

Separation of fiber and ash in deinking effluents: A case study

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ABSTRACT: *Sludge disposal remains a problem for mills that recycle post-consumer waste. This report evaluates the feasibility of reducing sludge generation by removing suspended solids from spent deinking liquor. One-stage screening removed 35% of suspended solids from the deinking liquor, while a lower-cost experimental air-flotation technique removed 33%. Ash content of the retained solids was about 24%. Resuspension and rescreening of the screened solids reduced the ash content to 16%. Repeating this cycle reduced the ash to 6%. The collected solids were high in fiber content, had good papermaking characteristics, and could be reused in a mill furnish. The filtrate had a higher ash content and thus would be more amenable to land disposal. Computer analyses of four experimental techniques for separating fiber and ash from spent deinking liquor are presented.*

KEYWORDS: *Ash content, deinking, effluents, fibers, filtration, flotation, screening, separation, simulation, sludge disposal, spent liquors, suspended solids.*

The objective of the deinking process is to separate inks and fillers from the fibers in recycled paper. The mechanisms involved include saponification of binders, abrasion of filler from the fiber surface, and dispersion of ink (1). Separation and emulsification of ink particles are facilitated through the use of such pulping reagents as so-

dium hydroxide, detergents, and solvents.

Spent deinking liquor is often partially recycled to the pulper to save heat and chemicals. The remaining spent liquor is usually combined with the mill effluent and treated by alum coagulation and sedimentation to remove suspended solids before discharge. The sludge produced by this

treatment is typically landfilled. However, with landfill space becoming increasingly scarce, efforts have been made to reuse the deinking and high-ash-content paper-mill sludges in the manufacture of linerboard and molded fiber articles (2, 3), composite building products (4–6), cement (4, 7, 8), plastics (5, 9), rigid foams and packing (10, 11), and as an admixture to concrete (12–14).

Direct reuse of the sludge in paper products was found feasible at several board mills where the cleanliness of the furnish was not critical. A board mill in Germany reported recycling primary sludge (ash content of 30%) without adverse effects on paper-machine operation or product quality (15). A U.S. paperboard mill also reported acceptable results for additions of waste sludge to the furnish (16).

A study on filler recovery by sludge incineration (17) indicated that the brightness of the recovered filler is satisfactory. Filler recovery by wet-air oxidation (Zimpro) was practiced at a U.S. coated-paper mill (18), and the recovered filler was reused in selected paper grades.

The focus of this study was a tissue mill using secondary fiber as the main furnish. The mill was landfilling 100 tons/day of thickened deinking sludge (25–30% solids with an ash content of 50%). Limited landfill space compelled the mill to seek an alternative method of sludge disposal.

The first step was to locate prospective users of the sludge. Batches of thickened sludge (500–1000 kg,

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Deinking

wet weight) were sent to several manufacturers of paper products (linerboard, chip-board, molded products, and roofing-paper mills) to test the feasibility of applying the sludge in their furnishes. The major obstacle to direct reuse of the sludge was its high ash content. Sludge pre-treatment to decrease the ash and increase consistency was deemed necessary to (a) minimize the pollution-control requirements at the recipient mills and (b) decrease the transportation costs.

This paper describes laboratory work that was carried out to evaluate techniques for separating the fiber and clay components of the recycled deinking liquor. Separation was accomplished through a combination of screening, modified dispersed-air flotation, and centrifugation. It was assumed that the fiber fraction could be reused in the tissue mill's furnish, although further studies are needed to confirm the feasibility of such an approach. The clay fraction was still to be landfilled. Because of its increased ash content, the clay fraction would likely be more biologically stable and thus acceptable for landfill.

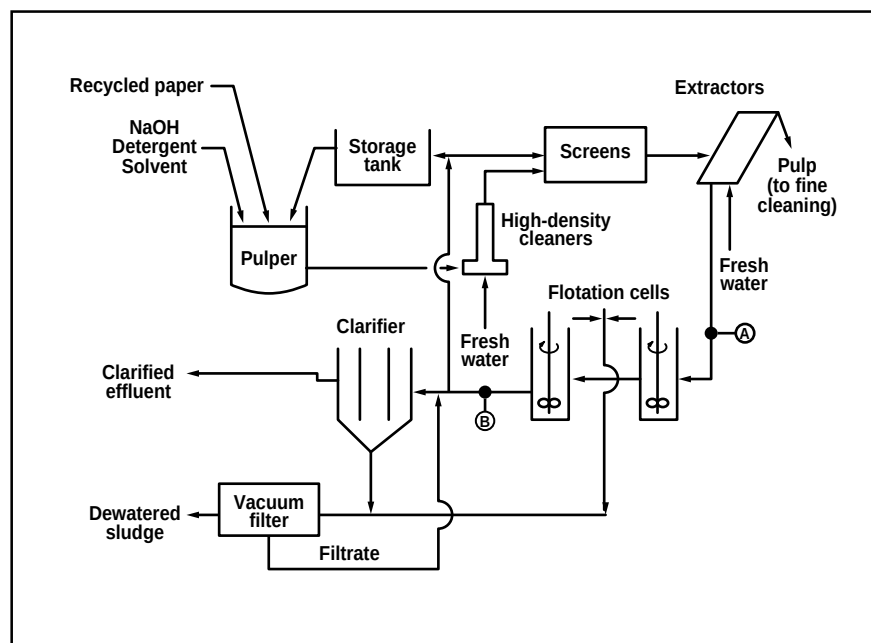
The study was performed with effluents from a deinking operation that has since been discontinued. However, the assessment of separation processes should be of interest to modern deinking mills facing the sludge-disposal problem.

Experimental

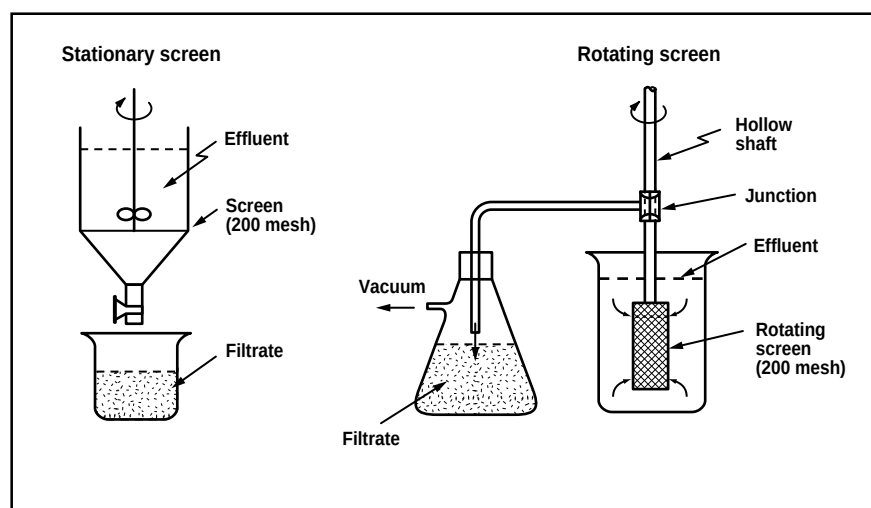
Process-water system

The process-water system in the tissue mill is illustrated in **Fig. 1**. The batch pulper processed about 107 oven-dried (o.d.) tons/day of white ledger and lightly printed uncoated paper. Pulping was carried out at 90°C and 5.5% consistency. The charges of NaOH, detergents, and solvents were 2%, 0.4%, and 0–0.25%, respectively (19). Ink was separated from the fiber by washing and then by dewatering on screen extractors.

1. Flow chart for deinking operation at tissue mill (A and B represent sampling points on either side of the flotation system)



2. Stationary and rotating laboratory screens

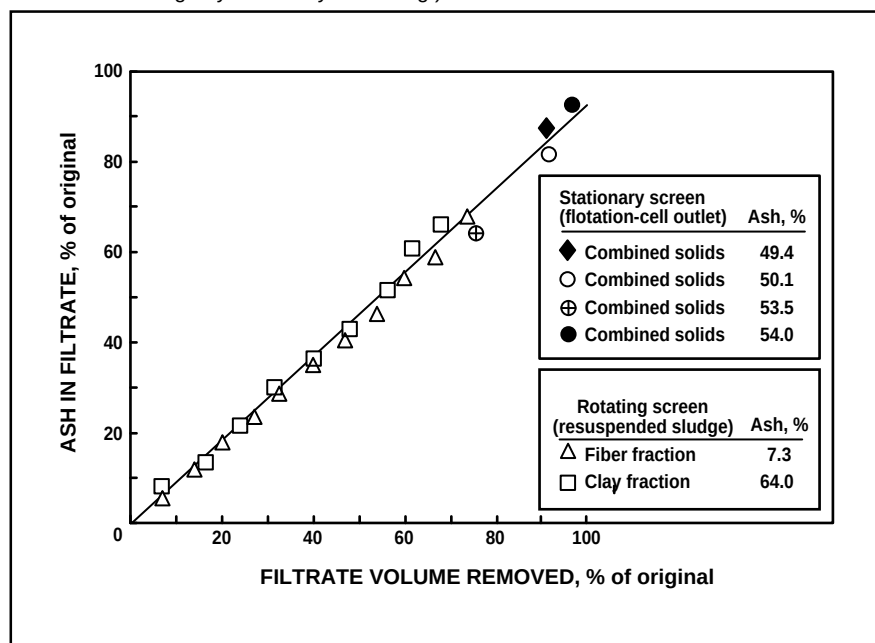


Ink was removed from the spent pulping liquor via dispersed-air flotation. The combined mill effluent was treated by coagulation with alum and polyelectrolyte followed by primary settling. The clarifier underflow had a consistency of about 5% and was further thickened to 25–30% using a vacuum belt filter.

Fresh water was added at the centrifugal cleaners and the extractor

showers. This diluted the spent pulping liquor (i.e., flotation-cell effluent) before recycle to the pulper. However, because suspended solids were not removed from the recycled liquor, large amounts of dead-load solids [about 33% (by weight) of the charge of recycled paper] were recycled to the pulper.

3. Ash separation on the stationary and rotating screens as a function of the filtrate volume removed. (The fiber and clay fractions used with the rotating screen were previously isolated from the mill sludge by stationary screening.)



I. Physical characteristics of secondary fiber as produced at the mill and fiber recovered from mill effluent by laboratory screening

Characteristics	Secondary fiber ^a	Fiber recovered from effluent ^b
Freeness, mL	204	134
Bulk, cm ³ /g	1.75	1.74
Burst index, kPa·m ² /g	3.12	2.64
Tear index, mN·m ² /g	8.80	6.86
Breaking length, km	4.92	4.45
Stretch, %	3.14	2.85
Brightness, % ISO	69.2	59.5
Zero-span breaking length (Pulmac), km	14.33	13.15

^a Mill-washed secondary fiber (unbleached)
^b Fiber recovered from flotation-cell effluent by one-stage screening (200-mesh) in the laboratory (ash content 24%)

Screening tests

A stationary screen and a rotating screen were tested in the laboratory. Both screens are illustrated in **Fig. 2**.

The stationary screen consisted of a stainless-steel funnel, a drainage valve, and a mixer. Typically, 1.5 L of spent pulping liquor (sample from Point B in Fig. 1, i.e., flotation-cell outlet) or resuspended sludge from the vacuum filter was placed in the funnel with the drainage valve

closed. After 5 min of mixing at 200 rpm, the drainage valve was opened and filtrate was collected for analyses.

A laboratory rotating screen was used to control mat formation during screening. The cylindrical screen (2.5-cm diameter, 10-cm high) was placed into a sample of resuspended sludge from the vacuum filter and rotated at 500 rpm. Filtrate exited the rotating screen under vacuum suction (typically 50 kPa) through

small holes drilled in the hollow drive shaft. The screened filtrate was collected in a separate flask.

Modified dispersed-air flotation

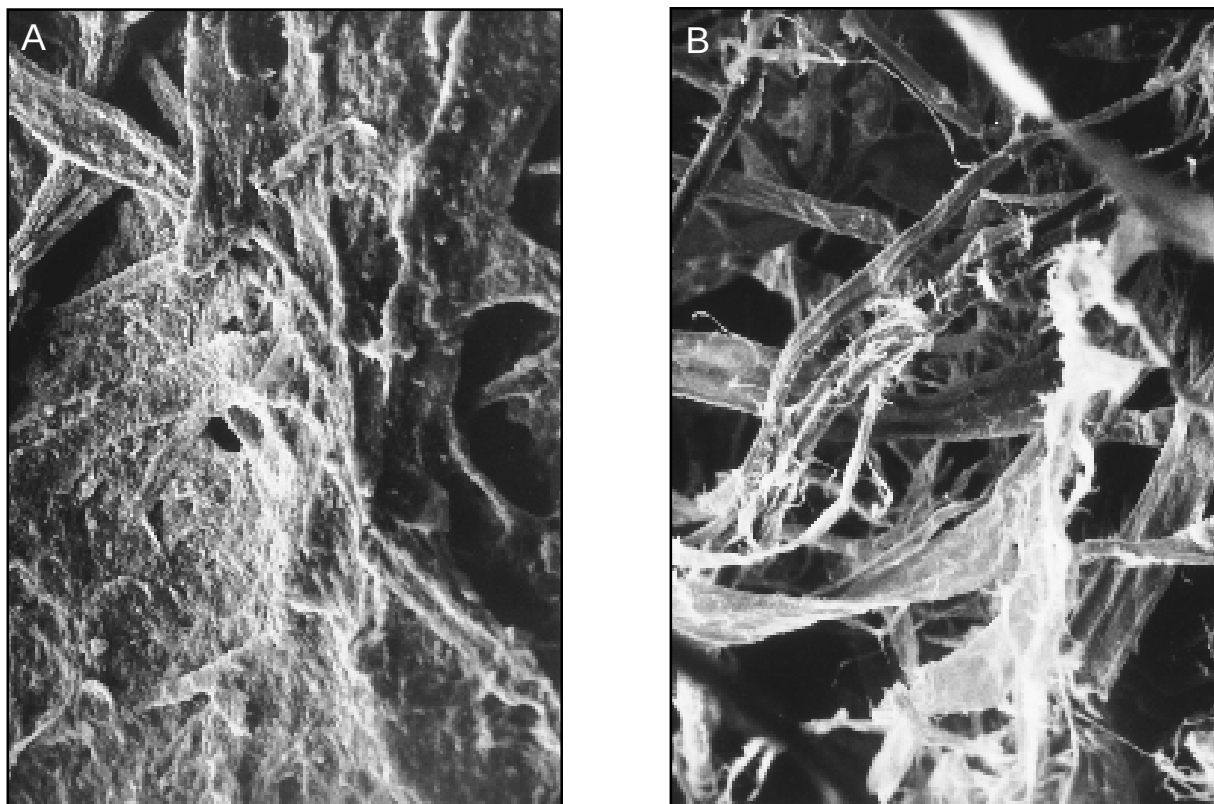
One liter of spent pulping liquor taken from flotation-cell influent or effluent (sampling points A or B, respectively, in Fig. 1) was placed into a beaker and agitated for 2 min at 500–3000 rpm using a Brookfield mixer. During the mixing, small air bubbles were entrapped in the effluent, and foaming occurred. The size of air bubbles was not measured, but it was speculated that higher mixing speeds would produce smaller air bubbles and enhance emulsification of the residual detergents. Within one minute after turning off the mixer, the fiber present in the effluent floated and formed a thick layer. Most of the foam and the floated fiber were collected by skimming with a perforated spoon.

The modified dispersed-air flotation, as used in our study, is not normally practiced. The objective of conventional flotation is to separate ink by foaming, so the flotation cells are maintained under high turbulence. Under those conditions, fiber and clay cannot float to the surface. The floating tendency of deinking solids probably results from the use of surface-active agents in the pulper. Floating of solids, including clay, on the level of the secondary clarifier has also been observed with biologically treated deinking effluents (20). In our approach, the floating tendency would be utilized directly in the deinking process to decrease the load of suspended solids to the pulper.

Analyses

Suspended solids and ash were analyzed using CPPA Standard Method H.1. The filtration rates were often very low, and filtration had to be repeated several times to obtain clear filtrate. Consequently, the total solids content was occasionally used as a measure of separation efficiency.

4. Scanning electron micrographs of solids isolated from flotation-cell outlet (sample obtained from point B). A: Solids isolated by settling. B: Clean fiber after five consecutive washings.



Results and discussion

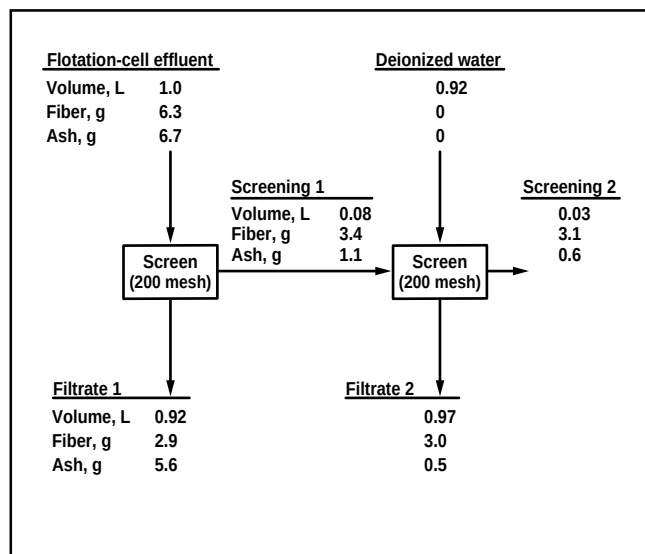
Fiber/ash separation by screening

Screening with a 200-mesh screen preferentially retained fiber and permitted the passage of ash. The results with respect to ash separation are shown in **Fig. 3**. Upon screening—using either a rotary or a stationary screen—the fraction of ash in the filtrate was equal to the fraction of liquor ending up as filtrate. This finding implies that the screens did not retain the clay particles to any significant extent.

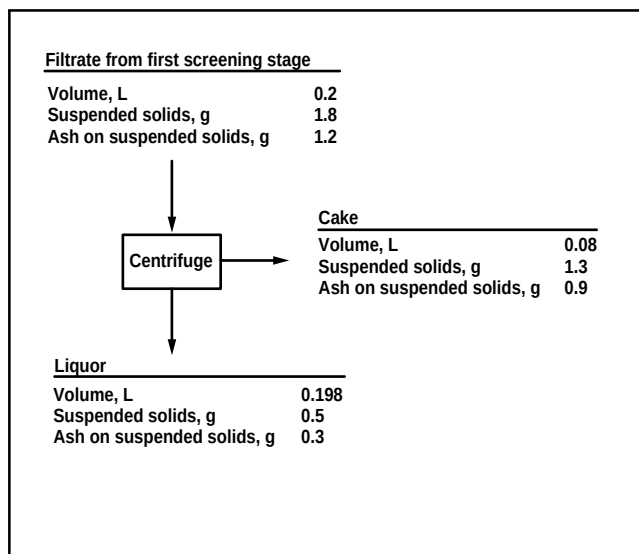
Figure 4 shows scanning electron micrographs of solids isolated from flotation-cell effluent. Figure 4A shows pulp fibers covered by layers of clay after settling. Because of the amounts of clay present, it is unlikely that, in suspension, the bulk of the clay particles was physically associated with the fibers (21). The suspended clay particles not associated with fibers were expected to pass freely to the filtrate. Figure 4B shows clean fiber after five consecutive washings.

Two-stage screening was performed in a separate experiment. Fibrous solids retained on the first screen were resuspended in the same initial volume of deionized water and then screened again. The results in **Fig. 5** show that about 84% of the ash introduced on the first screen passed into the filtrate. The breakthrough of the fibrous component was only 46%, so the ash in the filtrate increased from its initial level of 51% to 66% in the filtrate from the first-stage screening. In the subsequent second-stage screenings, a

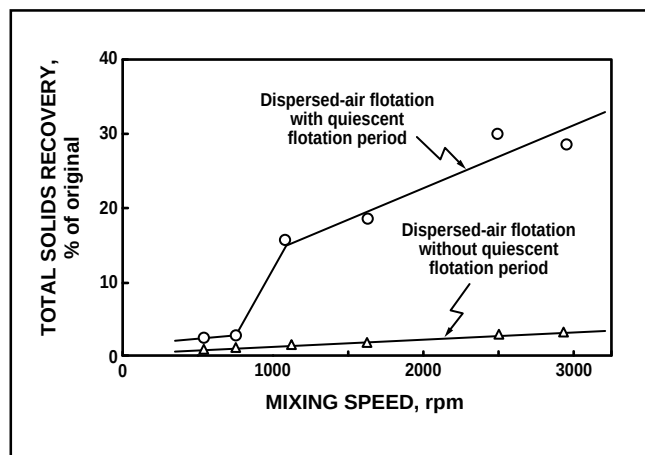
5. Mass balance of suspended solids and ash for two-stage screening using 200-mesh stationary screen (sample obtained from point B—flotation-cell outlet)



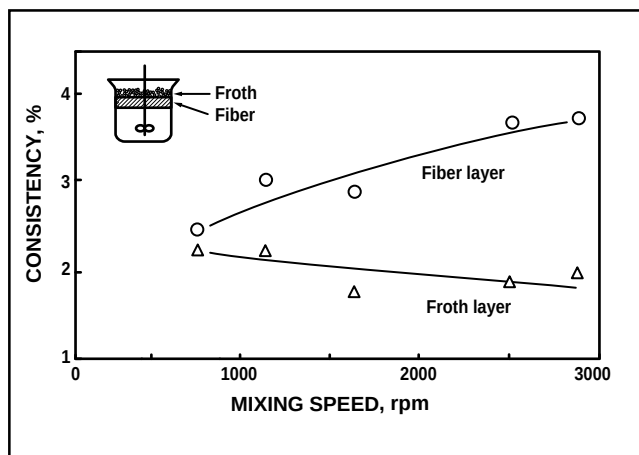
6. Mass balance of suspended solids and ash after centrifuging (1500 rpm, 10 min) filtrate from first-stage screening (sample obtained from point B—flotation-cell outlet)



7. Solids recovery as a function of mixing speed for air flotation with and without a quiescent period. (Solids recovered during quiescent period include both the ink froth and fiber layers.)



8. Consistency of suspended solids as a function of mixing speed for the froth and fiber fractions separated by modified flotation



lesser amount of ash (45%) was separated from the pulp retained on the screen. About half of the fibrous solids in the flotation-cell effluent was recovered after two-stage screening. The ash content of the combined solids (fiber plus ash) was 24% after the first screen and 16% after the second screen.

The number of screening stages needed to clean the fibrous fraction will be dictated by the ash-content requirement of the recovered pulp. With the sample of flotation-cell effluent tested, the pulp retained on

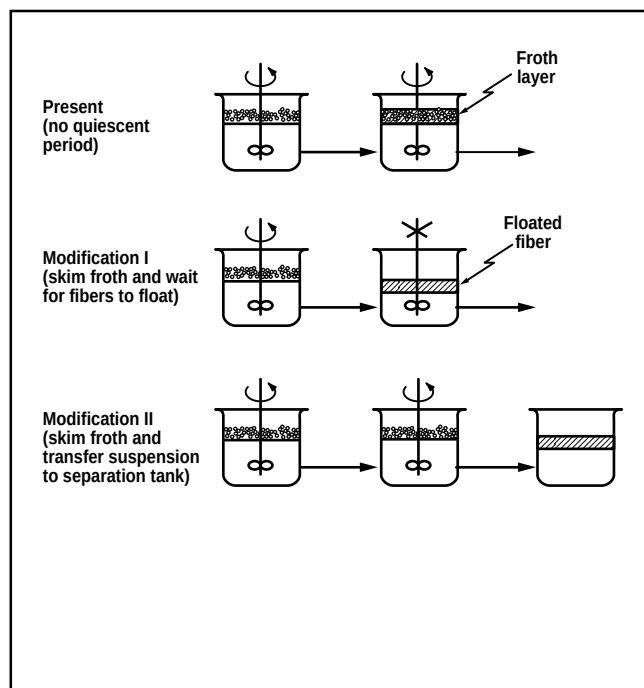
the screen after three successive screenings contained 5.7% ash.

The rotating screen—tested with two diluted suspensions (i.e., the fiber and the clay fractions isolated previously from the thickened mill sludge by screening)—gave ash separation comparable with that of the stationary screen, as illustrated in Fig. 3. There was no visible mat formation observed in experiments with the rotating screen. The similarity of results obtained with both screens suggests that the mat formed on the surface of the station-

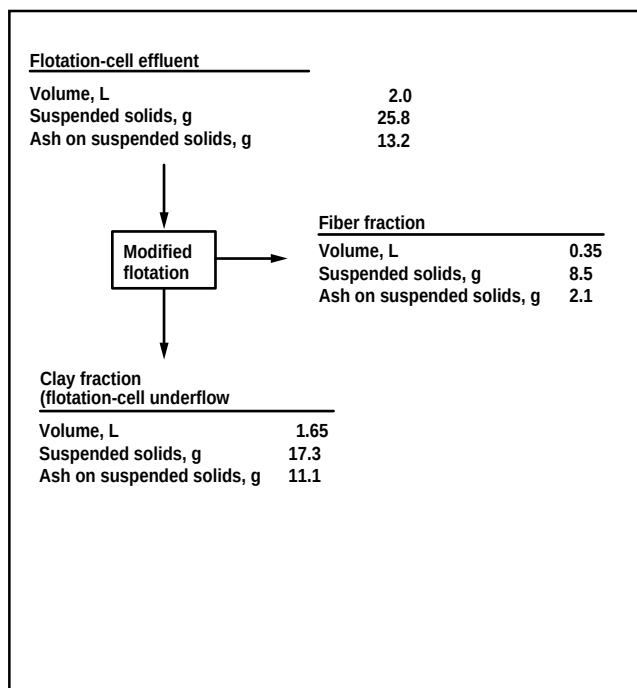
ary screen did not impede the passage of clay into the filtrate.

The fiber separated by screening might be suitable for use in a lower grade (commercial) tissue furnish. **Table I** compares the physical characteristics of the fiber recovered from the effluent (one-stage screening) and the secondary fiber produced at the mill. The fibers screened from the spent deinking liquor had only slightly inferior properties. The lower strength, freeness, and brightness of the screened fiber were probably caused by the high ash content

9. Process modifications for collecting fiber layer during a quiescent-flotation phase following dispersed-air flotation



10. Mass balance of suspended solids and ash for modified dispersed-air flotation (sample obtained from point B—flotation-cell outlet)



(24%). Based on the results in Fig. 5, two-stage screening would decrease the fiber ash content to about 16%. In practical application, the number of screening stages could be adjusted to assure that the quality of the screened fiber was sufficient for re-use in tissue production. Based on deinked-pulp tonnage of 77 air-dried (a.d.) tons/day (ash content 2.2%) and a 3% increase in pulp yield from recycling the screened fiber (ash content 16%), we estimate that the ash in the tissue furnish would increase from 2.2% to 2.5%.

To further decrease the suspended-solids content of the recycled spent liquor, we centrifuged the filtrate of the one-stage screen using a laboratory centrifuge. As seen in Fig. 6, the treatment removed large amounts of suspended solids (72%) and ash (75%).

Ash separation by dispersed-air flotation

In the flotation cells at the tissue mill, turbine agitation produces the small air bubbles needed to float ink

particles to the surface. This technique removes only modest amounts of solids. Our results show that only 1–3% of the total solids were floated into the ink foam. Jasmin (19) reported that the removal of suspended solids in mill flotation cells rarely exceeds 8%.

To increase the solids separation, we modified the flotation technique by introducing a short (1–2 min) quiescent period immediately after agitation. During the quiescent period, a compact layer of fibers and clay formed on the top of the liquid phase. Most of the fibers were floated by air bubbles that had attached themselves to the fiber surface. Floated solids were collected with a perforated spoon.

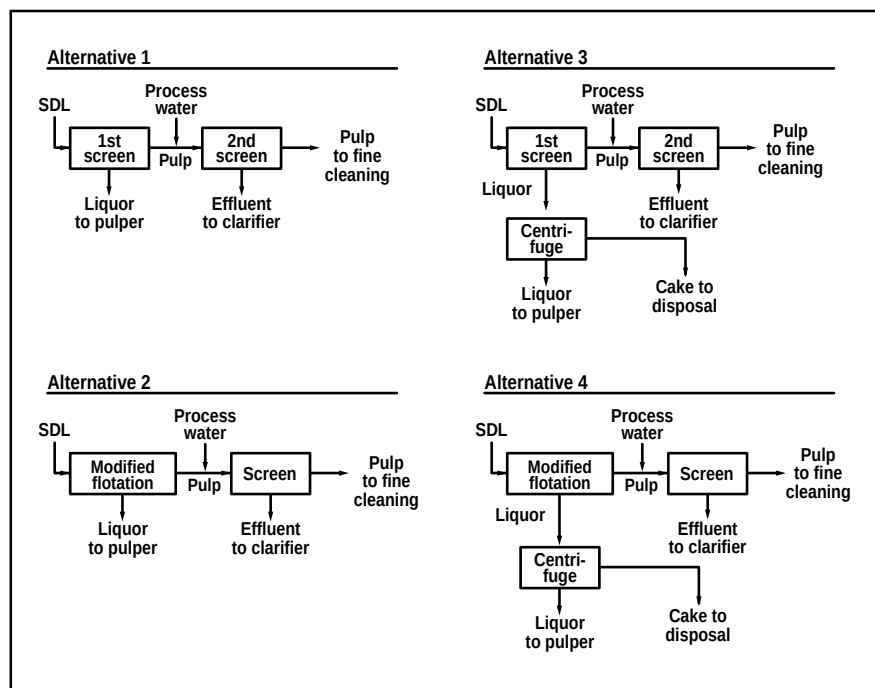
The mixing speed during the agitation phase had a strong effect on solids separation in the ensuing quiescent period, as seen in Fig. 7. Total solids recovery between 27% and 31% was achieved at mixing speeds between 2500 rpm and 3000 rpm. In contrast, the conventional flotation without a quiescent period removed only 2–3% of total solids under iden-

tical mixing conditions. It is possible that lower mixing velocities might be satisfactory in full-scale flotation units equipped with injection of compressed air through the turbine shaft.

The effect of mixing speed on the consistency of the froth and fiber layers is shown in Fig. 8. In these tests, the ink froth was skimmed during the mixing. The fiber was removed similarly after the formation of the fiber layer during the quiescent period. The reproducibility of this technique was $\pm 5\%$, by weight. This method of solids collection is representative of a two-stage flotation, where the ink is floated during the first-stage agitation and the fiber is floated in the second quiescent stage.

At the mill, the flotation units operated without a quiescent period. This could be converted to the modified method using either of the two techniques illustrated in Fig. 9. The second flotation cell could be operated without agitation (Modification I), or a separation tank could be installed after the last flotation cell to float the fiber (Modification II).

11. Four treatment alternatives for recycled deinking liquor (SDL = spent deinking liquor)



The mass balance of solids for flotation-cell effluent is shown in **Fig. 10**. The recoveries of suspended solids and ash in the floated layer, based on the original content in the diluted spent liquor (flotation-cell effluent), were 33% and 16%, respectively. These results compare favorably with the suspended solids and ash retention of 35% and of 16% that were achieved with a one-stage 200-mesh screen (Fig. 5). We expect that the capital cost of modifying the dispersed-air flotation unit would be lower than a screen installation. In particular, it would be advantageous to collect the fibers in the last section of the flotation cells (Modification I in Fig. 9), thus avoiding purchase of a separation tank. This approach is reasonable if the ink separation is not affected by the shorter flotation times.

Computer assessment of process modifications

Based on the present work, four alternatives are suggested for separating the fiber and ash components

in spent deinking liquor. The four options, illustrated in **Fig. 11**, are as follows:

1. Two-stage screening and recycle of the filtrate from the first screen to the pulper. The fiber retained on the second screen is routed to the pulp fine-cleaning department (or to fiber furnish). Filtrate from the second screen is sewerred.
2. Modification of the flotation process by introducing a quiescent stage in the last flotation cell, with subsequent screening of the collected fiber to decrease the ash content. The fiber recovered is used as in Alternative 1. Liquor from the modified flotation is recycled to the pulper. Filtrate from the screen is sewerred.
3. Two-stage screening (as per Alternative 1) with centrifugation of the liquor from the first screen. The fiber retained by the second screen is used as in Alternative 1. Liquor from centrifugation is recycled to the pulper. The centrifugation cake is landfilled.

4. Modified flotation and screening of the fiber fraction (as per Alternative 2), with centrifugation of the underflow from the modified flotation cell. The fiber recovered is used as in Alternative 1. Liquor from centrifugation is recycled to the pulper. The centrifugation cake is landfilled.

To assess these process modifications, a GEMS* computer model was constructed for the existing and suggested process configurations. The GEMS-derived process variables for the existing case agreed with the mill data within $\pm 1\%$. The solids-separation efficiencies of the proposed additional unit operations (screening, centrifugation, and modified flotation) were based on our experimental results, as shown in Figs. 5, 6, and 10, respectively.

Figure 12 shows the simulated results for the steady-state recycle rates of ash and suspended solids to the pulper for the four process alternatives. The figure also shows fiber yields and pulping capacities for the four process alternatives. A small increase in fiber yield (2–3%) was obtained in the alternatives excluding centrifugation (Alternatives 1 and 2). However, the alternatives utilizing centrifugation (Alternatives 3 and 4) showed a decrease in yield (up to 4.4%). This was caused by withdrawal of a small portion of the fiber from the system with the centrifuge cake. In the computer simulation of the existing operation, all fibers were assumed to be returned to the pulper with the recycled liquor and recovered as pulp. However, the significantly lower input of suspended solids to the pulper would allow for a proportionally higher charge feed of recycled paper. We estimate that the mill's pulping capacity could be increased 13–27%, depending on the process alternative selected.

*General Energy and Material Balance System: A Modular Computer System for Pulp and Paper Applications." Developed by L. L. Edwards and Associates at the University of Idaho, Moscow, ID.

Deinking

The steady-state ash contents of clay fractions resulting from our process modifications ranged between 79% and 85%, compared with existing sludge ash content of 50%. It should be noted that, in practice, such high-ash sludge would be available for disposal only in Alternatives 3 and 4, in the form of centrifugation cake. The diluted clay fractions (i.e., "effluent to clarifier" in Fig. 11) will likely be combined with the remaining deinking effluents and paper-machine white water and treated with alum followed by sedimentation. The ash content of the resulting sludge will depend on ash and fiber load in the latter effluent streams.

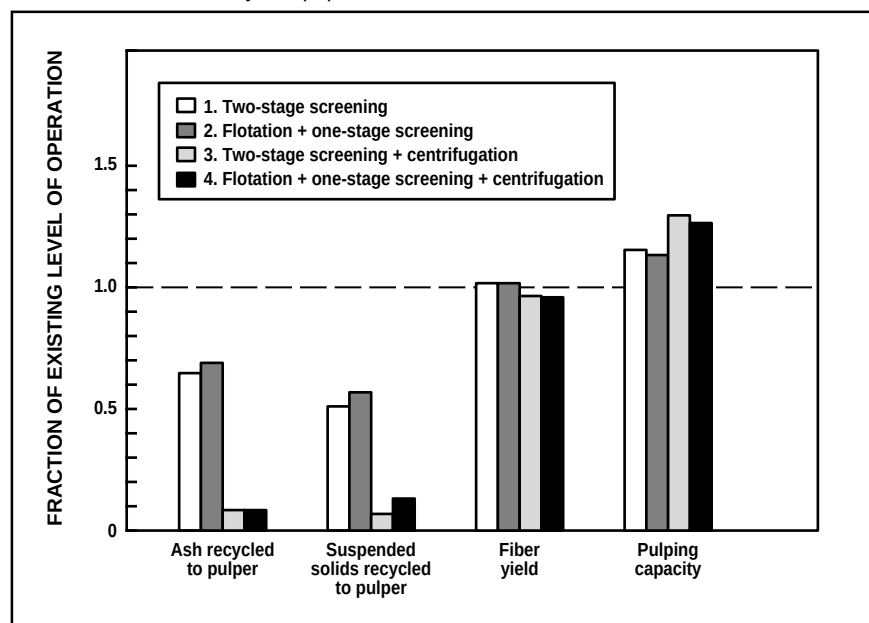
Given the small experimental scale, there was no attempt to assess the economic feasibility of the approaches tested. Continuous pilot-plant studies would be needed to assess the merits of the various solids-separation alternatives.

Conclusions

Methods of separating ash and fibers in spent deinking liquor were evaluated in the laboratory. The results showed that

1. One-stage screening of spent deinking liquor produced fiber with an ash content of 24%, much lower than the ash content of 49–54% in the initial spent deinking liquor. Ash content was decreased to 16% by resuspension and re-screening of the fiber. An additional round of resuspension and rescreening further reduced the ash content to 6%.
2. A modified dispersed-air flotation technique had the same separation efficiency as one-stage screening. Capital requirements for this alternative should be less than installation of a separate screening stage.
3. Centrifugation of the clay fraction from one-stage screening removed 72% of suspended solids and 75% of ash.

12. GEMS results for four process alternatives. Fiber yield (unbleached, after extractors) is based on the feed of recycled paper.



4. The solids-separation efficiencies of four process modifications were assessed using the GEMS process-simulation program. Calculations indicate that the recycle to the pulper of both suspended solids and ash could be decreased by 31–93%, depending on the process alternatives used. The corresponding increase in the pulping capacity was estimated to be 13–27%.
5. Pilot-plant studies would be needed to assess the technical and economic feasibility of the solids-separation methods studied. ■

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