A novel flotation deinking chemistry to remove flexo inks: 30 tons/day pilot-plant results

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ABSTRACT

Current recycling processes typically incorporate aggressive washing stages to successfully remove flexographic, or flexo, inks. The yield losses associated with such washing stages are often economically unacceptable. A goal for many old newsprint (ONP) deinking mills is to be able to efficiently deink ONP containing varying, moderate levels of flexo inks in the flotation deinking stage. Deinking systems utilizing only flotation cannot handle more than 3–5% flexo ONP content.

This paper discusses two flexo flotation deinking trials utilizing a novel organoclay chemistry in a 30 tons/day continuous-process pilot plant. Flexo ONP content was 25% and 30% in each of the two trial runs. The trials were performed with closed thickener filtrate loops without process water clarification. Brightness exceeded 60 points at the end of the process that involved predispersion flotation, conventional dispersion peroxide bleaching, postflotation, and thickening.

Application:

The organoclay chemistry is a viable technology for flexographic ink and ash removal in flotation-only deinking systems.

THE DEINKING PROCESS IS INVARIABLY a compromise of economics and deinking efficiency. Some residual ink particles are always present in deinked pulp (DIP). This is particularly true when the deinking process is not properly designed for handling a particular furnish or ink. For example, a deinking plant using a flotation process without incorporating aggressive washing stages is ill-equipped to deink recovered paper furnish containing flexo ink print [(as found in old newspapers (ONP)]. The fine flexo ink particles, when not efficiently removed by flotation, can cause operational difficulties in the paper mill using the resulting DIP.

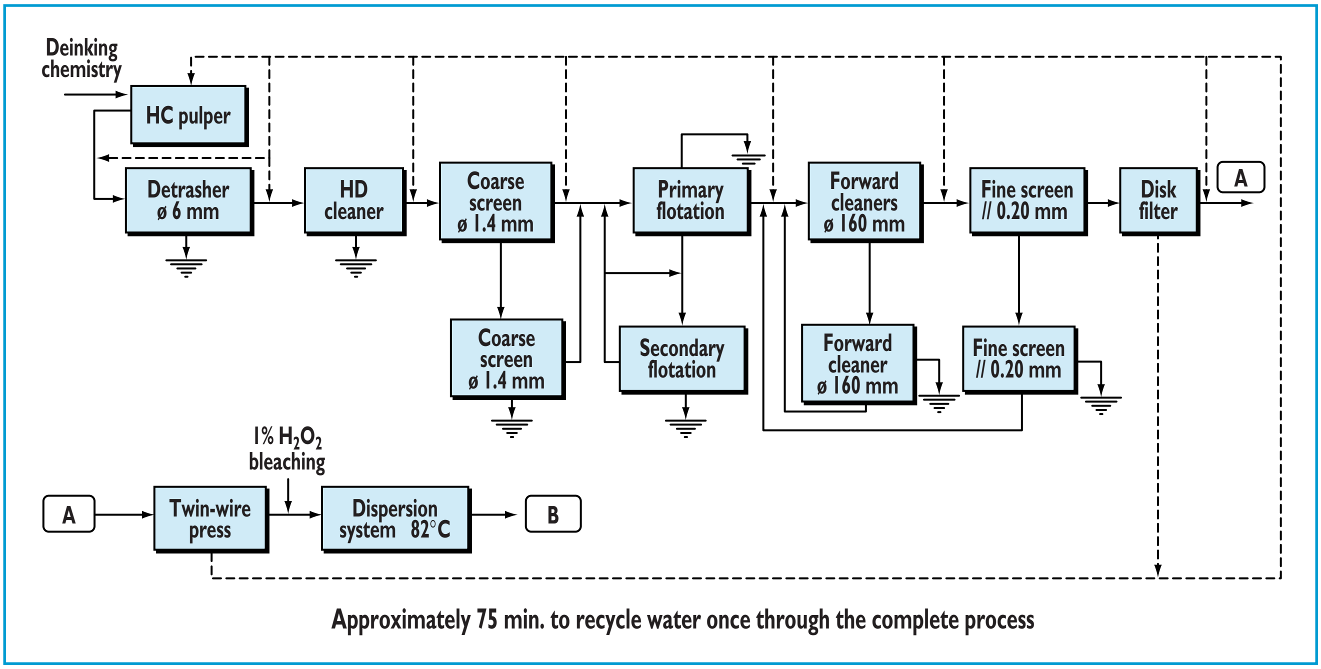
Mill experience has shown that residual ink can build up in the paper machine white water loop, causing a significant brightness drop at the reel. The residual ink, if complexed with stickies or pitch, can become black deposits appearing on the wire, felts, press, and dryer rolls. These deposits can cause dirt spots on the paper products, and in severe cases, cause frequent machine breaks.

A number of chemical strategies have been proposed for handling ONP furnishes containing flexographic inks levels above of 5–10%. While traditional collector-type surfactants are effective with hydrophobic inks, and laboratory studies have demonstrated that calcium soaps can remove flexo in the absence of fiber *(1),* these chemistries have not found success in handling higher levels of flexo on a commercial scale *(2).* In fact, the limit of 5–10% flexo levels has been determined empirically in several mills that are using both fatty acid and synthetic collector chemistries. Anecdotal evidence abounds in many ONP recycling mills regarding sudden system upsets associated with furnishes containing flexo ink. In some mills, the issue is so severe that even problems unrelated to flexo ink may nevertheless be attributed to it.

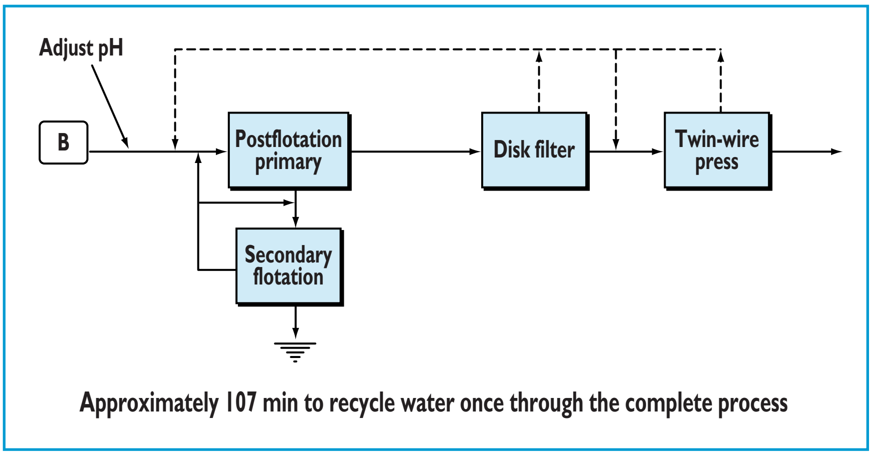
Cationic polymers, traditionally used in the process water clarification, may be added to a pulp stream containing dispersed flexo inks; however, such polymers are relatively indiscriminate in collecting anionic material and may result in unacceptable yield losses across the flotation stage *(3).* Even in the clarification systems associated with the washing stages of an ONP deinking system, significant challenges exist in achieving sufficient water quality using traditional cationic polymer chemistries *(4).*

A well-known characteristic of flexo inks is that they disperse into extremely stable suspensions under alkaline conditions. Research into the stabilization mechanisms of water-based inks suggests that both electrostatic and steric forces are contributing factors *(5, 6).* An alternative approach designed to counter these effects has been proposed by Galland and Vernac, who describe a flotation process involving pulping and floating at near-neutral pH *(7);* however, the resulting partially deinked pulp must still be processed through a complete traditional deinking system. Matzke *et al.* (8) suggested in their patent "washer filtrate flotation" to maximize the yield of a washing/flotation deinking process. The floated filtrate is mixed with washed pulp and then processed in an additional flotation step. This process sequence removes flexo inks with conventional collector-type chemistry. Yet another approach that has been suggested involves ultrafiltration of process water *(9).*

Our work involves pilot-plant trials that have been conducted using an organically modified smectite clay (organoclay) as a collector for both



1. Alkaline loop



2. Postflotation acid or neutral loop

Trial	Flexo ONP	ONP	OMG
1	25% Newspaper: Modesto Bee, Sacramento, CA 4 weeks old	45% Source: Midwest (Appleton, WI) 2 weeks old	30% Higher UV content than Trial 2, telephone books included
2	30% Newspaper: San Francisco Examiner, San Francisco Chronicle, 4 weeks old	40% Source: Midwest (Appleton, WI) 2 weeks old	30% -

I. Recovered paper furnish for each trial

the finely divided flexographic inks as well as the traditional, more hydrophobic newsprint inks. The composition and deinking mechanism of the organoclay material used have been described elsewhere *(10, 11)*. This approach to flotation removal of water-based inks, including flexo inks, involves the collection of the ink, including the submicron-sized flexo particles, onto an organically modified clay particle surface. The resulting clay–ink complex is hydrophobic and quite amenable to removal in the flotation stage.

An unexpected benefit observed in these particular trials was a much higher ash removal across flotation than is achieved with traditional collector chemistry. Using either fatty acid or synthetic collectors, ash removal rates of 10–20% (expressed as a weight percentage of ash in the feed) are typical. In the evaluations described herein, ash removals of 66–80% were achieved.

EXPERIMENTAL METHODS

One objective of the process trial was to demonstrate that flexo inks did not build up in the thickener filtrate loops, causing final recycled pulp brightness to drop with time. Many laboratory experiments have demonstrated successful deinking of flexo inks, but these efforts fail when tried on a commercial scale with

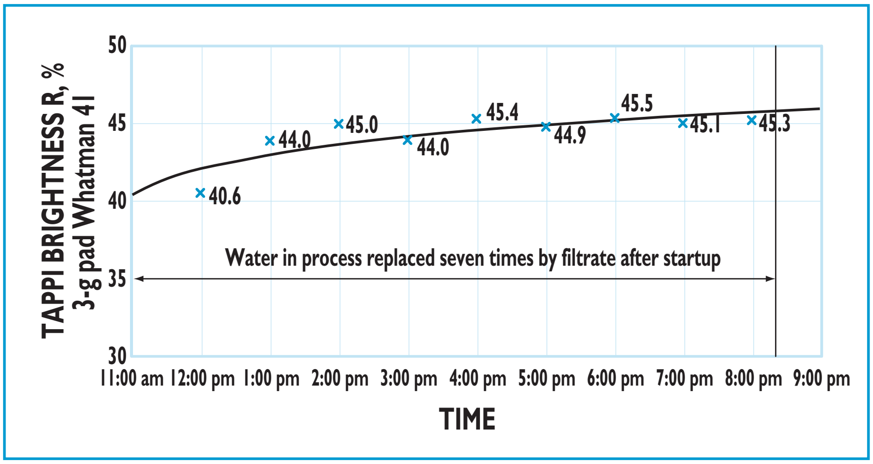
Chemical	Dosage
Silicate	1% as received
Peroxide	1% (100% active)
Caustic*	0.2% (100% active)
DTPA	0.2% as received

*Only added during Trial 1 (25%) flexo

III. Peroxide dispersion chemistry

Trial	Flexo ONP, %	Pulper chemistry	Pulper pH (*1*)	Prefl. pH (*1*)	Postfl. pH (*1*)
1	25	0.2% DPTA as received 1.0% silicate as received Adjust with caustic to pH 10.5 1.0% peroxide 100% active 1.0% Lionsorb 951 as received	10.5	8.5	5.5 0.15% Lionsurf 729
2	30	Same as Trial 1, but only to pH 10 and 1.5% Lionsorb	10.0	8.0	7.5 0.09% Lionsurf 729

II. Pulper chemistry



3. Filtrate brightness using 3-g brightness pads

closed water systems. To prove that flotation was the key process module removing the flexo inks, the disk filter and twin-wire press filtrate streams were returned to the process unclarified.

From a monitoring and testing standpoint, our objective was twofold:

- Monitor brightness after first-stage flotation (preflotation) over time
- Monitor disk filter and press filtrate quality over time.

Our goal was to establish a "performance equilibrium" with respect to brightness and filtrate quality after a finite number of total water turnovers. One total water turnover is defined as the time it takes to replace all the process water with recycled unclarified water.

The process design for the alkaline loop is shown in **Fig. 1** and for the postflotation acid or neutral loop in **Fig. 2.** For the two trials described here, the pulp was typically processed through the complete alkaline loop on one day and then sent through the second loop on the next day.

Recovered paper furnish specifications

The main difference between the two trials was in the amount and source of flexo ONP used. The recovered paper furnish for each trial is shown in **Table I.**

Deinking chemistry

During both trial runs, the flotation deinking chemistry was added to the high-consistency pulper at 12% consistency and 50°C. Batch pulping time was 20 min. Both trial runs had an alkaline preflotation loop. The lower-flexo-content run had an acid postflotation loop. The higher-flexo-content run had a neutral postflotation loop. During both runs, bleaching was performed with a conventional peroxide recipe in the disperser system.

The pulper chemistry is shown in **Table II.** The base water hardness was 100±10 ppm (mg/L $CaCO_3$). Note that initial pulping conditions were in the pH range 10.0–10.5 for both experimental runs. Hence, the organoclay functioned as a flexo ink collector, even under conditions in which the flexo ink is highly dispersed and which are typical for many ONP/old magazine (OMG) systems deinking traditional oil-based news inks.

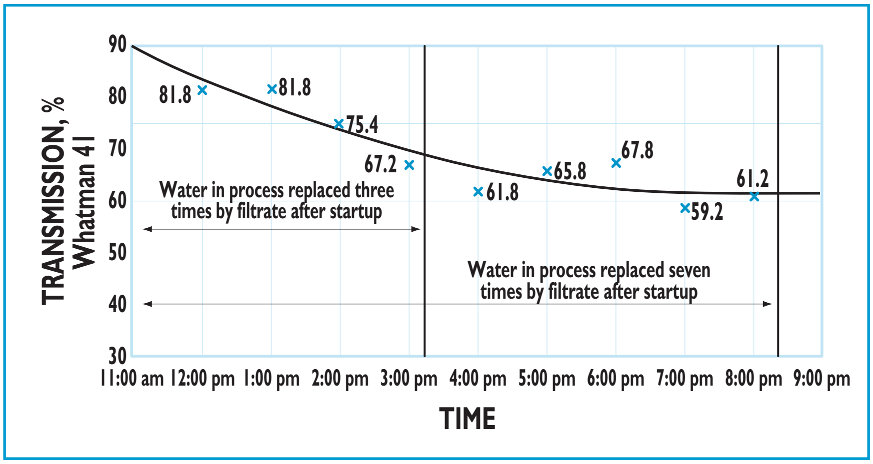
No additional surfactant was added in the pulper. For postflotation, 0.08%–0.15% of a commercially available surfactant, Lionsurf® 729, was used to stabilize froth formation and aid in collecting any inks released in the dispersion/bleaching stage. Froth stabilization became an issue due to much greater than expected ash removal in the preflotation stage (see "Results and discussion").

Dispersion chemistry

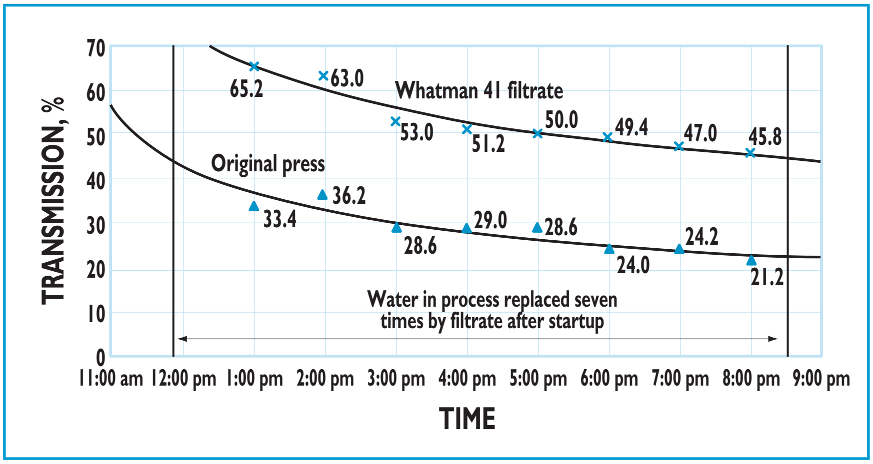
The peroxide dispersion chemistry, shown in **Table III,** was added prior to the heating tube and steam injection. The pulp feeding the disperser was at 82°C. During the second run, we did not add caustic and bleached at pH 7.5! During both runs, we applied 50–70 kW·h/ton on 30%-consistency stock, which is typical for newsprint dispersion systems.

Pulp and paper test methods

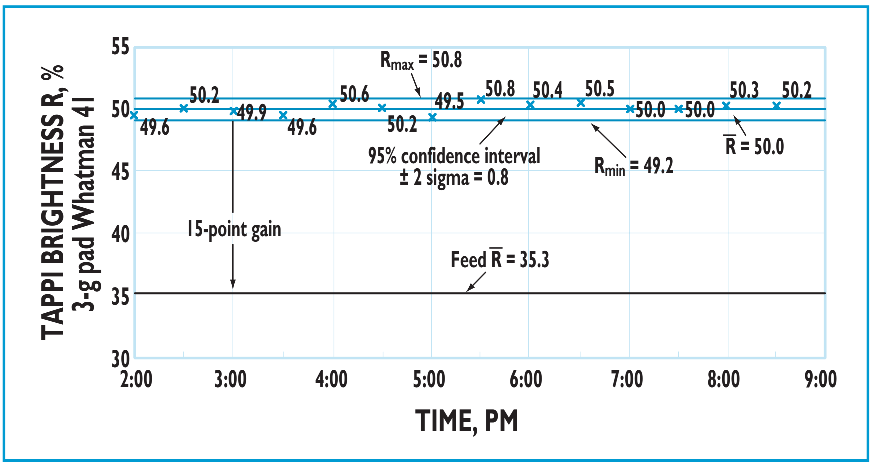
Brightness was measured with a Technidyne GE Brightness Meter (fluorescence component included)



4. *Brightness pad transmission quality*



5. *Transmission quality of twin-press filtrate*



6. *Brightness measured across Voith Sulzer Type E flotation machine*

following TAPPI Test Methods T-452 and T-218. From each sample point, 3-g brightness pads were made on Whatman 41 filters (pore size=20–25 μm) using a Büchner funnel. Additional brightness pads were made using Millipore® filters (pore size=1.2 μm). The filtrates of these two different pads were checked for light transmission (in percent) with a Hach DR/2000 Spectrophotometer. With this testing program, we were certain to evaluate:

- The impact of the flexo inks on brightness
- The occurrence of flexo ink after flotation in the filtrate of the brightness pads.

RESULTS AND DISCUSSION

Water circuits

After beginning the trial, we periodically monitored the disk filter filtrate. The filtrate was combined as 100% cloudy filtrate. We made 3 g brightness pads and checked the light transmission of the pad filtrate to evaluate if the filtrate quality deteriorated over time. **Figure 3** shows that the brightness of this filtrate was remaining consistent at 44–45 GE brightness.

As shown in **Fig. 4,** the brightness pad transmission quality leveled out after approximately three complete recycled water turnovers; the same trend was observed with the twin-wire press filtrate.

Since recycled dilution water after the disk filter is added before feeding the stock to the press, impacting final brightness, we also tested the filtrate of the twin-wire press. These results are shown in **Fig. 5.**

The transmission data were available during the trial, leading us to the conclusion that the flexo inks were not traveling with the water circuits, and the flotation performance had to be adequate in removing flexo inks.

Flotation accepts brightness for 25% flexo content

During the 25%-flexo run, brightness numbers did not decrease over time, and we achieved an average brightness gain of 15 points across the Voith Sulzer Type E flotation machine, as shown in **Fig. 6.** Remarkably, we observed a significant flotation side benefit—excellent ash removal efficiencies across the flotation machine—as shown in **Table III**.

During the 30% flexo run, we monitored the brightness over time at the twin-wire press discharge. Like the 25% flexo run, the brightness remained relatively constant at about 51.0, as shown in **Fig. 7**. The two runs are summarized in **Table IV**.

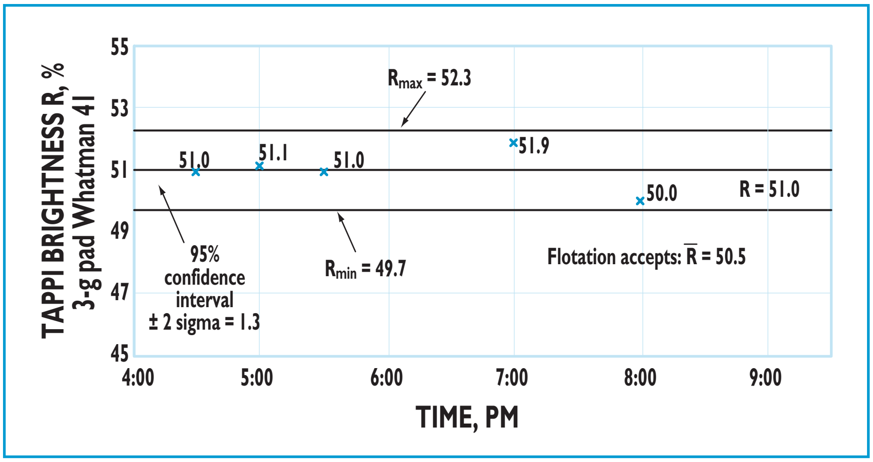
Peroxide dispersion

Thickening after flotation yielded only a 0.5 point brightness gain. Peroxide dispersion provided an 8 point brightness gain, as shown in **Table V**.

Postflotation

During both postflotation runs, there was no additional brightness increase. Image analysis showed a relative lack of ink particles greater than 40 µm in diameter in the stock following dispersion, and very little change in the quantity or distribution of microscopic (5–40 µm) particles across the postflotation stage. During a subsequent thickening stage using the disk filter and twin-wire press, the stock gained 2–3 brightness points, achieving a 61 GE brightness for both runs.

These data may indicate that the predispersion flotation was extremely efficient at removing all floatable ink, including ink bound to the organoclay particles. If this were the case, then the dispersion stage mainly provided a bleach mixing function; no additional ink was released or generated in the size range that was floatable. However, some fine ink may have been released from the fiber that was then washed out in the thickening stage. Additional organoclay may well have been more appropriate for this postflotation stage as a collector.



7. Brightness at the twin-wire press discharge

Flexo ONP	GE brightness feed flotation, %	GE brightness flotation accepts, %	Brightness gain points *(1)*	Ash removal, %	Total shrink, %
25% flexo, 1.0% Lionsorb	35.3	50.5	15	66	10[a]
30% flexo, 1.5% Lionsorb	38.6	50.5	12	80	12[b]

a Surfactant dosage addition showed good runnability in Voith cells; foam-breaking device can be turned off to improve yield.
b Surfactant dosage addition was too high and created excessive foaming; foam-breaking device was required, contributing to a low yield.

IV. Flexo flotation deinking with foam breaking

Flexo ONP	Feed, %	Discharge, %	Brightness gain, points *(1)*
25% flexo	50.2	58.1	8
30% flexo	51.0	59.0	8

V. Brightness values through peroxide dispersion

CONCLUSIONS

The ability to remove flexo ink in a full-scale process with a closed water loop has been demonstrated during trial evaluations in the 30-tons/day continuous-process pilot plant at Voith Sulzer. In addition, excellent ash removal rates—exceeding 65%—were also observed. These trials suggest that organoclay chemistry is a viable technology for ink, ash, and contaminant removal in flotation-only deinking systems.

Further work is required to optimize foam levels, yield, and process water quality. Characterization of the ink present following dispersion is also necessary to optimize postflotation chemistry, pH, and other process parameters. **TJ**

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