Characterization of Deinked Pulp for Newsprint

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ABSTRACT
Mill-made deinked pulp fibres for newsprint were compared with virgin single pulps in terms of pulp properties. The results showed that fundamental fibre properties, which had been proposed by many researchers, were useful for the characterization of deinked pulp, provided their fractions were compared. Possible reason for this is that fractionation sorts the fibres, and in this way helps to characterize the complicated fibre composition of deinked pulp. Notable characteristics of long fibres of deinked pulp seemed to be fibre coarseness, wet fibre flexibility and curl or kink as well as fibre swelling. These results suggested that the characterization of mill-made deinked pulps with these fibre properties should be carried out by taking the fibre types into account. For this reason, a new method for the determination of fibre composition has been proposed. It provides good results within a certain range of pulps, but further studies are required to devise a more sophisticated method.
INTRODUCTION
Technologies for the recycling of paper have been developed from ecological and economical viewpoints. Optical properties, such as brightness, colour and dirt, are most important for printing papers as a visual information medium. Therefore, most research and development efforts for the recycle of printing papers have been focused on the deinking process. They have led to be successful in advancing the deinking technologies. It is true that these optical properties of pulp for printing papers were important but there are still many other qualities to be controlled. For instance, a high quality newsprint is required good runnability as well as print quality. The recent runnability problems include not only web break but wrinkle, flattering and snaking. To solve these problems, the mechanical or physical properties of the newsprint should be controlled. Therefore, the qualities required for deinked pulp containing newsprint and printing paper are not only the optical properties. Many researchers have studied on recycling. Much work on the literature survey has been made by Howard(1). He reviewed the effect of recycling on pulp and paper quality, and found experimentally that different pulp types showed different recycling effects(2). As has been mentioned in his review, the effects of the recycling process, particularly deinking, has, however, not been studied systematically. Although some effects of deinking process have been examined by chemical and machinery manufacturers, much work remains to be carried out in fundamental aspects. From the practical point of view, there is another interest; a simple comparison of deinked pulp with other pulps in a mill. To produce a deinked pulp containing newsprint, mechanical pulp and/or chemical pulp is replaced with deinked pulp. This implies that the deinked pulp is regarded as a single pulp in the mill. In fact, the brightness and freeness of deinked pulp in a line usually seem not to fluctuate because it is easy for the mill to control them. There must be, however, variations of the nature of old newspaper (ONP) as a raw material and contamination of advertisement insert due to the economic conditions and the recycling systems. These variations lead likely to a variation of pulp qualities of deinked pulp, while no brightness change. Figure 1 shows daily changes in sheet density and brightness of handsheets made from deinked pulp produced at a mill in Japan. The brightness seemed to be constant but the density was fluctuating. To produce newsprint with the same deinked pulp content during this period, the mill was unable to keep a certain diameter of the roll without adjustment of calender nip pressures. Supposing the mill uses only a kraft pulp, some qualities of paper could be controlled by cooking and refining conditions on the basis of the well-known relationship between a paper quality and pulp properties. A considerable amount of work has been carried out to characterize recycled fibres. (3) In most cases, the "recycling" has been based on cycles of sheet-making and redisintegration with some treatments. The recycled
fibres have been characterized by the relationships between mechanical properties of the sheet made from those fibres and fibre properties such as fibre swelling, fibre length, curl, and fibre morphology. Few reports, however, have been published on characterization of deinked pulp for printing paper, even on a simple comparison of deinked pulp with single pulps. Many researchers have emphasized the significance of fibre length on the recycling. However, it is also seen from Fig. 1 shows that the average fibre length and freeness were not changing, although density was changing. Figure 2 shows the fibre length distribution of the deinked pulp, which implies that the deinked pulp was a mixture of different pulps. This finding suggests that the average fibre length of whole pulp has little practical significance for the characterization of deinked pulp.

As has been mentioned by Howard, much work on the effect of the deinking process on pulp fibres still remains; effect of chemical additives such as surfactants and alkali, mechanical treatment such as kneading, material loss due to screening or filtration, and particularly the quality of waste paper as a raw material.

![Fig. 1 Changes in average fibre length and freeness(C.S.F) of a deinked pulp for newsprint furnish obtained from a mill in Japan, and density and brightness of the handsheet made from the pulp.](image-url)
To carry out this work, it is necessary to identify the fundamental properties to characterize the deinked pulp. This study aims at revealing the properties for characterization of deinked pulp for newsprint furnish, which were performed by a comparison of mill-made deinked pulps with single pulps in the mill.

RESULTS AND DISCUSSION

FRACTIONATION AND AVERAGE FIBRE LENGTH

It is generally agreed that fibre length is a fundamentally important characteristic of pulp. Fractionation with a standard screen classifier is one of the conventional methods to evaluate the fibre length. Many papers have described the effect of recycling with the aid of a screen classifier(4) and the usefulness of fractionation in the process.(5,6) Figures 3 and 4 show the results of Bauer-McNett fractionation and the average fibre length (length weighted) of whole pulps and fractions of two mill-made deinked pulps, respectively. DIP-A was made at a newsprint mill in Japan. DIP-B was also produced at a newsprint mill but in the USA. Both deinked pulps were one of the furnishes for newsprint at the mills, obtained over a two hour sampling period. As is well-known, the recovery rate of waste paper in Japan is relatively high, and particularly that of ONP is over 95%.

Fig. 2 Fibre length distribution (length weighted) of a deinked pup (DIP-A).
Fig. 3  Comparison of Bauer-McNett fractions between single pulps and deinked pulps.

Fig. 4  Comparison of length weighted average fibre length of fractions between single pulps and deinked pulps.
Deinked pulp for newsprint is produced from the ONP which itself contains over 40% deinked fibres, and advertisement inserts. These advertisements are usually printed on uncoated wood free paper or LWC paper which contains from 50% to 100% hardwood KP in Japan. On the contrary, newsprint in the USA is usually produced from softwood mechanical and kraft pulps, and the quality of the deinked pulp must be quite different from that of Japan. Notwithstanding the difference between these pulps, the Bauer McNett fractions and the average fibre length of DIP-A seem to be much the same as those of DIP-B.

FIBRE ANALYSIS
A new method for the determination of fibre composition has been devised. This method is based on the standard fibre analyses such as Tappi T-401, but more accurate and objective than them because of the use of a computer and an image analyzer (see Appendix). Fibre compositions of DIP-A and B are shown in Fig. 5, which were obtained by this method. The DIP-A produced in Japan contains about 50% KP including hardwood KP. It is generally agreed that the deinked pulp for newsprint in Japan contains a high percentage of KP in comparison with the one in North America and Europe. The DIP-B made at a mill in the USA was composed of 20% softwood KP and 80% softwood MP. The MP in both deinked pulps contains TMP, CTMP, GWP and their fragmented fibres. Fibre analysis for each fraction gives more valuable information; the fraction of R 24 mesh of DIP-A contains no hardwood KP, but only softwood KP and MP. This MP was presumably identified as TMP by the morphological characteristics. Other fractions of DIP-A had high hardwood KP contents. It was found that the fragmented MP fibres or GWP increased in these fractions. The R 24 mesh fraction of DIP-B was also composed of softwood KP and TMP (or CTMP), and softwood KP content decreased with increasing mesh. It should be noted that a small amount of hardwood KP was observed in the R 42, 80 and 150 mesh fractions of DIP-B.

FIBRE COARSENESS
The difference between the two deinked pulps was not represented by the average fibre lengths of the whole pulps. The average fibre length of each fraction only showed that the Bauer McNett classified fibres according to their length. It is known that the fibre coarseness measurement with an optical fibre analyzer is not satisfactory in its accuracy. From a practical viewpoint, however, this device is still useful for the characterization of pulp, even though it gives only relative values.
Fig. 5 Fibre composition of DIP-A produced at a newsprint mill in Japan and DIP-B obtained from a newsprint mill in the USA.
Figure 6 shows the comparison of fibre coarseness for the whole pulps and fractions. The R 24 mesh fractions of both deinked pulps have lower fibre coarseness than those of virgin softwood KP or TMP. Provided the deinked pulps have the fibre composition as shown in Fig. 5, the coarseness of other fractions can be estimated from coarseness values of virgin pulps. The coarseness of these fractions of DIP-A are lower than those of DIP-B. Possible reason for this is a contamination of hardwood KP in DIP-A.

![Graph showing fibre coarseness comparison](image)

Fig. 6 Comparison of fibre coarseness of fractions between single pulps and deinked pulps.

**WATER RETENTION VALUE**

Though DIP-A and B have been beaten with a refiner to adjust to a certain level of freeness, it is seen from Fig. 7 that their Water Retention Value (WRV) is lower than those of the most of the fractions for virgin TMP and KP. It is generally agreed that the recycling of chemical pulps leads to stiffening of the fibre due to the reduced swelling capacity of the fibre wall. In view of this, it seems likely that deinked pulp has lower fibre swelling than virgin pulps, presumably because of a recycling effect.
WET FIBRE FLEXIBILITY

The results of WRV suggest that deinked pulp fibres may be stiffer than virgin pulp fibres. To evaluate flexural rigidity of both pulp fibres in wet state, the wet fibre flexibility (WFF) of long fibre fractions of deinked pulp and virgin pulps were measured according to Steadman and Luner's method. The original method was modified using a specific staining technique so that an image analyzer can identify KP and MP fibres in a deinked pulp separately. The results of WFF defined as the reciprocal of flexural rigidity or its median and WFF index defined as the natural logarithm of WFF are listed in Table 1. Since it was hard to distinguish long fibres of hardwood KP from latewood fibres of softwood KP, the results of KP fibres in R42 mesh fraction of deinked pulps in this table include those of both fibres. The frequency distributions of WFF index for kraft fibres of R 42 mesh fraction are shown in Fig. 8. A significant difference in distribution was observed between kraft pulp fibers in a deinked pulp and virgin kraft pulp fibres. A comparison of WFF of R 42 mesh fraction (Table 1) also shows that KP fibres in both deinked pulps have lower flexibility than virgin pulp fibres. This difference in WFF may be a result of difference in fibre swelling. However, it can be also seen that TMP fibres of R 24 mesh fraction of both deinked pulps have greater flexibility than those of virgin pulps. Softwood KP fibres of R 24 mesh fraction in both deinked pulps have slightly greater WFF than those of virgin pulps, while WRV of the deinked pulps was lower than that of virgin pulps. The fibre coarseness of long fibre
fraction of deinked pulp was relatively lower than the coarseness estimated from virgin TMP and softwood KP. The coarseness of a fibre is proportional to the net cross-sectional area, provided there are no significant differences in fibre wall density. In view of this, the long fibres in deinked pulp may have less cross-sectional area than virgin fibres. In addition to this, Howard pointed out that recycling made mechanical pulp fibres flatter, giving reduction of moment of inertia of area. Therefore, possible reasons for the difference in WFF between both long fibres in R 24 mesh fraction are thinning of the fibres and flattening of the mechanical pulp fibres during the recycling process.

Fig. 8 Frequency distribution of Wet Fibre Flexibility (WFF index) for virgin softwood KP fibers, virgin hardwood KP fibres and KP fibres in DIP-A (R 42' mesh fraction)
Table 1 Comparison of Wet Fibre Flexibility (WFF)

<table>
<thead>
<tr>
<th>Fibre type</th>
<th>Fraction</th>
<th>WFF $10^9$ N$^{-1}$m$^2$</th>
<th>WFF Index ln(WFF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin softwood KP</td>
<td>R 24 mesh</td>
<td>140</td>
<td>25.7</td>
</tr>
<tr>
<td></td>
<td>R 42 mesh</td>
<td>550</td>
<td>27.0</td>
</tr>
<tr>
<td></td>
<td>R 80 mesh</td>
<td>460</td>
<td>26.9</td>
</tr>
<tr>
<td>Virgin hardwood KP</td>
<td>R 42 mesh</td>
<td>530</td>
<td>27.0</td>
</tr>
<tr>
<td></td>
<td>R 80 mesh</td>
<td>480</td>
<td>26.9</td>
</tr>
<tr>
<td>Virgin TMP</td>
<td>R 24 mesh</td>
<td>7.3</td>
<td>22.7</td>
</tr>
<tr>
<td></td>
<td>R 42 mesh</td>
<td>18</td>
<td>23.6</td>
</tr>
<tr>
<td></td>
<td>R 80 mesh</td>
<td>64</td>
<td>24.9</td>
</tr>
<tr>
<td>Virgin GWP</td>
<td>R 42 mesh</td>
<td>126</td>
<td>25.6</td>
</tr>
<tr>
<td></td>
<td>R 80 mesh</td>
<td>290</td>
<td>26.4</td>
</tr>
<tr>
<td>Softwood KP in DIP-A</td>
<td>R 24 mesh</td>
<td>179</td>
<td>25.9</td>
</tr>
<tr>
<td>KP* in DIP-A</td>
<td>R 42 mesh</td>
<td>215</td>
<td>26.1</td>
</tr>
<tr>
<td>MP (TMP) in DIP-A</td>
<td>R 24 mesh</td>
<td>23</td>
<td>23.9</td>
</tr>
<tr>
<td>MP in DIP-A</td>
<td>R 42 mesh</td>
<td>66</td>
<td>24.9</td>
</tr>
<tr>
<td>Softwood KP in DIP-B</td>
<td>R 24 mesh</td>
<td>172</td>
<td>25.9</td>
</tr>
<tr>
<td>KP* in DIP-B</td>
<td>R 42 mesh</td>
<td>208</td>
<td>26.1</td>
</tr>
<tr>
<td>MP (TMP) in DIP-B</td>
<td>R 24 mesh</td>
<td>31</td>
<td>24.2</td>
</tr>
<tr>
<td>MP in DIP-B</td>
<td>R 42 mesh</td>
<td>53</td>
<td>24.7</td>
</tr>
</tbody>
</table>

*) including hardwood and softwood fibres

SHEET DENSITY
Sheet density is a fundamental property of paper that has been shown to be related not only to other physical properties but also to papermaking process variables. The relationship between apparent sheet density and grammage for whole pulps is given in Fig.9. Although fibre coarseness and WRV of deinked pulp are different from those of virgin single pulps, it seems from Fig. 9 that the fibre composition for the deinked pulps is apparently represented by the sheet density when compared at the same grammage; actual density
of DIP-A is 520 kg/m³ at 60 g/m² and the estimated density from density of virgin pulps on the basis of fibre analysis results (29% softwood KP, 20% hardwood KP and 51% MP) is 518 kg/m³, actual density of DIP-B is 480 kg/m³ and the estimated density is 472 kg/m³ (20% softwood KP and 80% MP), provided the density of MP is an average of the densities of TMP and GWP. On the other hand, it is seen in Table 2 that the estimated values from densities of the fractions of virgin pulps do not always correspond to the results of deinked pulps. Although long fibres in R 24 fraction of both deinked pulps have greater flexibility than those of virgin single pulps, their sheet densities are greater than the calculated value from the data of virgin TMP and softwood KP (values in parentheses in Table 2) or the density of handsheet made from a blend of those pulps with the same fibre composition.

Fig. 9 Apparent sheet density vs. Grammage for single pulps and deinked pulps.
VIRGIN, RECYCLED AND DEINKED PULP

Generally, deinked pulp is considered to be a recycled fibre. This report has shown differences in some fundamental fibre properties between deinked pulps and never-dried virgin pulps which were used as the furnish for newsprint at the mills. Using a simple laboratory procedure, *recycled* fibres were compared with never-dried virgin fibres and deinked pulp fibres. To avoid an influence of the loss of fines during sheetmaking, and to simplify the experiment, the R 24 mesh fractions of pulps were used. Handsheets were made from never-dried virgin softwood KP, TMP and a blend of these pulps (75% TMP and 25% softwood KP, corresponding to the fibre composition of R 24 fraction of DIP-B) according to the standard method. Recycled fibres were obtained by soaking a part of these sheets in water and disintegrating them. Subsequently the recycled pulps were used to prepare handsheets once again. The characteristics of these fibres and handsheets are also given in Table 2.

As expected, recycling, even one time, reduced the sheet density of softwood KP, probably because of loss of fibre flexibility caused by the reduction of fibre swelling. The sheet density of TMP slightly increased with recycling, while the WRV was reduced. This finding is consistent with the results observed by Howard and Bichard (2), who found that mechanical pulp fibres became flatter and more flexible giving a denser sheet with recycling. Though DIP-B has greater WRV than recycled pulps, the long fibre fraction of DIP-B has lower density than that of virgin or recycled pulp. As mentioned before, this finding is also observed with DIP-A, when compared with the estimated densities from the values of single pulps; assuming its fibre composition to be 55% TMP and 45% softwood KP, the estimated density from virgin pulps is 341 kg/m\(^3\) and 328 kg/m\(^3\) from recycled pulp. The difference in sheet density between deinked pulps and virgin pulps may not be attributed to the differences in wood species or pulp type. Curl index defined by Jordan and Page (9) and kink index defined by Kibblewhite (10), of these fibres are also given in the table. These results indicate that recycling removes the curl. It is also seen that softwood KP and TMP fibres in deinked pulp are more curly than recycled pulp fibre and have many kinks. These kinks and curl of the deinked pulp fibre may be caused by recycling or deinking process such as kneading. Page et. al have investigated effects of curl on sheet properties. Kibblewhite has also emphasized abrupt kink rather than gradual curl. It is seen from their papers that, in some cases, curl and kink of fibres reduce the sheet density unless the fibres are affected by other concomitant action. For this reason, the lower sheet density of long fibre fraction of the deinked pulps may be attributed partly to their curliness and kinkiness.
Table 2  Characteristics of Long Fibres of Never-dried virgin, Recycled(once time) and Deinked pulps (R 24 mesh fraction)

<table>
<thead>
<tr>
<th></th>
<th>Softwood KP</th>
<th></th>
<th></th>
<th>Blend (25:75)*</th>
<th></th>
<th></th>
<th>DIP-A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Virgin</td>
<td>Recycled</td>
<td>Virgin</td>
<td>Recycled</td>
<td>Virgin</td>
<td>Recycled</td>
<td>DIP-B</td>
</tr>
<tr>
<td>WRV gr²O/100g</td>
<td>160.0</td>
<td>85.9</td>
<td>129.1</td>
<td>95.2</td>
<td>136.8</td>
<td>92.9</td>
<td>108.9</td>
</tr>
<tr>
<td>Curl Index</td>
<td>0.267</td>
<td>0.196</td>
<td>0.029</td>
<td>0.018</td>
<td>-</td>
<td>-</td>
<td>KP 0.208</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>KP 0.257</td>
</tr>
<tr>
<td>Kink Index</td>
<td>2.3</td>
<td>1.9</td>
<td>0.05</td>
<td>0.03</td>
<td>-</td>
<td>-</td>
<td>2.5</td>
</tr>
<tr>
<td>Density kg/m³</td>
<td>470</td>
<td>424</td>
<td>235</td>
<td>249</td>
<td>292</td>
<td>307</td>
<td>283 (294)**</td>
</tr>
</tbody>
</table>

*) Weight ratio of softwood KP and TMP

**) The estimated density from values of never-dried pulps.
FINAL REMARKS

Mill-made deinked pulp fibres for newsprint were compared with virgin single pulps in terms of pulp properties. The results show that fundamental fibre properties which have been proposed by many researchers are useful for characterization of deinked pulp, provided their fractions are compared. Possible reason for this is that fractionation sorts the fibres, in this way helps to characterize the complicated fibre composition of deinked pulp. The finding suggests that the characterization of deinked pulp requires the development of either of the following two methodologies; development of a new fibre analysis technique which is more reliable, objective and rapid; or development of a universal characterization method which is not influenced by fibre composition or any fluctuating process parameters. To establish the universal law of the relationship between fibre characteristics and sheet properties, theoretical studies have been made by Page et al.(11,12) for some mechanical properties and by Dodson(13) for sheet formation, but much work still remains for a practical use. For the former, we have proposed a new method for determination of fibre composition. It provides good results within a certain range of pulps, but further studies are required to devise a more sophisticated method.

Notable characteristics of deinked pulp fibres seem to be fibre coarseness, wet fibre flexibility and curl or kink as well as fibre swelling. Though it is well-known that these fibre properties influence on the sheet properties, characterization of mill-made deinked pulps with these fibre properties should be carried out by taking the fibre types into account.

On the basis of this result, the mill found with a statistical analysis that the observed variation of sheet density as shown in Fig. 1 were partly explained by variations in hardwood content, ash content and curliness of fibres in the deinked pulp used.

The present work dealt only with the long fibre fraction. It is well-known that the fines play an important role in the newsprint quality. The fines in mill-made deinked pulp, however, contain various materials - a rather complex system. In view of this, further works are required to establish the characterization of this fraction of deinked pulp.

EXPERIMENTAL

ORIGIN OF PULP USED

The deinked pulp identified as DIP-A, semi-bleached softwood kraft pulp identified as softwood KP or SKP, semi-bleached hardwood kraft pulp identified as hardwood KP or HKP, thermomechanical pulp(TMP), groundwood pulp(GWP) were obtained from the same
newsprint mill in Japan. For comparison, the deinked pulp identified as DIP-B was obtained from a newsprint mill in the USA, which was also producing a thermomechanical pulp, and exporting chips for TMP to the Japanese mill. The TMP was the third refiner discharge pulp using the chips having a high spruce and hemlock content. The softwood KP was produced from domestic wood chips of spruce, larch and Douglas fir, and bleached with an oxygen alkali extraction. The hardwood KP was made from imported chips of eucalyptus, domestic birch and beech, and bleached in the same way as the softwood KP. The GWP was a pit sample. DIP-A was produced with a similar deinking process system to DIP-B, which consists of high consistency pulping with caustic soda, coarse screening, soaking in deinking chemicals, flotation, kneading (dispersing), cleaning, washing, bleaching with peroxide and finally post-refining. As already mentioned, the waste paper furnish of DIP-A was different in the kraft pulp content from that of DIP-B. Hardwood kraft pulp fibres in DIP-A were identified by morphological characteristics and results of a sugar analysis as similar wood species to the virgin hardwood KP fibres. Wood species of long fibres of mechanical pulps in DIP-A and B were also similar to those of thermomechanical pulps produced at both mills.

FLEXIBILITY, CURL AND KINK OF SINGLE PULP FIBRES
Wet Fibre Flexibility (WFF) was determined by the procedures developed by Steadman and Luner(8), using a Zeiss IBAS image analyzer. To measure the WFF of KP and TMP fibres in a deinked pulp separately, the original WFF method was modified using a specific staining technique so that an image analyzer can identify those fibres. Curl and Kink were also determined with the image analyzer.

FIBRE LENGTH AND COARSENESS
Length weighted fibre length and fibre coarseness were measured using a Kajaani FS-200 Fibre Length Analyzer.
APPENDIX
FIBRE ANALYSIS
The standard fibre analysis method such as TAPPI T401 requires the analyst be skillful and experienced in the field of pulp and paper microscopy. No matter how skillful the analyst is, the measurement is still lacking in an objectivity. A new method for determining fibre composition of a pulp has been devised. This method is partly based on the standards but uses an image analysis and fibre length distribution data so that more accurate and objective results can be obtained.

FIBRE LENGTH DISTRIBUTION
Using fibre length measurements, several methods for fibre analysis have been proposed. (14,15) This study deals with a simple application of the fibre length distribution to the fibre analysis.

To determine the weight ratio of fibre composition in a blend of different pulps, the fibre length distribution of a pulp was assumed to be a result of the superimposition of distributions of known pulps. If the data sets of the number of fibres $y_i$ for a pulp and $x_{ij}$ for reference pulps ($i=1,...,m$, $m$: number of reference pulps) in different length fractions ($i=1,...,n$, in the case of Kajaani FS-200, n=144) are obtained, the data set of length weighted fibre length for those pulps $Y_i$ and $X_{ij}$ are given by,

$$Y_i = \frac{\sum L_i^c y_i}{\sum L_i^c}$$

$$X_{ij} = \frac{L_i^c x_{ij}}{\sum L_i^c x_{ij}}$$

where

$L_i$ is the average length of the fraction $i$

$C$ is a weighting factor that should be empirically determined for each measurement method; the factors we have obtained are $1 - 1.5$ for the Kajaani FS-200 fibre analyzer and $0 - 1$ for a projector or an image analyzer.

Assuming that $Y_i$ is expressed as the following function of $X_{ij}$ for different reference pulps and the error $e_i$,

$$Y_i = \sum_{j=1}^{m} a_{ij} X_{ij} + e_i$$

we can estimate the weight ratios $A_j$ for each pulp in an unknown sample from $a_{ij}$ which are obtained with multiple regression analysis to this equation and the weight factors $w_j$ for each reference pulp which are available in the literatures, it follows that:
COMBINATION WITH IMAGE ANALYSIS DATA

As is well-known, the weight ratio based only on fibre length data is not satisfactory. To obtain the required accuracy for characterization of DIP, we have combined fibre length data with image analysis results.

Slides for microscopy are prepared with various blends of reference pulps with known weight ratios according to the standard method. Subsequently, these fibres on the slide are stained by a usual staining method for fibre analysis such as the Graff *C* stain. The stained slide is placed at an automated stage of a Zeiss Axioplan type microscope and viewed with a precision RGB TV camera for image analysis manufactured by Hamamatsu TV or Sony. In the case of the *C* staining, mechanical pulps give yellow or orange, bleached softwood KP stains bluish gray, and bleached or unbleached hardwood KP give blue or greenish blue. Therefore these fibres on the image can be separated into three images by the colour tone, gray level and contrast. After the separated fibres on each image are transformed into a binary image, the projection area of the fibres is determined with an IBAS system manufactured by Zeiss. A calibration curve for the determination of fibre composition in an unknown pulp is obtained from the relationship between the projection area and weight ratio of fibres in the blends of reference pulps. It has been found that this image analysis method is excellent in the determination of hardwood KP and MP contents in any blend of pulps. It is difficult, however, to separate TMP from a blend of mechanical pulps only with the image analysis. On the other hand, the fibre length distribution of GWP is quite different from that of TMP. The fibre length distribution of GWP is usually located to a short fibre range, while TMP and softwood KP have wide fibre length distributions. Thus, the weight ratio of these different fibres in a blend can be determined accurately using the both results of the image analysis and the fibre length measurement. A comparison of the results obtained by this combined method and by the standard method is given in Table A. The results show that this method is more accurate than the standard microscopic technique.
### Table A. Comparison of the results obtained by this combined method and by the standard method.

<table>
<thead>
<tr>
<th>Blend</th>
<th>Hardwood KP</th>
<th>Softwood KP</th>
<th>TMP</th>
<th>GWP</th>
<th>(MP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>60</td>
<td>0</td>
<td>12</td>
<td>28</td>
<td>(40)</td>
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<tr>
<td></td>
<td>65</td>
<td>0</td>
<td>-</td>
<td>-</td>
<td>(35)</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0</td>
<td>13</td>
<td>27</td>
<td>(40)</td>
</tr>
<tr>
<td>B</td>
<td>20</td>
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### References


Dr J D Peel, (formerly Kusters, Germany)
Could you please make a comment on the difference between the papermaking properties of the two pulps you are examining, the Japanese deinked pulp and the American. What are the main differences between the papers made from DIPA and DIPB?

T Naito
Actually DIPB was produced by one of our related companies in the USA and they have to export to Japan and they have so many competitors in Japan so apparently they had to produce similar properties of newsprint in Japan. The furnish they used was very different from the Japanese one so we have to identify what different furnish affects the end use properties exactly.

J Peel
I think what you are saying is that the properties of the papers are very similar.

T Naito
Yes.

G Jones, Bridgewater Paper Co Limited, UK
How important would you regard filler content for characterisation of deinked pulp?
T Naito
Do you mean the ash content of pulps? Ash and fines are other important issues so our study avoided the effect using only the long fibre fractions.

Prof P Stenius, Helsinki University of Technology, Finland
Being a surface chemist I cannot refrain from asking you if you were talking about the need for better ways of characterising the fibres. Have you tried to look at the surface properties of the fibres and do you think that you would get relevant information that way?

T Naito
We have done some work on properties but that is very difficult for us. Generally speaking, it is difficult to evaluate the surface properties of deinked pulp because of the lack of information on the effects of the deinking process on pulp fibres. It raises different issues.

Prof D Wahren, Stora Teknik, Sweden
There are two concepts which we will have to live with in the paper industry, namely, Recycling and Quality. Both are necessary – at the same time and in the same product. Quality is necessary. This is so obvious so why say it? I say it because I would like to see some redirection of research in the recycling area.

There are mills which run furnishes consisting of 100% recycled fibres, but considering even the simplest recycling system there is an input of virgin fibres because collection systems cannot be perfect, and some material is lost in sorting and in the cleaning processes in the mill. These losses, however, do not consist of prime fibrous material. What is lost at the mill is primarily fibre
fragments, fines and fillers. The more worn the fibres are the more fibre fragments will have to be lost in order to keep the quality up to standard.

So, I do not believe we need more studies on how many times a fibre can be rotated through the system and how much the properties will deteriorate. The properties will not be allowed to deteriorate. End product quality is the driving factor; fractionated losses are a consequence. I would like to see studies resulting in indications of how much of what fractions need to be lost in order to keep the end product quality on an acceptable level. This is the kind of information on which LCAs and recycled fibre flows can be calculated.

**Dr A H Nissan, Westvaco Corporation, USA**

Yesterday Derek Page made two brilliant speeches. One was here and one was at the banquet. I would like to refer to his talk here and make an added comment. He pointed out, you remember, that when cellulose is deuterated you get at first a step function with an asymptote around 50% or thereabouts and then if you wet and dry and deuterate and so on you have further steps and you go to 100% substitution. He pointed out that this gave a clue to another phenomenon which is bothering us and that is the mechano sorptive effect in that you open up the crystalite every time you deuterate cellulose. He suggested that this takes place stepwise, axially and that this explained the extra creep when RH was oscillated between high and low values. I thought it was typical, divergent thinking that students of creativity tell us characterise the creative thought and I was really taken with the idea. Then I had a problem. Two days before, there was another mechanism proposed. Again hypothesis, again speculative thinking, on the mechano-sorptive effect, namely
that it could be that the micro-corrugation would be opened up by successive steps under shear. It was Derek who came out with the brilliant criticism that in fact this cannot be the total explanation because it applied solely to paper whereas the mechano-sorptive phenomena are much more general and they apply even to non-heterogeneous systems. At that time I thought that was brilliant and I said so but it began to bother me here. You see Page's new hypothesis is also specific to paper because it's peculiar to cellulose. Therefore if we could not accept immediately the mechanism of the micro-corrugation because it was specific whereas the phenomenon is more generic, similar questions arise in one's mind about this one. Then I thought how can we explain the complete deuteration of cellulose by another mechanism. I was reminded of a book written at about the same time as Corte and Schaschek were doing these deuteration studies. The book was by Prof Meyer. In his book published in 1953 by Interscience series on polymers Meyer pointed out that the crystalite of cellulose is very small. Later it was shown that it was 35Å wide. Still later work by a PhD student in Syracuse showed that there could be a smaller dimension of 18Å but that has never been confirmed by others. If you take 35Å as the side of a square, that means that you have only 7 molecules of cellulose a side which means that we have 49 molecules per crystallite of which 7 + 6 + 6 + 5 or 24 molecules are on the surface. They are so called accessible. That's how we measure the amorphous region. We measure it by the accessibility ie by the rate of hydration, deuteration, or whatever. This means that when you tell me that cellulose is 50% amorphous I don't think of the amorphous region as a higgledy-piggledy spaghetti dish. I think of the 50% of molecules on the surfaces of the crystallites. But of course there are further amorphous regions; they are hemi-cellulose moities in fibre. That's why you can have fibres with
60/70% amorphous regions. But look what is happening here, there are only 3 diffusion steps from the surface of the crystallite to the core. The surface molecules are free to rotate in segments, because the kinetic energy of only 4-5 kcals per mole in the air can knock a hydrogen bond out. Therefore you can diffuse deuterium inside by 3 radial steps of diffusion and you don't have to invoke axial flow by Page's mechanism. Therefore this is an alternative explanation of 100% deuteration. The difference is that this explanation doesn't give us any explanation of mechano-sorptive excess creep. This leads me to the purpose of my talk now. When a theory like Page's is proposed by a highly respected and famous person it tends to be quoted and then repetition establishes it as being fact. We don't want to see that. What we need is a crucial experiment to see whether stepwise deuteration occurs axially as Page has suggested or by other ways as I have just speculated. Actually, I don't believe either is happening. The reason I don't believe that either is happening was given by Dr Scallan this morning, p1236, in which he gave the deuteration curve. When you deuterate cellulose, it shows 4 whole peaks. I am surprised there are only 4 because there should really be more types of hydroxyl groups joining hydroxyl groups in cellulose. This means both the structures of the H-bonds and the deuteration mechanism is more complex than we think. So it is not a simple mechanism and we have to understand that before we can decide. What I am saying is that yesterday Derek Page talked about the science in my generation and his and he praised it as having a human face but it did have this one weakness. Whenever we came across a difficult problem we put a hypothesis and it got repeated and repeated and nobody did the critical experiment and you know what we had? We had a particular feature of a human face. These conferences used to have major controversies and the controversies disappeared in
time because people got tired of them but we never solved the problem exactly. I call them pseudo controversies. We had no controversies in this Symposium. But it is not controversies that we need. We need crucial experiments to resolve ambiguities. What I am saying is please could you possibly conceive of a little experiment - (a) to tell us how, exactly, we get 100% deuteration and (b) why the mechano-sorptive phenomenon appears. Critical experiments, not speculation alone. We have had another area where we have talked about it for 20 years - glass or secondary transition temperatures in paper. For 20 years they have been talking about Tg and how water brings it down, I accept it; everyone accepts it, but Dr Padanyi told us yesterday that he finds he can't measure it. He said "I could not find it and when I asked authorities about it they said that it was difficult to measure". But if we cannot see it where is it? So what I am begging is that someone will take paper, determine Tg accurately, then change the humidity and measure it again and give us what is an observed fact. This also applies to other areas.

I am as guilty as others in that when I come to something I do not understand I immediately put a hypothesis to it. I must warn you that the most dangerous hypothesis is the most believable, because if you did not believe it you would check it. Please think of crucial experiments, experiments that say "yea or nay" because science has only advanced because of crucial experiments. There is no alternative and there are several problems needing this approach. Even if you cannot think how to achieve the crucial experiment it is worth writing a letter to the editor asking for one, whenever there is an ambiguity in the field because some young PhD student might think of a way of doing it. Speculative hypotheses may be dangerous to our science. Thank you very much.