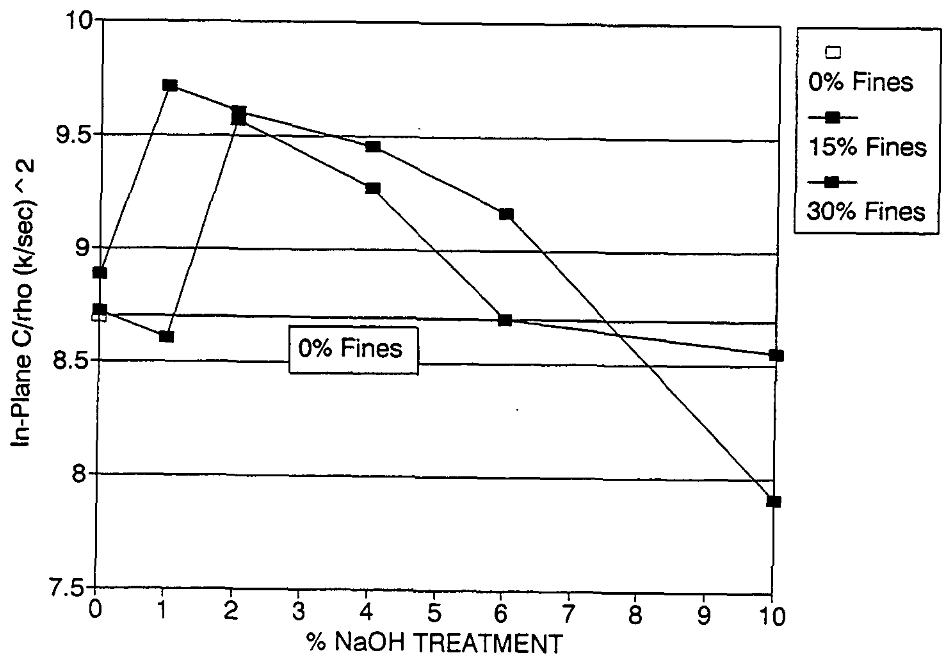


**Figure 30** Variation of In-Plane Longitudinal Specific Modulus with Sodium Hydroxide Treatment of OCC Fines.

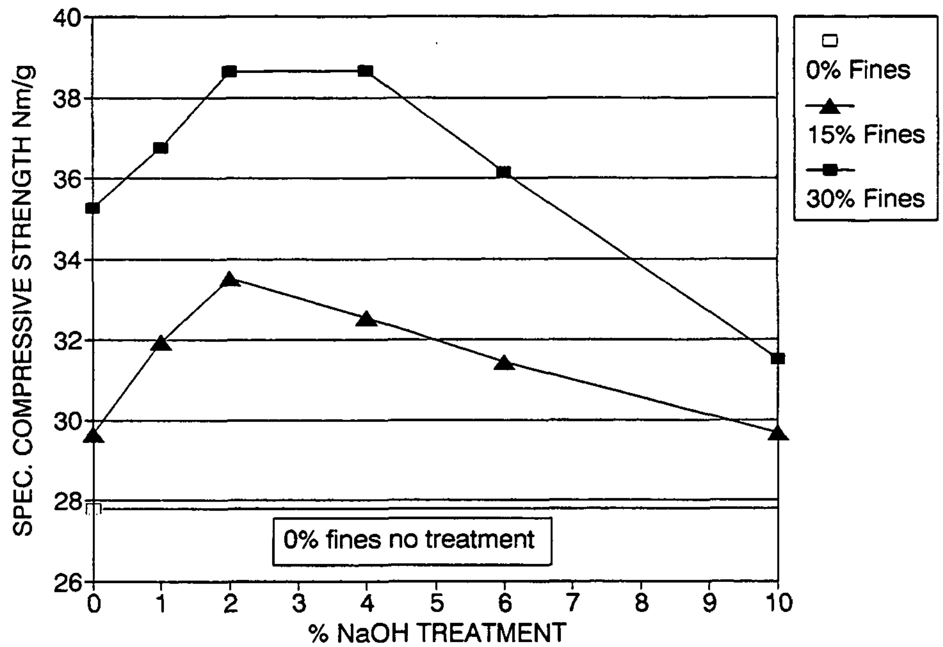


**Figure 31** Variation of Specific Compressive Strength with Sodium Hydroxide Treatment of OCC Fines.

## APPENDIX I

"The Effect of Recycling on the Fines Contribution to Selected Paper Properties"

John F. Waterhouse and Keiichi Omori

IN VOLUME 2

TRANSACTIONS OF THE 10TH FUNDAMENTAL RESEARCH SYMPOSIUM, OXFORD, SEPTEMBER 1993

Edited by C.F. Baker

# PRODUCTS OF PAPERMAKING

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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.

## LITERATURE REVIEW

### 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 (1). 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.



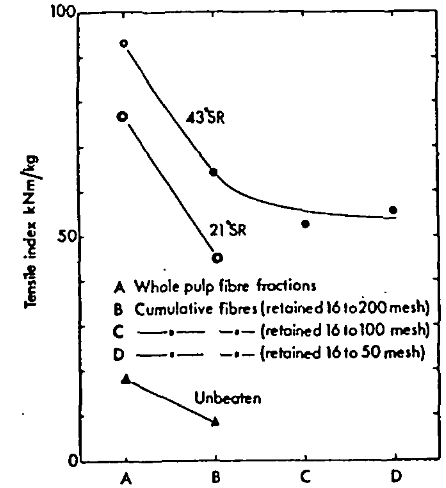


Fig 1 The effect of fines addition (<200 mesh) on the strength of paper made from a bleached kraft pulp beaten to different °SR (Taken from Htun and de Ruvo (2))

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 larg influence on the drainage properties of the pulp. With crill removal sheet densit decreased resulting in an increase in tear and a decrease in tensile strength. It wa stated that the loss in tensile strength, attributed to a loss in density, could b compensated for by an increase in wet pressing, although no evidence was provided t 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 µm and length 40-70 µm. 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.



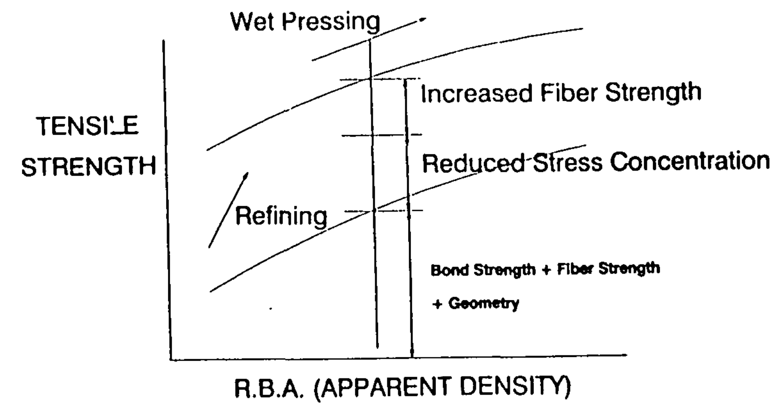


Fig 2 Strength Development by Refining and Wet Pressing

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 (11). 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 (11), 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 (12) 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. (13) 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.(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 (15) also found that fines and fibers become hornified with recycling, and that strength properties decrease.

On the other hand, Hawes and Doshi (16) 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 (16), 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.

## Handsheet Testing Procedures

Nondestructive measurements included grammage, hard and soft platen (17) caliper, in-plane and out-of-plane elastic constants, formation, and porosity. The elastic constants were made using ultrasonic wave propagation techniques developed at IPST (18), (19). Formation measurements (optical and mass density) were made using the IPST formation tester (20). 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.

TABLE 3 BAUER McCNETT CLASSIFICATION RESULTS

| SCREEN SIZE | >14 % | 14-28 % | 28-48 % | 48-200 % | <200 % | FINES LOSS % |
|---|---|---|---|---|---|---|
| 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 |



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.

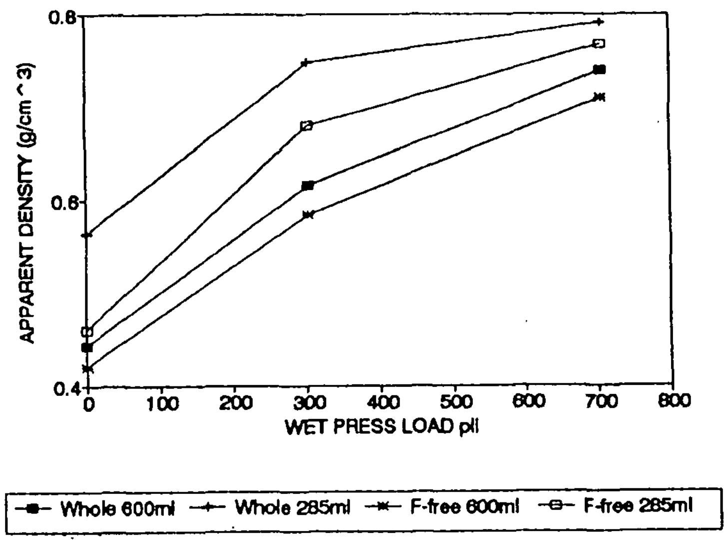


Fig 6 Dependence of sheet densification on wet press load and the effect of refining and fines removal

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 to a higher level of wet pressing and were further dried in an air circulating oven at 105° C for one hour. |
| "Secondary Fines"(H) | same as "Secondary Fines," but the handsheets were subjected to a higher level of wet pressing and were further 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$^2$. 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$^2$, 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.

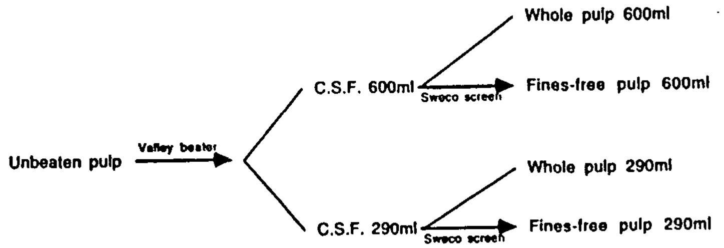


Fig 4 Outline of experiment 1

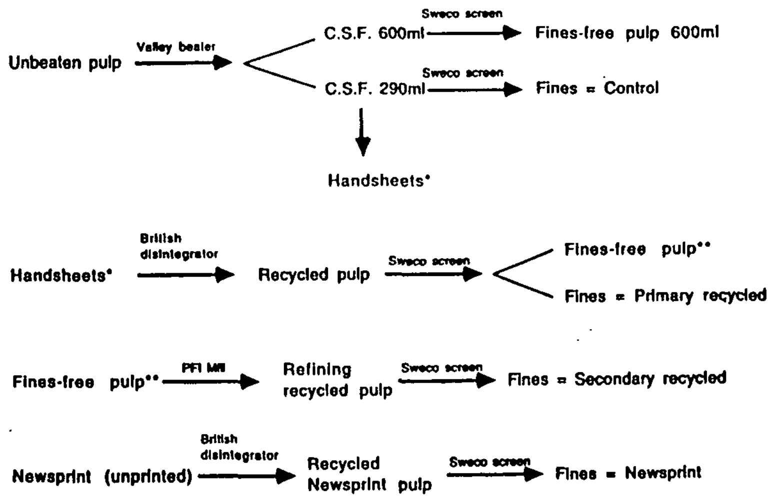


Fig 5 Outline of experiment 2



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 | | | | | | |
| $t_D$ = 30 min | 680 | 54.6 | 17.9 | 13.6 | 7.2 | 6.7 |
| NEWSPRINT | | | | | | |
| $t_D$ = 15 min | 180 | 12.9 | 18.2 | 21.6 | 19.6 | 27.7 |
| $t_D$ = 30 min | 130 | 13.4 | 18.2 | 21.2 | 18.5 | 28.7 |
| TISSUE | | | | | | |
| $t_D$ = 2 min | 700 | 50.3 | 25.6 | 13.1 | 6.9 | 4.1 |
| $t_D$ = 30 min | 650 | 44.5 | 27.6 | 12.8 | 6.7 | 8.4 |
| NOBLE AND WOOD HANDSHEETS (290 ml CSF) | | | | | | |
| $t_D$ = 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

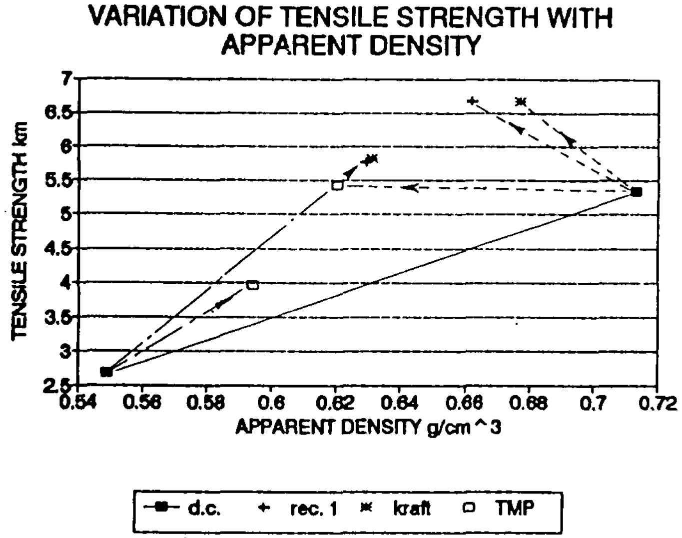


Fig 3 Variation of tensile strength with apparent density based on data of Hawes and Doshi (16)

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.

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.



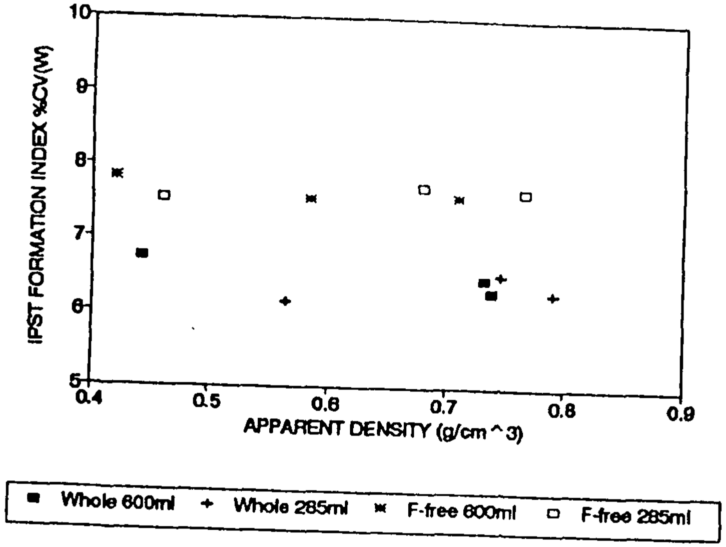


Fig 7 Effect of refining and fines removal on formation index %CV(W) (based on mass density measurements) variation with apparent sheet density

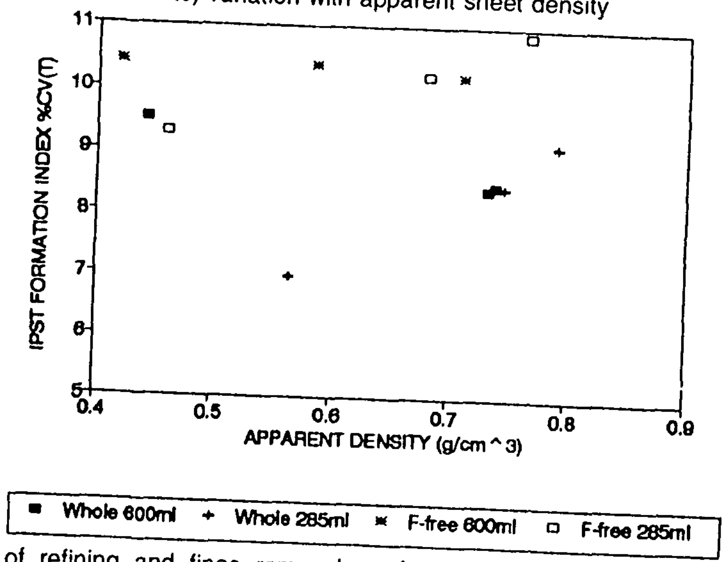


Fig 8 Effect of refining and fines removal on formation index %CV(T) (based on transmitted light measurements) variation with apparent sheet density

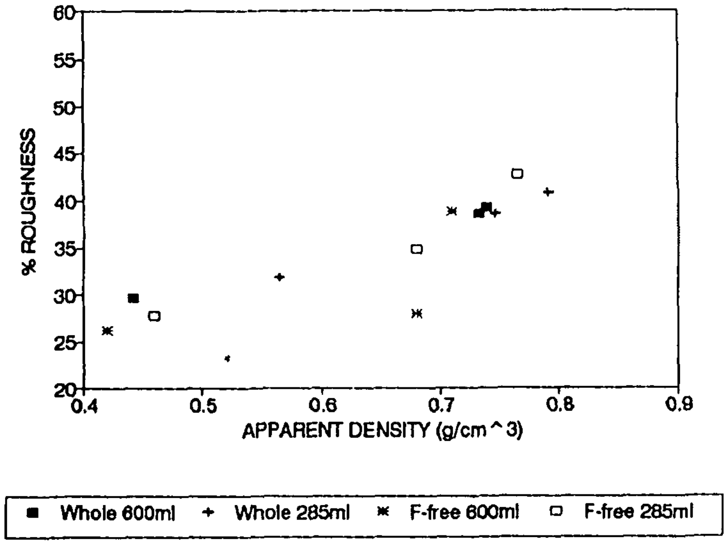


Fig 9 Variation of sheet roughness with apparent density and the effect of refining and fines removal

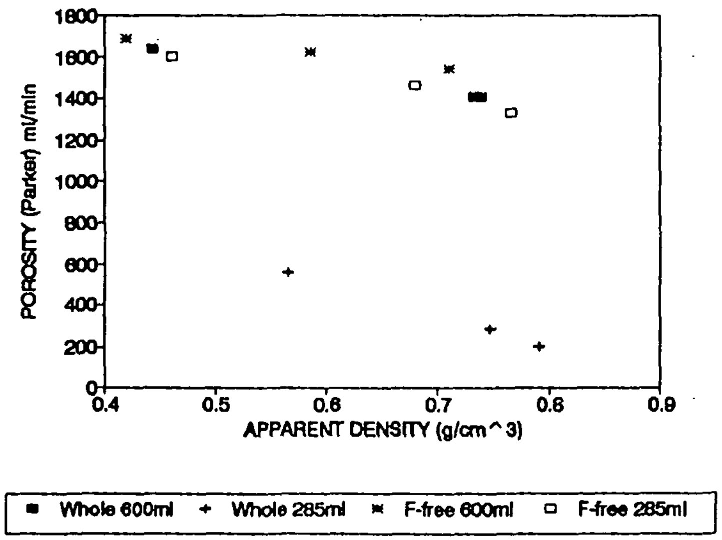


Fig 10 Variation of sheet porosity with apparent density and the effect of refining and fines removal



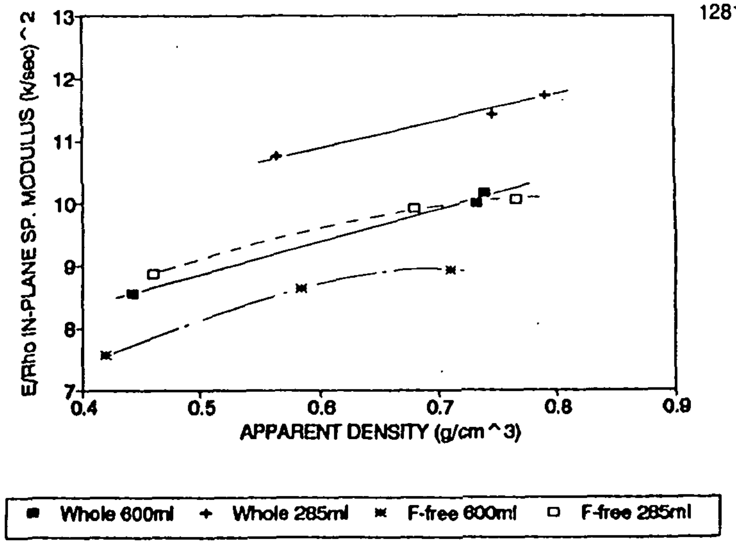


Fig 11 Influence of refining and fines removal on the variation of in-plane specific elastic modulus with apparent sheet density

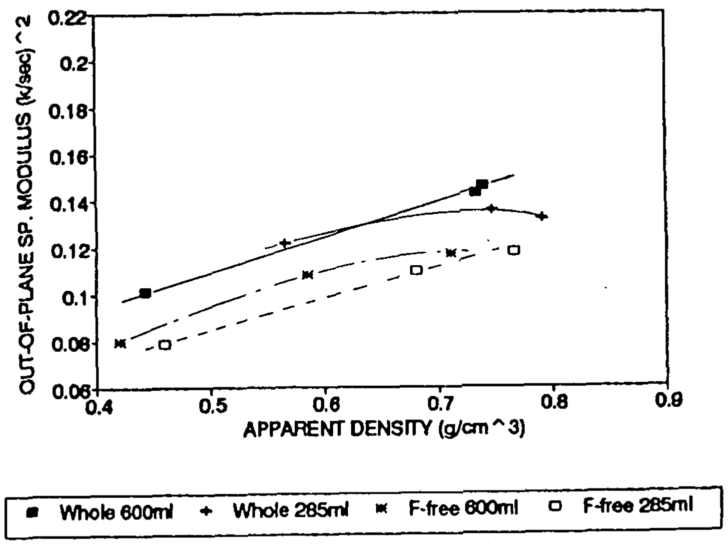


Fig 12 Influence of refining and fines removal on the variation of out-of-plane specific elastic modulus with apparent sheet density

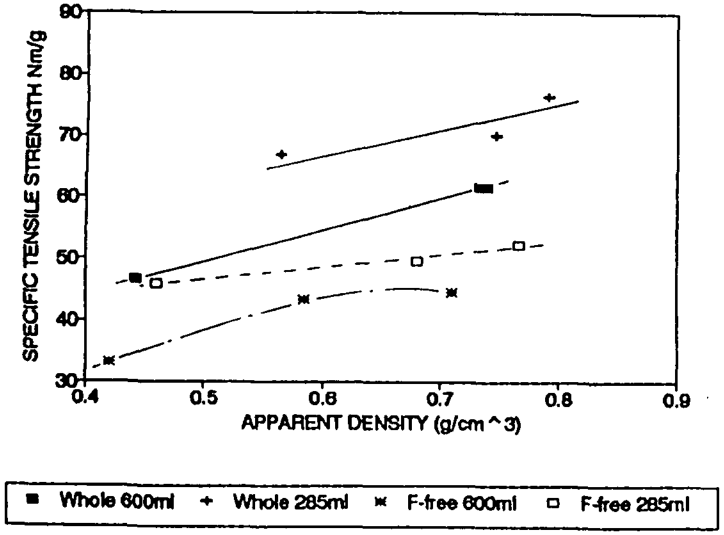


Fig 13 Influence of refining and fines removal on the variation of in-plane specific tensile strength with apparent sheet density

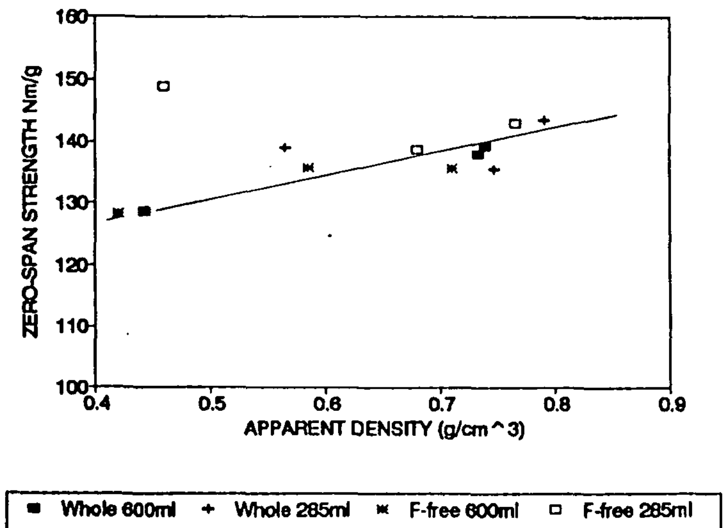


Fig 14 Influence of refining and fines removal on the variation of zerospan strength with apparent sheet density



Fiber strength or the ultimate strength of the sheet (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.

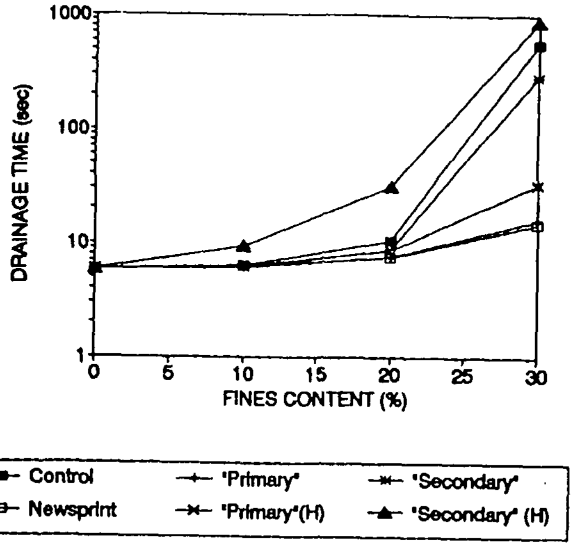


Fig 15 The effect of fines type and content % on sheet mold drainage time

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.



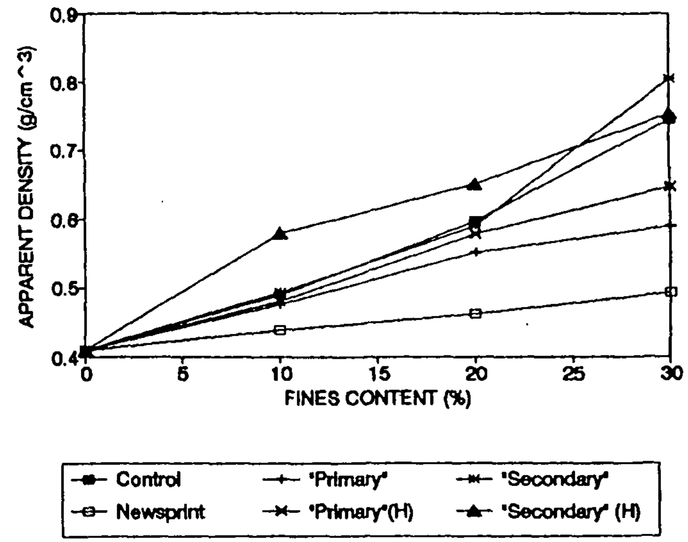


Fig 16 The influence of fines type on the variation of sheet densification with level of % fines addition

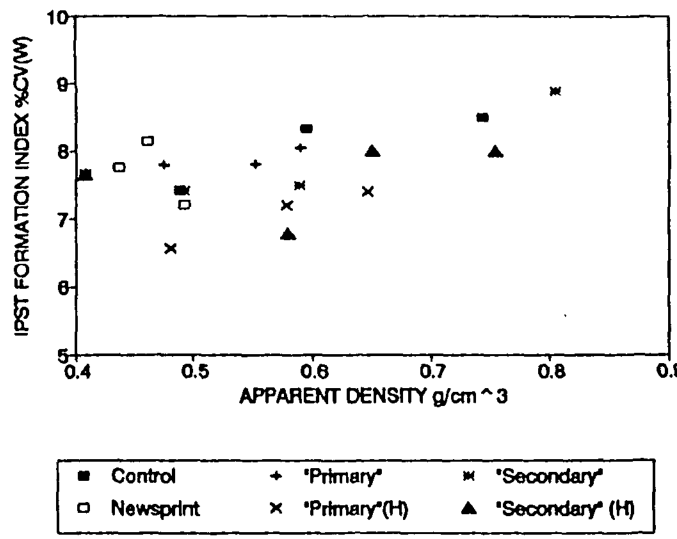


Fig 17 The influence of fines type on the variation of formation index %CV(W) (based on mass density measurements) with apparent sheet density

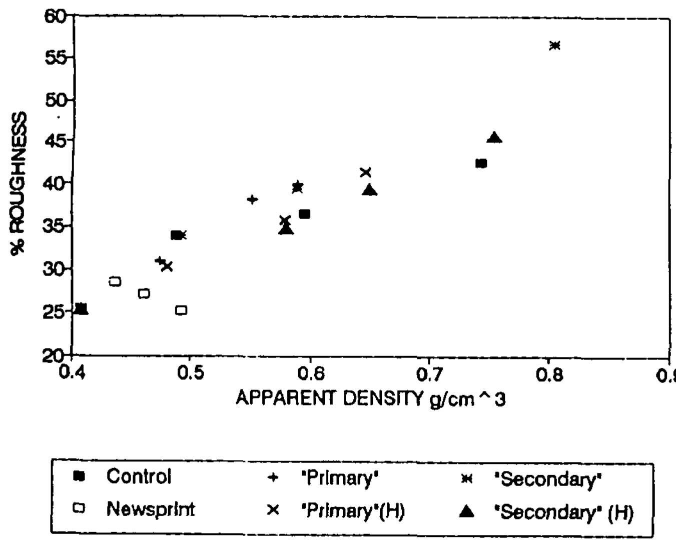


Fig 18 The influence of fines type on the variation of sheet roughness with apparent sheet density

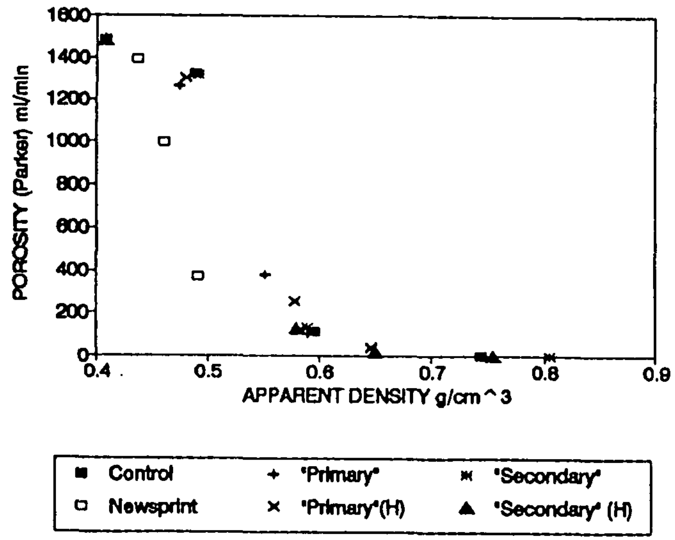


Fig 19 The influence of fines type on the variation of sheet porosity with apparent sheet density



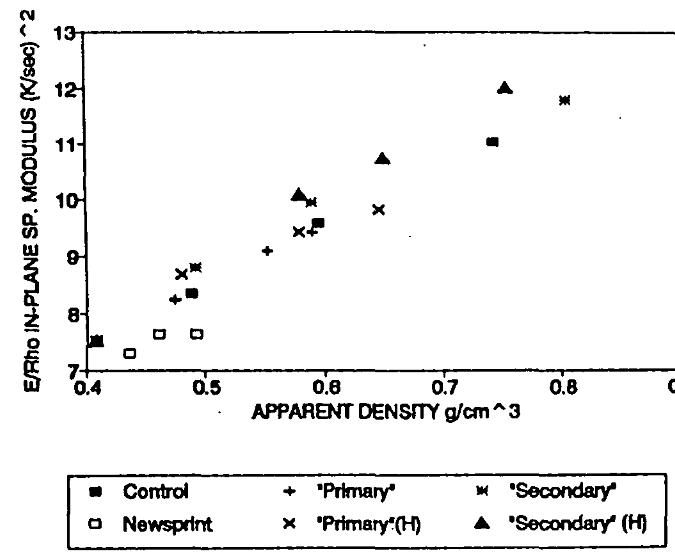


Fig 20 The influence of fines type on the variation of in-plane specific elastic modulus with apparent sheet density

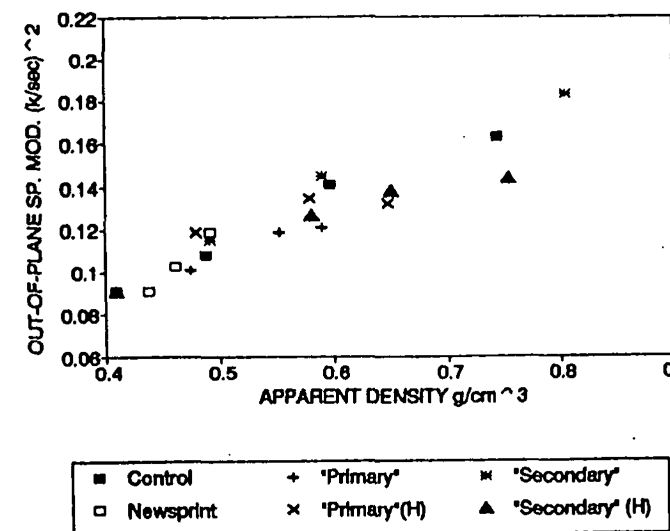


Fig 21 The influence of fines type on the variation of out-of-plane specific elastic modulus with apparent sheet density

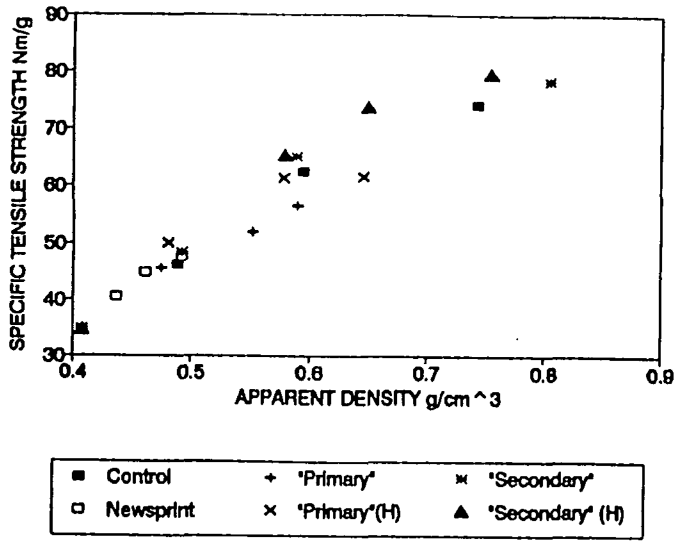


Fig 22 The influence of fines type on the variation of in-plane specific tensile strength with apparent sheet density

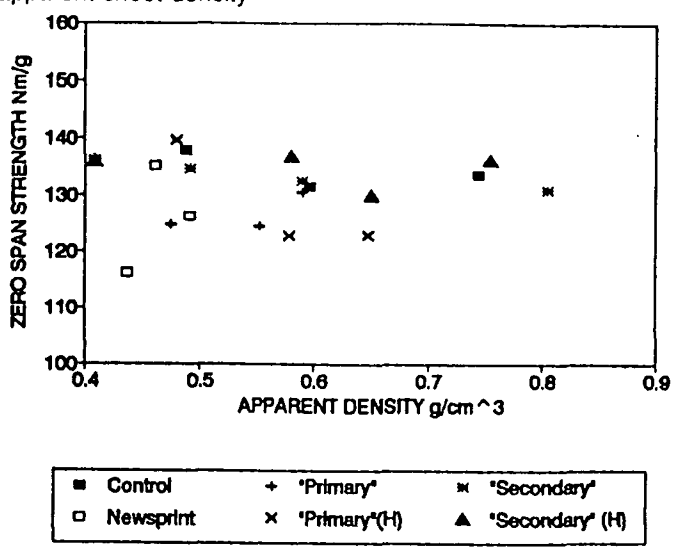


Fig 23 The influence of fines type on the variation of zero-span tensile strength with apparent sheet density



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 (16).


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**The effect of coating components and fillers in the recycling of paper**

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