Application of fibre characterization techniques to industrial papermaking operations

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SUMMARY

The applications of fibre measurement and characterisation procedures to industrial pulping and papermaking operations are discussed. Two commercial paper products were examined in the attempt to identify factors which could influence:

- Unexplained changes in the opacity of white offset printing papers;
- The presence of darkly coloured 'bruised' areas (< 3 cm²) in calendered kraft liner paper.

Samples of white offset printing papers (70 g/m²) with 'good' and 'poor' opacity were examined to determine whether the changes in opacity could be related to differences in fibre characteristics, fibre proportions, or filler content and distribution. Techniques of quantitative microscopy showed that the papers with 'poor' opacity were more consolidated and contained fibres which were more collapsed and bonded to one another than those in the papers with 'good' opacity. This observation indicated that opacity was lowered through a decrease in web light-scattering properties brought about by the increased fibre bonding. Mean fibre lengths and fibre length distributions, and mean fibre diameters and fibre diameter distributions were similar for the papers with 'good' and 'poor' opacity. This showed that the differences in opacity were not related to changes in the quality of the wood source or the proportions of radiata pine (Pinus radiata) or tawa (Beilschmiedia tawa) fibres in the furnish. Clay contents varied in both quantity and quality in the paper samples examined and few conclusions could be drawn concerning their relative effects on paper opacity. Experimental data did, however, suggest that the clay which contained a proportion of large particles was least effective in bringing about an improvement in paper opacity.

Samples of kraft liner paper which contained darkly coloured 'bruised' regions were examined. Microscopic examination showed that the bruised areas contained highly collapsed and bonded fibres, and that fibre surfaces appeared to be covered by an amorphous coating. Sectioned faces of paper sheets confirmed the high degree of bonding because sheet thickness was 15 to 20 per cent lower in the bruised areas. The numbers of fibres contributing to the thickness of the bruised and unbruised regions were similar. The microscopic studies suggested that bruising was related to a concentration in the blemished areas of a contaminant with adhesive-like properties. This was confirmed by chemical analyses.

The measurement of fibre characteristics such as fibre dimensions and the collapse behaviours of fibres in wet pulps and in situ in handsheets has been shown to have definite application for research in the identification of differences between pulps, papers, and fibre populations (1). In the present paper two instances are described in which fibre characterization techniques were applied to commercial operations in the attempt to explain and/or identify differences in industrial paper products produced under nominally the same pulp and papermaking conditions. These two studies were concerned with the identification of factors which influenced:

- The opacity of certain grades of printing papers;
- The development of blemishes or bruised areas in kraft liner paper.

The studies were undertaken in conjunction with the staff of the Kinleith mill of N.Z. Forest Products Limited. Paper samples with similar compositions but with 'good' and 'poor' opacity or with bruised and unbruised areas were collected from the mill and examined at the Forest Research Institute. Preliminary examination showed that visual differences existed between samples and, therefore, detailed quantitative analyses were made in attempts to explain and/or identify factors causing the deleterious effects. Machine direction orientations, and wire-side and felt-side surfaces of each sample were identified either before or during the microscopic studies. The fibre measurement and microscopic procedures used have been described in detail elsewhere (1).

OPACITY OF WHITE OFFSET PRINTING PAPERS

White offset printing papers (70 g/m²) produced at Kinleith since late 1975 tended to have lower than normal opacities, and more difficulty was experienced in meeting specifications for opacity. According to technical personnel at the mill the pulping, the pulp processing, and the papermaking conditions had remained similar to those used before the problem manifested itself late in 1975. No changes in wood quality, pulp

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blend, or filler mix had apparently occurred.

**Paper samples**

Two sets of paired paper samples with ‘good’ and ‘poor’ opacity were examined. For the first set of samples (Set-I), paper with ‘good’ (1975) and ‘poor’ (1976) opacity had similar grammages but markedly different bulk and ash values (Table 1). Clay had been added at the expense of fibre to increase the opacity of the 1976 sample. For the second set of samples (Set-II) a larger difference in opacity corresponded with similar grammages but smaller differences in bulk and ash values (Table 1). Flakt-dried pulp samples corresponding to the papers of Set-I were also examined. Pulp contents were nominally 70 per cent radiata pine and 30 per cent tawa, and clay mixes were nominally 1 part titanium dioxide to 22 parts clay.

<table>
<thead>
<tr>
<th>Test Quantity</th>
<th>Air-dried</th>
<th>Paper Set-I 1975</th>
<th>Paper Set-II 1976</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grammage g/m²</td>
<td>a.d.</td>
<td>69.6</td>
<td>69.6</td>
</tr>
<tr>
<td></td>
<td>o.d.</td>
<td>66.2</td>
<td>66.5</td>
</tr>
<tr>
<td>Bulk cm³/g</td>
<td>a.d.</td>
<td>1.54</td>
<td>1.49</td>
</tr>
<tr>
<td></td>
<td>o.d.</td>
<td>1.54</td>
<td>1.49</td>
</tr>
<tr>
<td>Density kg/m³</td>
<td>a.d.</td>
<td>649</td>
<td>671</td>
</tr>
<tr>
<td></td>
<td>o.d.</td>
<td>710</td>
<td>660</td>
</tr>
<tr>
<td>Ash %</td>
<td>a.d.</td>
<td>8.3</td>
<td>11.0</td>
</tr>
<tr>
<td></td>
<td>o.d.</td>
<td>10.0</td>
<td>11.2</td>
</tr>
<tr>
<td>Opacity %</td>
<td>a.d.</td>
<td>82</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>o.d.</td>
<td>85</td>
<td>85</td>
</tr>
</tbody>
</table>

**Fibre characteristics**

Papers with ‘good’ opacity (1975 making) were less consolidated and showed lower degrees of fibre bonding and fibre collapse than those with ‘poor’ opacity (Fig. 1). The small-diametered tawa fibres and the large-diametered radiata pine fibres were most clearly distinguishable in the papers with ‘good’ opacity (Fig. 1A). Additionally, tawa fibres in the papers with ‘good’ opacity appeared to be more numerous and to have more circular cross-sectional shapes (less collapsed) than those in the papers with ‘poor’ opacity. Examination of papers in sectional view confirmed that fibres were more collapsed in the samples with ‘poor’ opacity (Fig. 2). Papers with ‘good’ opacity (Fig. 2A) contained fibres with distinctly unbounded and roughly circular lumens when compared with those of ‘poor’ opacity (Fig. 2B).

Quantitative measurement of fibre configurations in the 1975 and 1976 papers showed that mean fibre lengths (Table 2), as well as fibre length distributions (Fig. 3) were generally similar, which suggested that differences in wood quality were minimal and unlikely to be related to the measured differences in opacity (Table 1). Cumulative frequency distributions indicated that the long radiata pine fibres were possibly slightly longer in the 1976 than in the 1975 papers (Fig. 4). The peaks at the lower ends of the distribution curves showed the large numbers of short tawa fibres in the two sets of papers examined (Fig. 3). Mean fibre lengths (Table 2) and fibre length distributions (Fig. 5) of the papers and of corresponding flakt-dried pulps were generally similar. The longer radiata pine fibres were, however, apparently shortened slightly by pulp processing (pumping and refining, etc.). The consistent fibre length distributions of the four papers and of the two pulp samples showed that tawa/radiata pine pro-

![Fig. 1 — Surface views of the wire-sides of papers with 'good' (A) and 'poor' (B) opacity.](image1)

**Table 2**

<table>
<thead>
<tr>
<th>Fibre lengths and fibre diameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Samples</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>Set-1:</td>
</tr>
<tr>
<td>Paper 1975</td>
</tr>
<tr>
<td>Paper 1976</td>
</tr>
<tr>
<td>Pulp 1975</td>
</tr>
<tr>
<td>Pulp 1976</td>
</tr>
<tr>
<td>Set-II:</td>
</tr>
<tr>
<td>Paper 1975</td>
</tr>
<tr>
<td>Paper 1976</td>
</tr>
</tbody>
</table>

**Note:**
- Only values within each set can be compared
- Fibre lengths different at 95% level if different by more than 0.19 mm
- Fibre diameters different at 95% level if different by more than 1.86 μm.
produced during this period. The different curves for Set-I and Set-II of Figure 6 are the result of measuring fibres photographed at different angles of tilt.

Quantitative measurement of the extent of fibre collapse confirmed the conclusions from the microscopic examination (Fig. 2). For all papers examined, the number of uncollapsed fibres was greater in the papers with "good" opacity by more than 10 per cent (Table 3). Actual magnitudes of lumen diameters in the 1975 papers, measured in the vertical plane of the sheet, were consistently almost twice those in the papers with "poor" opacity (Table 3). Mean fibre diameters (Table 2), and fibre diameter distributions (Fig. 6), supported the collapse data in so far as papers with 'poor' opacity contained fibres with slightly larger diameters when measured in the surfaces of the paper samples. The slightly larger fibre diameters in these 1976-produced papers apparently represent the end effects of their greater ease of fibre collapse and intra-fibre bond development during papermaking.

Table 3
Fibre Collapse

<table>
<thead>
<tr>
<th>Paper samples</th>
<th>Uncollapsed fibres</th>
<th>Lumen diameter per uncollapsed fibre examined</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%</td>
<td>μm</td>
</tr>
<tr>
<td>Set-IA:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1975</td>
<td>34.8</td>
<td>5.01</td>
</tr>
<tr>
<td>1976</td>
<td>22.2</td>
<td>2.69</td>
</tr>
<tr>
<td>Set-IB:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1975</td>
<td>26.9</td>
<td>3.08</td>
</tr>
<tr>
<td>1976</td>
<td>26.0</td>
<td>2.85</td>
</tr>
<tr>
<td>Set-II:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1975</td>
<td>28.0</td>
<td>3.02</td>
</tr>
<tr>
<td>1976</td>
<td>17.5</td>
<td>1.93</td>
</tr>
</tbody>
</table>

Note:
1. Only values within each set can be compared
2. The two pairs of values for Set-I represent the results of repeat measurements and different methods of statistical analyses
3. Statistical significance:
   - Set-IA: Different at 0.05 level if lumen diameters different by more than 0.98 μm
   - Set-IB: Different at the 0.00002 level of significance
   - Set-II: Different at the 0.0001 level of significance.

Fig. 2—Sectional view of fibres in papers with 'good' (A) and 'poor' (B) opacity.

Fig. 3—Fibre length distributions in papers.

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Clay characteristics

To maintain specifications for opacity, the clay contents in the papers of Set-I (Table 1) were higher in the 1976 than in the 1975 product (Fig. 7). The examination of felt-side surfaces showed broad coverage of clay in the paper with 'poor' opacity (Fig. 7B). Clay was less obvious in the paper with 'good' opacity and its distribution was generally patchy (Fig. 7A) which suggested that the 'good' opacity of this paper, when compared with the 1976 product, is probably not related closely to clay content. The papers of Set-I appeared to contain clays of similar quality (Fig. 7).

Clay contents of the paper of Set-II (Table 1), although present in roughly similar amounts, were of different quality (Fig. 8). The product with 'poor' opacity contained larger particles than the papers with 'good' opacity. The different particle sizes of the two clays prevented definite conclusions being made concerning the relative effects of clay distributions on paper opacity. It is most likely, however, that the apparent
high specific surface of the finer clay would bring about
the greatest improvement in paper opacity.

**Discussion and conclusions**

The results strongly suggest that the low opacity of
the 70 g/m² white offset printing papers produced in
1976 can be explained by a loss in light-scattering within
the fibre webs due to substantial increases in both inter-
fibre and internal fibre bonding. These high extents of
fibre bonding or web consolidation are most probably
related to increased degrees of refining. An alternative
explanation could be that pulps produced in 1976 were
more easily refined than those produced in 1975. This
hypothesis implies changes in pulping and/or bleaching
processing. Distribution trends for fibre length and
fibre diameter were generally similar in the papers with
'good' and 'poor' opacity, which eliminated the
possibility that the apparent changes in opacity could
be related to differences in wood quality and to the propor-
tion of tawa to radiata pine.

Clay content of the paired sets of papers examined
varied in either their quality or quantity:

For the samples of Set-I (Table 1), clay content was
high for the paper with 'poor' opacity, and fibre con-
tent and web thickness was correspondingly low com-
pared with the paper with 'good' opacity. Thus, to
maintain a constant grammage (70 g/m²), clay was
added at the expense of fibre, and sheet density was
correspondingly increased.

For the samples of Set-II (Table 1), clay contents
and consequently fibre contents were roughly similar
although their opacity difference was twice that of
the samples in Set-I. On the basis of the examination
of the Set-I samples (Table 1), only part of the four
per cent difference in opacity of the Set-II samples
can be attributed to the increased consolidation and
fibre bonding in the paper with 'poor' opacity (Table
3) (Fig. 1 and 2). A proportion of the difference in
opacity must, therefore, be related to the different
particle sizes of the two clays used in the samples of
Set-II (Fig. 7 and 8). The apparent high light-
scattering properties (high specific surface) of the
finer clay used in the 1975 sample would account in
part for its high opacity. It is of interest to speculate
whether the opacity of the 'poor' paper of Set-I
(Table 1) would have been substantially improved if
the finer clay had been used in its manufacture.

**PAPER 'BRUISING' IN KRAFT LINER**

Kraft liner produced on the No. 6 paper machine
often contained blemishes or bruises in the form of
dark-coloured areas of up to 3 cm². Paper 'bruising'
ocurred during calendering but was distinct from paper
blackening which normally manifests itself when papers
are calendered at moisture contents greater than 6 to 8
per cent (2).

**Microscopic studies**

Preliminary examination of normal and bruised
papers showed that bruised surfaces contained strongly
bonded and collapsed fibres when compared with those
in unbruised material (Fig. 9). On this basis the follow-
ing alternative hypotheses were examined:

That the high degree of fibre bonding and collapse
was related to the development of excessive bonding
as water was removed from the paper on passing
through calender stacks. Papers are normally con-
sidered to be dry and interfibre bonding to be com-
plete by the time they reach the calenders and,
therefore, this hypotheses required that web moisture
distributions were uneven and moisture concentra-
tion occurred in pockets at sites of bruising.

That the high degree of fibre bonding and collapse
in bruised regions was caused by the presence of a contaminant or contaminants, on or in the paper web during calendering.

Careful examination of the paper sheets showed that the second hypothesis was the more likely for the following reasons:

The surfaces of fibres in bruised areas appeared to be covered with an amorphous coating (Fig. 9).

Fibres were strongly collapsed and very tightly bonded together (Fig. 9). It was inconceivable that such a high degree of fibre collapse and bonding could be accounted for by drying paper webs from water alone (3). Bonding and subsequent paper bruising which developed during calendering were apparently irreversible and minimized post-treatment web relaxation. This behaviour suggested that adhesive bonding rather than normal hydrogen-bonding was involved in the development of paper bruising.

Sectioned faces of bruised and normal portions of kraft liner showed that sheet thicknesses in the bruised regions were 15 to 20 per cent lower than those in the unbruised areas (Fig. 10 and 11). This observation was confirmed by thickness measurements. Fibres were visibly more collapsed and bonded together in the cut faces of the bruised samples (Fig. 11). The number of fibres contributing to the thickness of the bruised and unbruised regions was similar. Thus, blemishes could not be related to fibre agglomerates within or on paper webs at the time of calendering.

The above observations strongly suggested that "bruising" was caused by the presence of a contaminant or contaminants on or within kraft liner paper at the time of entry into the calender stack.

Chemical studies
Kraft liner produced at Kinleith normally contains up to 0.5 per cent rosin size, and up to 0.5 per cent of a polyacrylamide added as a dry strength agent.

Chemical analyses and paper disintegration studies of unbruised and bruised papers were carried out by J. A. Lloyd (unpublished data). He showed that extraction of the paper samples with 1M NaOH solution at 100°C
caused the unbruised material to disintegrate. In contrast, the bruised samples remained intact after 2 hours of extraction and agitation, and even after a further 2 1/2 hours of extraction at 125°C.

Analyses of the NaOH extracts of bruised and unbruised samples from two separate makings of kraft liner showed very different compositions. In the first instance extracts of bruised material contained about four times more resin acids than the extracts of unbruised material. In the second instance an oil-based substance (possibly defoamer) was a major component in the bruised extract. It is noteworthy, however, that fibres were not disintegrated and most of the 'contaminant' was not extracted from the bruised samples by the NaOH treatments. Polycrystal contents of unbruised and bruised samples were similar.

**Conclusions**

Bruising in kraft liner was the result of a 15 to 20 per cent decrease in sheet thickness which was brought about by corresponding increases in intrafibre collapse and both internal and interfibre bonding. This increased bonding and the inability of the 'bruised' areas to return to normal sheet thicknesses were related to uneven distributions of additives and/or other contaminants present in the paper network.

**SUMMATION**

The fibre characterization studies described in the present paper identified factors which influenced the opacity of white offset printing papers and the 'bruising' of kraft liner papers. This was in accordance with the aims of the investigations which were to explain and/or identify differences in industrial papers produced under nominally the same pulp and papermaking conditions. The isolation of the identified factors within the complexities of the mill system by mill personnel was the final stage of the investigations and is not described in detail in this paper.

Detailed examination of mill records showed that there had been a small change in pulp quality consistent with the findings of the opacity study. Pulps used in 1976 were found to have been slightly easier to refine.
than those used in 1975.

The overall effectiveness of the fibre characterization studies in eliminating 'bruising' in kraft liner was less obvious because of difficulties inherent in isolating the source(s) and location(s) of contaminants within the mill system. The fact that different substances were extracted from bruised samples collected from different makings suggested that factors or substances other than those already identified were involved in initiating or nucleating the uneven distribution of additives or contaminants in kraft liner papers.

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REFERENCES

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