

# Effect of process variables in laboratory deinking experiments

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**ABSTRACT:** *Laboratory test conditions can greatly affect the results of deinking experiments. A series of laboratory tests were performed using a model of a flotation-wash deinking process. Handsheet brightness increased with decreasing flotation cell consistency of a 70/30 newsprint/magazine pulp slurry. Yield losses increased with decreasing flotation cell pulp consistency. Increased consistency in the pulping step did not substantially affect handsheet brightness or yield. Increased content of clays and fillers in the furnish generally leads to increased yield losses in both flotation and washing steps of deinking experiments.*

**KEYWORDS:** *Deinking, flotation, laboratories, pilot plants, process variables, test methods.*

The role of surfactants in wash and flotation deinking has been recently reviewed (1-3). Deinking processes utilize surfactants for the dispersion and removal of various inks and coatings from a variety of furnishes. Surfactants function in the pulping step to promote ink particle detachment from cellulose fiber. The surfactant promotes dispersion of the detached ink in the aqueous medium. This ink-containing dispersion can be separated from the fiber in one or more washing steps. Alternatively, flotation can be used to collect the dispersed ink particles on the surfaces of air bubbles and trap them in a froth layer. Separation of the froth layer from the pulp slurry removes the ink particles.

The removal of dispersed ink from the pulp slurry by flotation or by washing or by both methods prevents ink redeposition on cellulose during papermaking.

The optimum deinking surfactant to use is dependent on:

1. The deinking process—wash, flotation, or a combination of wash and flotation. The type of deinking equipment and the detailed engineering of the process can also influence surfactant deinking effectiveness.
2. The furnish—both the type of paper and the ink composition can affect deinking surfactant performance. Fillers and coatings can also

influence surfactant effectiveness. Representative ink compositions and printing processes are described in Ref. (3). Laser and xerographic inks present particularly difficult deinking problems.

3. The effect of deinking surfactant on wet-end chemicals. Some deinking surfactants may become adsorbed on cellulose fibers (4). Adsorbed surfactants may decrease performance of wet-end chemical additives. Hence, higher loadings of these additives may be required for secondary fiber. This is particularly the case for wet-strength additives and sizing agents (5, 6). Other properties of secondary fibers can also affect final paper properties. These properties include fiber shortening, lower fiber flexibility, and reduced fiber bonding (7).

## Deinking testing

Laboratory tests play a critical role in deinking surfactant product and process development. Pilot mill tests and commercial mill trials are expensive and time consuming. Therefore, they should be performed only after laboratory experiments have defined the optimum chemical structures and deinking process conditions.

Laboratory tests have been designed, conventionally, to model mill operations. However, factors 1 and 2, above, are difficult to scale accurately. Many compromises must be made in designing laboratory-scale pulping and deinking equipment and procedures.

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## Secondary Fiber

As a result, laboratory experiments are not exact scale models of commercial deinking processes. Therefore, scaleup of laboratory test results to mill operations is not obvious and should be verified.

The performance of laboratory and pilot mill scale tests under the same conditions will allow some of the effects of scaleup to be determined. Important process performance properties that can be affected by scaleup include deinked paper brightness, ink speck count of the deinked paper and the relative sheet surface area occupied by ink specks, and yield loss during the deinking process.

At this date, few pilot mills include whitewater recycling in their operations. For a limited series of tests, it is easier to design procedures to model process water recycle on a laboratory scale than on a pilot mill scale.

If laboratory test results can be correlated with scaled-up deinking operations, one can use laboratory tests to predict absolute or relative deinking performance. The effect of different process variables on flotation deinking can also be conveniently studied in the laboratory. The effect of flotation time, screen mesh size, pulp consistency, furnish composition, and pH on flotation deinking of newsprint/magazine mixtures are discussed below.

### Results

Pilot mill and laboratory test procedures are summarized in the the Testing Procedures section. The abbreviation SDA stands for Shell Deinking Agent. These developmental products are available under the tradename Nonatell™ deinking surfactants. Other flotation deinking surfactants are identified by the acronym OS for other surfactant.

#### Correlation of laboratory and pilot mill flotation deinking results

Before studying the effect of process conditions on laboratory test results, it is critical to demonstrate that our laboratory tests produced results relevant

I. Brightness results obtained in WRC laboratory and WMU pilot mill studies<sup>a</sup>

| Test<br>surfactant | <u>Brightness<sup>b</sup></u> |      | WMU Pilot mill<br>results <sup>b</sup> | <u>Brightness difference</u><br><u>Denver cell — Pilot mill</u> |          |
|--------------------|-------------------------------|------|----------------------------------------|-----------------------------------------------------------------|----------|
|                    | <u>WRC Laboratory results</u> |      |                                        | <u>Lab flotation time, min</u>                                  |          |
|                    | <u>Flotation time, min</u>    |      |                                        | <u>3</u>                                                        | <u>6</u> |
|                    | 3                             | 6    |                                        |                                                                 |          |
| SDA-1              | 47.8                          | 48.3 | 49.5                                   | -1.7                                                            | -1.2     |
| SDA-2              | 46.4                          | 46.7 | 49.2 <sup>c</sup>                      | -2.8                                                            | -2.5     |
| SDA-3              | 48.2                          | 48.3 | 47.3                                   | 0.9                                                             | 1.0      |
| SDA-4              | 47.5                          | 48.3 | 46.3 <sup>c</sup>                      | 1.2                                                             | 2.0      |
| OS-1               | 48.0                          | 48.8 | 48.7                                   | -0.7                                                            | 0.1      |
| OS-2               | 47.7                          | 49.4 | 44.6                                   | 3.4                                                             | 4.8      |

<sup>a</sup>Test procedures are described in the Testing Procedures section.

<sup>b</sup>Brightness values given are from the flotation cell accepts after the pulp was passed over a 75 mesh sidehill screen used in the pilot scale tests.

<sup>c</sup>These two tests were performed with a different bale of old newsprint purchased from the same broker.

to scaled-up deinking operations. Pilot-scale deinking studies were performed on a contract basis at Western Michigan University (WMU). Our focus was on flotation deinking because we already have a large knowledge base on the effect of scaling-up our laboratory wash deinking procedures. The objective of these tests was not to demonstrate the effectiveness of particular optimized deinking surfactants. Instead, we wanted to verify the validity of our laboratory tests before performing additional laboratory tests to develop improved deinking surfactants and to study the effect of process variables on ink removal efficiency.

Pilot mill tests using 250 lb of a 70/30 mixture of newsprint and magazines (actually, mostly newspaper inserts) were performed at WMU on a contract basis. The sequence of pilot mill operations was pulping at 5% consistency followed by dilution to 1% consistency in a storage chest. The diluted pulp was then passed through a Voith flotation cell. The flotation accepts were transferred back to the storage chest (to be passed through the flotation cell an average of eight times). The pulp slurry was then thickened over a sidehill screen. To focus on only deinking surfactant effects, bleaching was omitted from both the laboratory and pilot mill studies.

Deinking tests using 20 g of the same furnish were performed in our labora-

tory (abbreviated WRC) using Denver flotation cells. The pilot mill scale-up factor from laboratory tests was 5675.

#### Scaleup of newsprint deinking

**Sheet brightness.** Deinked sheet brightness results are summarized in Table I. In general, the pilot mill test brightness values were close to those obtained in the corresponding laboratory experiments. The absolute values of the individual differences between laboratory and pilot-scale brightness values in Table I were used to calculate the average brightness difference. (This avoids an overly optimistic average difference resulting from the addition of positive and negative numbers.)

The average brightness difference between the laboratory 3-min flotation tests and that of the pilot-scale tests was 1.8 units. Applying the same analysis to the 6-min laboratory flotation data, the average brightness difference between laboratory and pilot-scale tests was 1.9 units. These values compare well with the minimum experimentally significant brightness difference of 2 units we usually use to evaluate differences in surfactant performance in single laboratory experiments.

The flotation accepts passed through the flotation cell about eight times during each pilot-scale test. Therefore, it was mildly surprising that

II. Flotation step yield loss results obtained in WRC laboratory and WMU pilot mill studies<sup>a</sup>

| Test<br>surfactant                                        | Flotation yield loss <sup>b</sup> |      |                           | Yield loss difference<br>Denver cell — Pilot mill |      |
|-----------------------------------------------------------|-----------------------------------|------|---------------------------|---------------------------------------------------|------|
|                                                           | WRC Laboratory results            |      | WMU Pilot mill<br>results | Lab flotation time, min                           |      |
|                                                           | Flotation time, min               |      |                           | 3                                                 | 6    |
|                                                           | 3                                 | 6    |                           | 3                                                 | 6    |
| SDA-1                                                     | 17.6                              | 19.6 | 24.7                      | -7.1                                              | -5.1 |
| SDA-2                                                     | 24.4                              | 27.4 | 14.4                      | 10.0                                              | 13.0 |
| SDA-3                                                     | 11.6                              | 13.3 | 16.1                      | -4.5                                              | -2.8 |
| SDA-4                                                     | 19.2                              | 22.2 | 27.7                      | -8.5                                              | -5.5 |
| OS-1                                                      | 31.2                              | 36.2 | 12.7                      | 18.5                                              | 23.5 |
| OS-2                                                      | 24.1                              | 27.5 | 9.3                       | 14.8                                              | 18.2 |
| Average flotation step yield loss difference <sup>b</sup> |                                   |      |                           | 10.4                                              | 11.4 |

<sup>a</sup>See Table I, footnotes a and c.

<sup>b</sup>Calculated from the absolute values of the yield loss differences in the individual experiments.

<sup>a</sup>See Table I, footnotes a and c.

<sup>b</sup>Calculated from the absolute values of the yield loss differences in the individual experiments.

III. Total yield loss results obtained in WRC laboratory and WMU pilot mill studies<sup>a</sup>

| Test<br>surfactant                       | <u>Total yield loss<sup>b</sup></u> |      |                                        | Yield loss difference          |       |
|------------------------------------------|-------------------------------------|------|----------------------------------------|--------------------------------|-------|
|                                          | WRC Laboratory results              |      | WMU Pilot mill<br>results <sup>b</sup> | Denver cell — Pilot mill       |       |
|                                          | <u>Flotation time, min</u>          |      |                                        | <u>Lab flotation time, min</u> |       |
|                                          | 3                                   | 6    |                                        | 3                              | 6     |
| SDA-1                                    | 21.2                                | 22.0 | 41.3                                   | -20.1                          | -19.3 |
| SDA-3                                    | 17.3                                | 15.3 | 29.5                                   | -12.2                          | -14.2 |
| SDA-2                                    | 34.5                                | 31.3 | 21.2                                   | 13.3                           | 10.1  |
| SDA-4                                    | 22.4                                | 24.7 | 34.5                                   | -12.1                          | -9.8  |
| OS-1                                     | 33.2                                | 37.5 | 31.6                                   | 1.6                            | 5.9   |
| OS-2                                     | 26.4                                | 29.2 | 27.9                                   | -3.5                           | 1.3   |
| Total yield loss difference <sup>b</sup> |                                     |      |                                        | 11.8                           | 10.1  |

<sup>a</sup>See Table I, footnotes a and c.

<sup>b</sup>Calculated from the absolute values of the yield loss differences in the individual experiments.

<sup>a</sup>See Table I, footnotes a and c.

<sup>b</sup>Calculated from the absolute values of the yield loss differences in the individual experiments.

brightness agreement with the pilot mill results was actually better for the 3-min laboratory flotation time tests than for the 6-min laboratory flotation experiments.

With the exception of Other Surfactant 2 (OS2), deinked sheet brightness rank order was the same in the laboratory and pilot mill flotation deinking tests. OS2 also provided the poorest agreement between laboratory and pilot mill results of any surfactant tested. Different production lots of two competitive deinking products were used in the WRC and WMU tests. It is possible that OS2 lot-to-lot variation could account for the brightness differences

for this competitive surfactant in laboratory and pilot-scale tests.

**Yield Loss.** Yield losses in the flotation step of laboratory and pilot-scale deinking are summarized in **Table II**. For three of the deinking surfactants, the Denver cell flotation yield losses were greater than the pilot-scale flotation step yield losses. For the other three deinking surfactants, the Denver cell flotation yield losses were less than the pilot-scale flotation step yield losses.

The absolute values of the yield loss differences were used for comparison of laboratory and pilot-scale data. The average yield loss difference for the

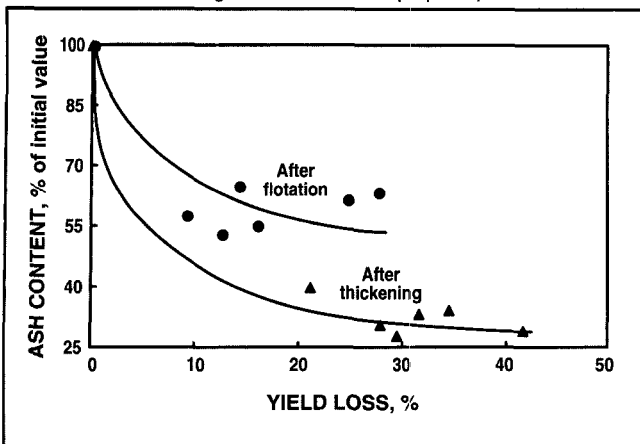
flotation step between laboratory and pilot-scale tests was 10.4 percentage points for the 3-min laboratory flotation time. For the 6-min laboratory flotation time, the corresponding difference was 11.4 percentage points. The laboratory procedure involves assisting foam removal by using a spatula to scrape the froth off the surface and into a catch basin. (This action mimics the effect of the pilot-scale Voith flotation cell paddles.) A single operator performed the laboratory test series. (Without care, this procedure can be operator dependent)

Additional yield loss was observed in the thickening step. In the laboratory, thickening was performed by pouring the flotation cell contents over a 100-mesh screen. In the pilot-scale thickening step, the flotation cell accepts were passed over a 75-mesh sidehill screen. The total fiber losses for the flotation and thickening steps are summarized in **Table III**. Results are consistent with the flotation step yield losses described above. For half the measurements, the laboratory deinking experiment provided a higher fiber loss than did the pilot-scale test. In the other tests, the laboratory experiment produced a lower fiber loss.

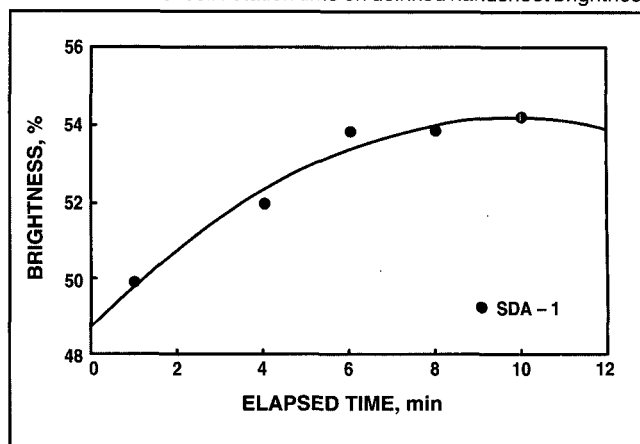
The average total yield loss differences for the laboratory and for the pilot mill tests (**Table III**) were determined as described above for the flotation step yield loss. Absolute values of the total yield loss difference were used in the calculation of the average total yield loss difference. This difference was 11.8 and 10.1 percentage points for the 3-min and 6-min laboratory flotation times, respectively. These differences were very similar to those for the flotation step alone. In the laboratory, the thickening step can be somewhat operator dependent. This is because different technicians may use different degrees of force in pressing the pulp against the wire mesh screen.

The rank order of total yield loss in the laboratory experiments was similar for the 3-min and 6-min flotation tests, the exception being OS-1 and SDA-2. The OS-1 and SDA-2 rank or-

1. Ash content and brightness of deinked pulps in pilot mill tests



2. Effect of Denver cell flotation time on deinked handsheet brightness



der in the 3-min tests were 1-2 and in the 6-min test 2-1.

The results summarized in Fig. 1 indicate that, above a flotation step yield loss of 10%, ash content in the deinked pulp did not decrease with increased yield loss. However, the ash content in handsheets made after both flotation and thickening steps did decrease with increasing yield loss. Most of the decrease appeared to occur at less than 20% total yield loss.

The effect of the pulping step on average fiber length and on the fiber length distribution could play a role in the differences in yield losses observed on scaleup. Comparison of average fiber length and fiber length distributions obtained after laboratory and pilot mill pulping should aid in determining the effect of changing from a TAPPI disintegrator (laboratory) to a Black Clawson Hydrapulper (pilot mill) on yield loss.

## Laboratory studies of deinking process variables

The above results demonstrate the correlation of brightness results in laboratory and pilot mill scale experiments and provide some indication of the limitations of yield loss predictions on scaleup. This defines the usefulness and limitations of laboratory tests. Laboratory experiments are appropriate to study the effect of process variables on flotation deinking processes. Therefore, laboratory experi-

ments were performed to determine the effect of deinking process variables on ink removal and yield loss. Unless otherwise noted, deinking surfactant concentration was 0.2% by weight relative to furnish. The deinking process was a generic flotation—wash process. It was not a model of the process used in a particular mill (see Testing Procedures).

**Effect of Denver cell flotation time.** Deinked sheet brightness did not continuously increase with time but reached a plateau after 6–10 min (Fig. 2). Despite increasing yield loss (Fig. 3), brightness did not increase at flotation times of more than 6–10 min. Any remaining ink is, probably, still attached to the fibers and is unavailable for removal by flotation.

Yield loss increased steadily with increased flotation time (Fig. 3). (The furnish was a 70/30 mixture of a Canadian newspaper and magazine.) The increasing yield loss even at longer flotation time was expected because the critical micelle concentration (9) of the deinking surfactant (SDA-1) is quite low. Therefore low concentrations of this surfactant provide foaming and ink removal activity.

## Effect of washing step screen mesh size

Fiber loss in washing operations can be significant. Various screen mesh sizes have been used in experiments performed in different laboratories. A

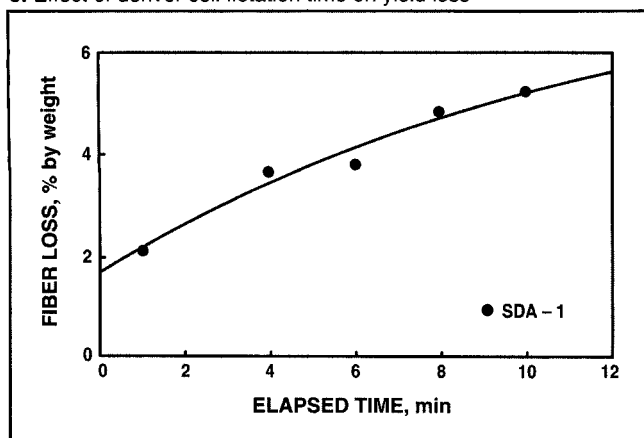
set of experiments was performed to determine the effect of screen size on yield loss of the wash step of a flotation—wash process. Results are summarized in Table IV.

A decrease in mesh size from 30 to 100 U.S. mesh (0.0234 in. or 0.6 mm to 0.0059 in. or 0.15 mm) did not increase handsheet brightness. Because the flotation steps were identical, flotation yield loss should not differ significantly for the two flotation steps. The flotation yield loss difference (3.3 percentage points) serves as an indication of flotation yield loss experimental uncertainty. Washing step yield loss was 4.4 percentage points greater when using the more porous 30 U.S. mesh screen. The increased washing yield loss is in the direction expected but may not be experimentally significant. Total yield loss for the two tests was similar and definitely within experimental uncertainty. The 100 U.S. mesh screen may already be so permeable that additional increases in yield loss on going to the 30 U.S. mesh screen might not be significant.

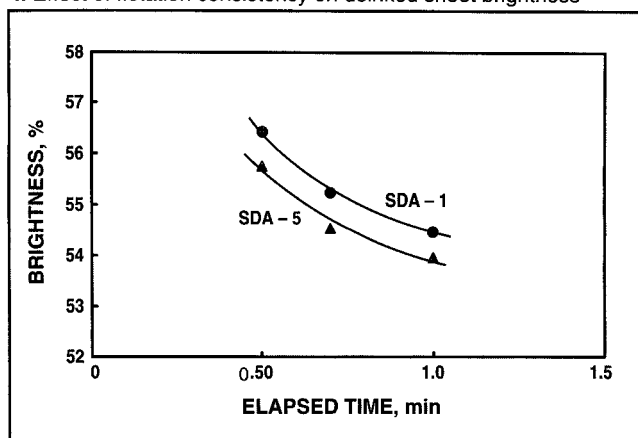
**Effect of pulp consistency.** The results summarized in Table V indicate the effect of increasing pulping consistency from 1% to 2% to 4%. Overall, handsheet brightness did not change significantly with increased pulp consistency. (Brightness differences were less than the two points considered experimentally significant.)

Experimental uncertainty makes it difficult to say whether the ink par-

### 3. Effect of denver cell flotation time on yield loss



### 4. Effect of flotation consistency on deinked sheet brightness



### IV. Effect of washing step screen mesh size on yield loss<sup>a</sup>

| Washing screen<br>U.S. mesh | Size of openings, |        | Brightness,<br>% | Yield loss, % by weight |      |       |
|-----------------------------|-------------------|--------|------------------|-------------------------|------|-------|
|                             | mm                | in.    |                  | Flotation               | Wash | Total |
| 30                          | 0.6               | 0.0234 | 58.0             | 13.5                    | 18.2 | 31.8  |
| 100                         | 0.15              | 0.0059 | 58.2             | 16.8                    | 14.1 | 30.9  |

<sup>a</sup>See Testing Procedures for descriptions. The deinking surfactant was 0.25% (by weight relative to furnish) SDA-5.

### V. Effect of consistency during pulping on deinking in a flotation — wash process<sup>a</sup>

| Pulp<br>consistency | Brightness,<br>% | Image analysis      |                          | Yield loss, % by weight |            |              |
|---------------------|------------------|---------------------|--------------------------|-------------------------|------------|--------------|
|                     |                  | No. of<br>particles | Ink surface<br>area, ppm | Flotation               | Wash       | Total        |
| 4                   | 54.8             | 238                 | 182                      | 23.5                    | 21.1       | 44.6         |
| 2                   | 55.2             | 277                 | 440                      | 14.5                    | 8.8        | 23.2         |
| 1                   | 55.2 ± 0.5       | 308 ± 56            | 354 ± 102                | 13.4 ± 1.8              | 13.4 ± 2.6 | 6 28.0 ± 3.2 |

<sup>a</sup>Test procedures are summarized in the Testing Procedures section. A Hamilton Beach blender was used for the laboratory pulping. The deinking surfactant was 0.2% (by weight relative to furnish) SDA-5.

### VI. Effect of 70:30 newsprint:magazine pulp consistency during flotation on brightness and yield loss<sup>a</sup>

| Deinking<br>surfactant | Pulp consistency<br>in Denver cell, % | Brightness,<br>% | Yield loss, % |      |       |
|------------------------|---------------------------------------|------------------|---------------|------|-------|
|                        |                                       |                  | Flotation     | Wash | Total |
| SDA-1                  | 1.0                                   | 54.4             | 18.3          | 5.0  | 23.3  |
| SDA-1                  | 0.7                                   | 55.2             | 25.0          | 5.3  | 30.3  |
| SDA-1                  | 0.5                                   | 56.4             | 28.6          | 10.5 | 39.1  |
| SDA-5                  | 1.0                                   | 53.9             | 17.7          | 6.1  | 23.7  |
| SDA-5                  | 0.7                                   | 54.5             | 24.8          | 6.1  | 30.9  |
| SDA-5                  | 0.5                                   | 55.7             | 28.4          | 8.4  | 36.8  |

<sup>a</sup>Test procedures are detailed in the Testing Procedures section.

ticle count (Table V) increased significantly with decreasing pulping consistency. The handsheet surface area occupied by ink particles did appear to increase significantly on decreasing pulp slurry consistency from 4% to 2%. A further decrease to 1.0% had little effect on handsheet ink particle surface area.

Patterns in the flotation step and wash step yield losses suggest that any differences in yield losses were not experimentally significant. However, high consistency (8–15%) pulping should be studied under similar shear conditions before firm conclusions are drawn.

The results summarized in Table VI indicate that decreases in flotation step pulp consistency resulted in increased solids loss during flotation. This was true for both surfactants studied.

While changes in handsheet brightness were small, they were continuous on decreasing pulp consistency from 1.0% to 0.7% to 0.5% (Table VI and Fig. 4). The consistency of the brightness trend for two different surfactants suggests that the trend is real. The brightness increase on going from 1.0% to 0.5% consistency using SDA-1 is greater than two points. This is an experimentally significant difference.

All studies were done using a Denver cell. Observed trends in brightness and yield loss may be due to the design of the Denver flotation cell and may not occur in flotation cells of different design.

**Newsprint/magazine ratio.** Two different paper blends were chosen for study. The first is a 70/30 mixture (by weight) of an offset printed newspaper, *The Houston Post*, and magazines. This newsprint/magazine ratio is planned for use in most of the new U.S. deinking mills. The magazine portion was a 1/1 mixture of *Time* and *People*. These magazines were chosen because of their large circulation and wide distribution in North America.

The other newsprint/magazine ratio chosen was 49/51 (by weight). *The Houston Post* was the newsprint used. In contrast to the 70/30 blend, the newspaper inserts were not omitted. Instead, the inserts were included in the magazine portion of the paper blend. The average level of inserts present in *The Houston Post* was determined by weighing 14 consecutive issues. The inserts were then separated and weighed. The inserts comprised 17.7% by weight of these newspapers. This was considered a representative average. The magazine used was *Time* magazine.

Ash plays an important role in the flotation process (10). Clays and other ash components are believed to provide sites for ink attachment and ink particle agglomeration. The ink-coated particle is then trapped in the foam layer and separated from the pulp slurry. Calcium ion, often originating from calcium carbonate paper filler, is believed to improve the effectiveness of fatty acids often used in flotation deinking. For these reasons, ash content of the discarded paper is an important parameter. Paper ash content values are summarized in **Table VII**.

These results indicated that the ash content of the two newsprint/magazine mixtures differed significantly. The two newsprint/magazine blends can be considered as high clay/filler and low clay/filler furnishes.

The small size of clay and mineral filler particles is thought to promote their separation into the foam layer during the flotation. This would lead to higher ash content in the solids trapped in the foam lost fiber than in

the original discarded-paper mixture. To test this hypothesis, the solids trapped in the foam in the flotation step were collected and the ash content determined. The 70/30 furnish was used in this deinking experiment.

Surprisingly, the ash content of the collected solids was 5.41%, less than the 8.11% in the 70/30 furnish being deinked. This indicates that either the conventional wisdom cited above is wrong or only a small amount of clays are required to promote improved ink separation. The clays and mineral fillers are thought to be smaller than most of the cellulose fibers. If this is correct, the low ash content in the material trapped in the foam suggests there is surprisingly little segregation by size during flotation. Again this is contrary to conventional wisdom. Additional studies are necessary to confirm these single test findings. However, results are consistent with those reported in Ref. (10).

Conventional wisdom would predict that the higher ash content of the 49/51 blend would result in an increased "yield loss" during flotation as more clays and minerals are trapped in the foam. However, this expected increase was not observed during flotation of the 49/51 and 70/30 newsprint/deinking blends (see **Table VIII**). The difference in flotation step yield loss for the two furnishes was 5.1 percentage units. The higher flotation step yield loss for the 70/30 mixture was more than compensated by the substantially lower washing step solids loss.

In the washing step, the 70/30 mixture yield loss was 10.6 percentage units less than that of the 49/51 mixture. This is more than a 50% decrease in yield loss. The higher level of small mineral particles in the 49/51 furnish would pass more easily through the wire screen used in the washing step. Thus, the lower yield loss for the 70/30 furnish is in the direction expected.

The nature of the fillers and clays probably differ for the newspaper inserts and the magazines. This possible difference can also affect solids losses. The grade of paper used in the

**VII. Ash content<sup>a</sup>**

| Paper sample                       | Ash content,<br>% by weight |
|------------------------------------|-----------------------------|
| <i>Houston Post</i> (no inserts)   | 0.87                        |
| <i>Houston Post</i> (with inserts) | 21.30                       |
| <i>Time</i> magazine               | 27.82                       |
| <i>People</i> magazine             | 22.16                       |
| 70:30 newsprint:magazine           | 8.11                        |
| 49:51 newsprint: magazine          | 13.82                       |

<sup>a</sup>Determined at 575°C using ASTM Method D-482. This method appears to be virtually identical to TAPPI Method T 211 om-85.

newsprint, inserts and magazines also may reflect differences in cellulose fiber properties such as fiber length. This could also affect results.

**Effect of pH.** The results summarized in **Table IX** indicate that an increase in pulping pH from 9.0 to 12.0 in the presence of 1.0% hydrogen peroxide does not decrease handsheet brightness. (The flotation—wash deinking process described in the Testing Procedures was used.) Handsheet image analysis indicated a small particle count increase. However, the handsheet surface area occupied by ink particles was slightly less at the higher pH. This suggested that the higher pH resulted in greater ink dispersion into smaller particles. This is not unreasonable, particularly if the ink contained components that would saponify in the presence of base.

Results summarized in **Table IX** suggest that the increase in pH from 9.0 to 12.0 had little effect on yield loss.

Results for the flotation deinking of a different newsprint suggest that decreasing the flotation pH from 9.0 to 5.5 resulted in a three point brightness increase (**Table X**).

## Testing procedures

### Pilot-scale tests

These tests were performed at Western Michigan University. The scale-up factor from laboratory experiments was 5675.

Pulping was performed in a Black Clawson Hydrapulper. A 70/30 by

VIII. Effect of newsprint:magazine ratio on brightness and yield loss in flotation — wash deinking<sup>a</sup>

| Newsprint:magazine<br>weight ratio | Brightness,<br>% | Yield loss, % by weight |      |       |
|------------------------------------|------------------|-------------------------|------|-------|
|                                    |                  | Flotation               | Wash | Total |
| 49:51 <sup>b,c</sup>               | 54.8             | 23.5                    | 21.1 | 44.3  |
| 70:30 <sup>c,d</sup>               | 55.7             | 28.6                    | 10.5 | 39.0  |

<sup>a</sup>See Testing Procedures for test details.  
<sup>b</sup>Pulp consistency during flotation was 0.32% by weight.  
<sup>c</sup>Ash content of these paper blends is given in Table VII.  
<sup>d</sup>Pulp consistency during flotation was 0.50% by weight.

weight mixture (250 lb) of old newsprint and magazines was added to a stirred 2271 L (600 gal) test surfactant solution. (The magazine fraction actually consisted primarily of old newspaper inserts.) Surfactant concentration was 0.25% surfactant relative to the weight of discarded paper. Solution pH was 9.0 and was obtained by addition of sodium hydroxide solution. Pulp consistency was 5% by weight. Pulping temperature was 110°F. Examination of handsheets suggests that pulping was not quite complete in at least some cases. None of the surfactants exhibited excessive foaming during pulping.

The 5% consistency pulp slurry was transferred to a storage chest where it was diluted to 1% consistency with hot water. The mixture was heated back to the 110°F test temperature. It was then pumped through the 1500-L Voith Morden flotation cell. Flow rate through the cell was about 1363 L/min (360 gal/min). Flotation cell accepts were returned to the storage chest. At a flotation time of 70 min, the pulp slurry passed through the cell an average of about eight times.

The flotation cell rejects, the foam layer containing ink and fiber, was collected. After flotation, the flotation cell accepts were thickened by passing them over a 75 U.S. mesh sidehill screen. The pulp slurry was not washed during this step. Both the accepts and rejects from the sidehill screen were collected.

Two types of paper samples were prepared for brightness determina-

tions: Buchner funnel pulp pads and TAPPI handsheets. There is a washing action in preparing the handsheets that does not occur when preparing the Buchner funnel pads. Solids analysis of the pulp slurries permits yield losses to be calculated.

#### Comparative laboratory scale tests

Paper to be deinked was cut into approximate 1/2-in. squares. Pulping was performed using the same newsprint and magazine blend as employed in the pilot scale tests. The pH of 1000 mL deionized water (110°F) was adjusted to 9.0 using aqueous 10% sodium hydroxide solution. The surfactant, 0.25%w relative to the dry paper, was added as a 1% solution. The paper was added to produce a 5% consistency slurry. This was pulped for 20 min in a TAPPI disintegrator.

The pulp slurry was divided into two equal portions. The first was diluted to 1% consistency in 110°F deionized water and transferred to a 2500 mL Denver laboratory flotation cell. Flotation time was 3 min at a rotor speed of 1200 rpm. The foam was collected and saved. The second portion of pulp was treated in a similar manner except that the flotation time was 6 min.

After flotation, the pulp slurry was poured over a 100 mesh screen. The fluid was allowed to drain through the screen until about 2300 to 2400 mL is collected. The remaining pulp was transferred to a beaker.

#### Flotation—wash deinking procedure

Pulping was performed at 110°F for 12 min at a consistency of 4% by weight. The procedure was the same as described above except that 1.0% hydrogen peroxide, 0.2% diethylenetriaminepentaacetic acid, and 0.2% surfactant were present in some tests as noted in appropriate data tables and figures. (All percentages are by weight relative to dry furnish.)

Flotation was performed as described above for 6 min at 0.32% consistency and 110°F. The froth layer was collected and saved.

A 30 U.S. mesh screen was placed over a large clean plastic dish pan. The flotation slurry was poured over the screen. The pulp collected on the screen while much of the water drained through. The pulp was pressed against the screen to remove liquid. No additional water was used to wash the pulp.

#### Handsheet preparation

Deionized water is added to a slurry volume of 800 mL. This slurry is divided into four equal portions. Handsheets are prepared from each using both the TAPPI handsheet maker and a Buchner funnel procedure.

#### Brightness and image analysis

Brightness was determined using a Technidyne S-4 brightness meter at both Westhollow Research Center and Western Michigan University (WMU). Image analysis was performed at WRC using a Tracor Northern 8502/C Image Analyzer located in the Analytical Department.

#### Yield loss

**Flotation.** The froth that collected during flotation was vacuum-filtered using tared Whatman No. 40 (8 micron) filter paper. The filter paper was dried and weighed to determine fiber loss.

**Washing.** The fluid that collected in the dishpan was saved. The fluid that collected in the dishpan was vacuum-filtered using tared Whatman

## IX. Effect of pulping pH on deinking in a flotation — wash process<sup>a</sup>

| Pulping pH | Deinked sheet brightness, % | Image analysis   |                       | Yield loss, % by weight |      |       |
|------------|-----------------------------|------------------|-----------------------|-------------------------|------|-------|
|            |                             | No. of particles | Ink surface area, ppm | Flotation               | Wash | Total |
| 12.0       | 54.8                        | 238              | 182                   | 23.5                    | 21.1 | 44.6  |
| 9.0        | 53.9                        | 278              | 129                   | 21.9                    | 20.0 | 41.9  |

<sup>a</sup>Test procedures are summarized in the Testing Procedures section. The deinking surfactant concentration was 0.2% (relative to furnish) SDA-5. The furnish was 70% *The Houston Post*:30% magazines. Hydrogen peroxide (1.0% relative to furnish) and diethylenetriaminepentaacetic acid (0.2% by weight relative to furnish) were present during pulping.

No. 40 filter paper. The filter paper was dried and weighed to determine yield loss.

## Conclusions

Laboratory flotation deinking brightness values in WRC tests scaled-up well in the pilot-scale tests. However, yield loss values in laboratory Denver cell and Western Michigan University pilot-scale flotation deinking tests showed significant differences. Both test procedures and furnish variability could have caused this. No definitive conclusions could be drawn concerning relevance of laboratory yield loss data to pilot-scale results.

Product handsheet brightness increased with decreasing flotation cell consistency of a newsprint/magazine furnish in a flotation—wash process. As the flotation step pulp consistency was decreased, both flotation step yield loss and total yield loss increased. Handsheet brightness did not increase with increased pulping consistency within the limited consistency range studied. Observed flotation step and total yield loss increases at higher pulping consistency probably were not experimentally significant.

Increased ash content in the discarded paper resulted in increased total yield losses in a laboratory flotation—wash deinking process.

Use of smaller mesh sizes in the screen used in the wash deinking step did not result in reduced yield losses over the range of mesh sizes studied.

## X. Effect of flotation step pH on flotation deinking of newsprint<sup>a</sup>

| Deinking surfactant | Flotation pH | Deinked sheet brightness, % | Yield loss, % |
|---------------------|--------------|-----------------------------|---------------|
| SDA-6               | 9.0          | 50                          | 12.2          |
| SDA-6               | 5.5          | 52                          | 13.2          |
| SDA-7               | 9.0          | 47                          | 7.8           |
| SDA-7               | 5.5          | 49                          | 4.7           |

<sup>a</sup>Test procedures are described in the Testing Procedures section. Surfactant concentration was 0.2% by weight relative to furnish. Pulping was performed using a Hamilton Beach blender at pH 9.0. Flotation step pulp consistency was 0.32% by weight. The newspaper studied was the *Atlanta Journal and Constitution*.

Increasing the pulping pH from 9.0 to 12.0 in the presence of hydrogen peroxide did not significantly alter handsheet brightness in a flotation wash deinking process. In the absence of hydrogen peroxide, decreasing the pH from 9.0 to 5.5 in flotation increased sheet brightness. **□**

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