Deinking process water flotation: An innovative solution to improve deinking and papermaking processes efficiency

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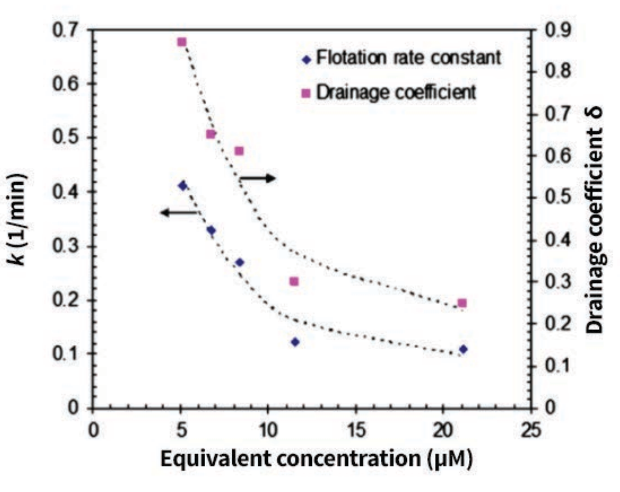
ABSTRACT: High and uncontrolled surface active substances (SAS) concentrations in deinking line process waters strongly disturb flotation deinking. Indeed, their adsorption at air/water and ink/water interfaces stabilizes air bubbles and ink particles, reducing ink collection during flotation. In addition, foam stabilization promoted by SAS reduces froth drainage and consequently enhances suspended solids losses. All in all, selectivity of flotation is negatively affected by excessive SAS concentrations. Floating process water with adapted technology, a patented solution, is an efficient way to decrease SAS concentration and thereby recover flotation deinking selectivity. This concept was tested at industrial scale with success. First loop clear filtrates of a deinked pulp line (for newsprint production) were floated. At steady state, this treatment allows a 20% reduction of the SAS concentration in first loop process water. Evaluation of ink removal selectivity at pre-flotation and post-flotation showed a significant improvement in comparison with reference values. During the trial, the mill could either produce pulp with lower ink content and consequently higher brightness (50 ppm effective residual ink concentration [ERIC] less, corresponding to a 1 brightness point increase) for similar losses, or similar pulp quality with 1% to 2% yield improvement, representing a huge cost reduction. In addition, beneficial side effects were reported, such as reduced deinking chemicals consumption (soda, silicate, and bleaching agents) and better paper machine runnability, especially in terms of stickies breaks.

Application: Mills might be able to increase brightness or yield in their deinking line using the process described here.

In a deinking line, concentrations of surface active substances (SAS) are usually high. These SAS come from many sources. Some are voluntarily added in the pulp and paper manufacture processes, such as deinking chemicals, dispersants (in coating colors, sizing, glues, etc.), and defoamers. Some, such as fatty and resin acids, are released by the fibers themselves. The amounts of SAS released from recovered paper or mechanical pulp can be very significant. For recovered papers, their release depends on the grades of recovered papers, but also on the alkaline treatments used during the recycling process [1].

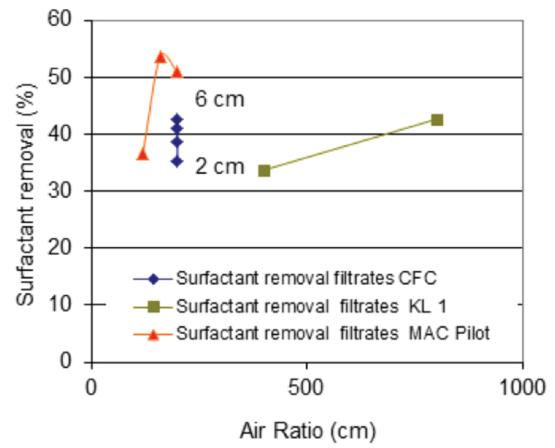
The presence of SAS in process water is reported to impair flotation deinking by decreasing ink removal and fiber yield [2-5]. **Figure 1** shows the influence of increasing surfactant concentrations on the reduction of ink removal efficiency by flotation [6]. Similarly, the foam drainage coefficient is also decreased when surfactant concentration increases, meaning that more stable foam is generated, resulting in an increase of flotation losses.

The detrimental action is explained by the surfactant adsorption onto the hydrophobic surfaces, mainly ink particles and air bubbles, thus reducing their adhesion energy [7] and by the froth stabilizing action that reduces the drainage of the fibers back to the suspension [8].



1. Influence of surface active substances (SAS) concentration on ink flotation removal constant and foam drainage coefficient [6].

Process water flotation by concentrating SAS in foam that is rejected has a great potential to decrease SAS concentrations in process water and to improve flotation deinking efficiency and selectivity. SAS removal achieved by process



2. Surfactant removal achieved during deinking process water flotation. Comparison of various flotation cells (laboratory batch, laboratory continuous, and pilot continuous) [9].

water flotation can vary from 35% to 70%, depending on the flotation technology used [9,10] (**Fig. 2**). The influence of this process water treatment on flotation deinking efficiency was also evaluated at laboratory scale showing improvement of ink removal, and flotation losses were significantly reduced (**Fig. 3**) [9]. Gendron et al., Ben et al., and Ricard et al. [11-13] reported the results of long-term mill trials of column flotation implemented in a thermomechanical pulp (TMP) newsprint mill. The treatment is claimed to be efficient on resinous materials (most having structures similar to surfactants) contained in the treated filtrate. Removal efficiency achieved by the flotation column is in the range of 30%-45%.

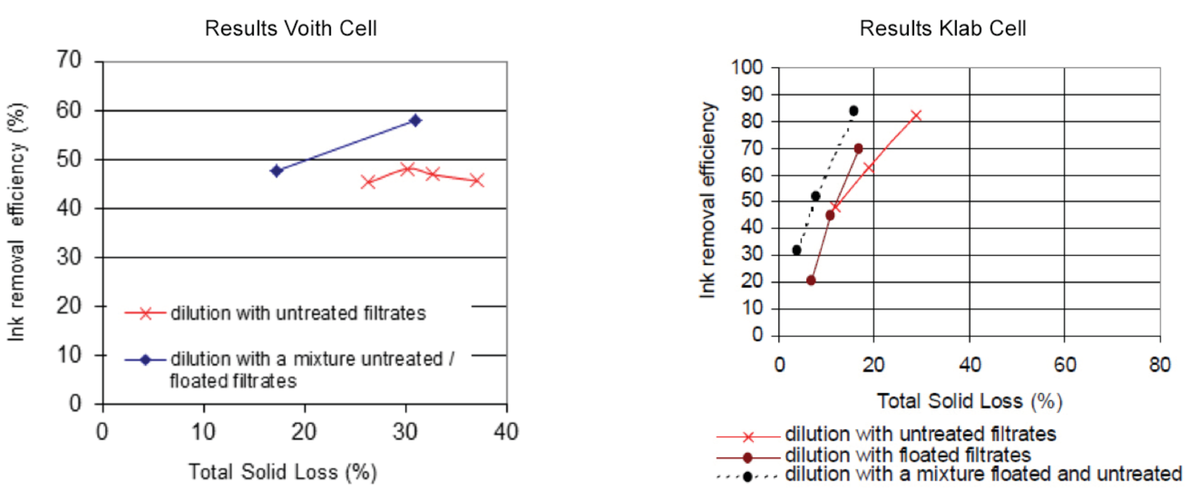
All in all, previous works at the Centre Technique du Papier (CTP) in Grenoble, France, have emphasized the importance of released surface active substances on ink flotation selectivity. This led CTP to apply for a patent [14] aiming at removing these substances from the process waters by an adapted flotation process. If the major effects have been identified at laboratory and then pilot scale, the real impact of this treatment on the runnability of an industrial process was not quantified yet. Since that time, CTP was able to evaluate such a treatment at mill scale. This paper reports the main outcomes of these trials, focusing on reduction of SAS concentration in process water, improvement of deinking selectivity, and some additional beneficial effects regarding chemicals consumption and pulp runnability on the paper machine side.

MATERIALS AND METHODS

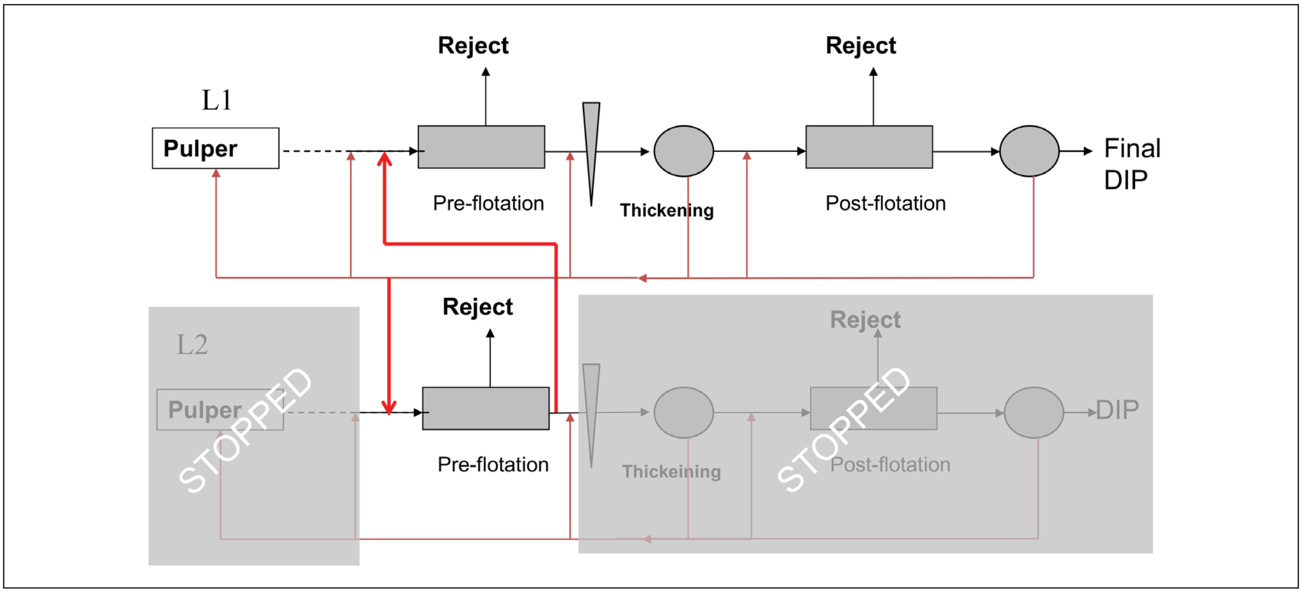
Description of the trial

A trial was conducted in a deinking mill producing newsprint based on deinked pulp (DIP) and TMP. The mill owns two deinking lines organized according to a conventional two loops process. The presence of these two parallel lines made the trial possible. One of the two lines was stopped and its pre-flotation was used to treat a part of the first loop water from the other line, which was still running. **Figure 4** describes the structure of the trial.

The first loop filtrate flow treated represents 610 m³/h, corresponding to 25% of the water available in the first loop. The foam that is generated is rejected from the mill through the effluent treatment plant. The rejected foam flow is estimated at 12% of the entering flow, representing 2.9 m³ additional rejected water per metric ton of DIP produced. Total suspended materials concentration of the treated water is 1.2 g/L.



3. Ink removal efficiency versus total solid losses achieved during deinking flotation. Influence of process water nature (unfloated versus floated waters) [9]. Results obtained with two types of laboratory flotation cells (left: Voith cells; right: Klab cell from Kadant).



4. Schematic of the mill trial, with only pulper flotation and thickening represented.

Water flotation process characterization

Surface tension measurement is done using a maximum bubble pressure technique based on the Laplace Law. A SITA apparatus (SITA Messtechnik GmbH; Dresden, Germany) is used, with average bubble time life set at 15 s. In all cases, surface tension is given for a water temperature of 25°C. This measurement is used to assess the SAS concentration in the water.

The SAS rejection rate was determined by measurement of surface tension. Serial dilutions of water flotation rejects (highly concentrated in SAS) are produced and their surface tension (dynamic measurement) is measured. This enables us to draw a dilution curve reporting the evolution of surface tension versus dilution rate of the foam. Then, determining the surface tension in the inlet and flotation accepts enables us to estimate SAS concentration relative to the flotation reject SAS concentration. SAS rejection rate was determined using relative SAS concentrations at inlet, outlet, reject, and flotation flows balance. The method is fully described elsewhere [3].

Total suspended solids (TSS) losses are determined through ash measurements in the inlet, accepts, and rejects of the cell treating the filtrate. The calculations were made according to the following equation:

$$TSS\ losses = \frac{(ash\ in - ash\ acc)}{(ash\ rej - ash\ acc)}$$

where:
ash in = ash percentage at the flotation inlet
ash acc = ash percentage at flotation accept
ash rej = ash percentage at flotation reject

Flotation deinking characterization

Pre-flotation and post-flotation were investigated before the trial to set the baseline and then during the trial at 3 h, 16 h, 20 h, and 24 h. Ink removal efficiency and TSS losses were evaluated.

The ink removal rate was followed through the effective residual ink concentration (ERIC), and brightness measurements were performed on pulp pads produced during the trial. The ink balance was calculated using the ERIC value and TSS mass flow. Ink mass flow rate is approached in multiplying ink concentration (ERIC) by pulp mass flow rate.

TSS losses were determined using ashes measurement as already described in the case of the water flotation (yield calculation method).

Apart from these laboratory measurements, online mill records were collected over the trial period (before and after) regarding ERIC and brightness at different positions in the process.

RESULTS AND DISCUSSION

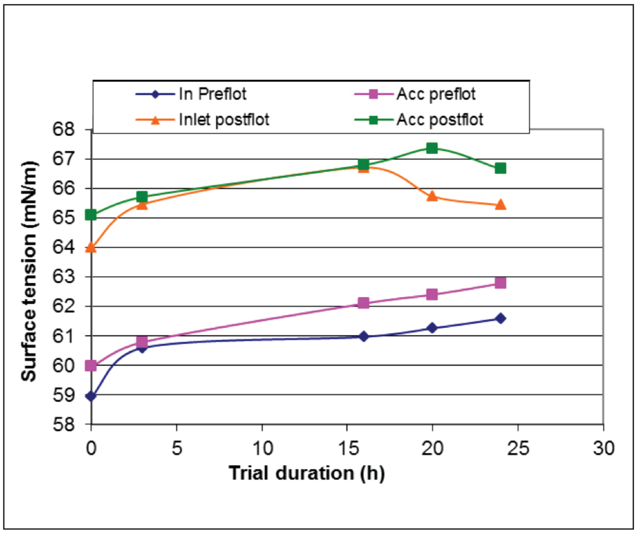
Water flotation efficiency

Water flotation efficiency was determined primarily in terms of SAS removal. Calculations were based on the dilution curve obtained from flotation rejects, surface tension measurements at the inlet, and rejects for the different times of the trial (3 h, 16 h, 20 h, and 24 h). Reject water flow was estimated at 12% for all measurements.

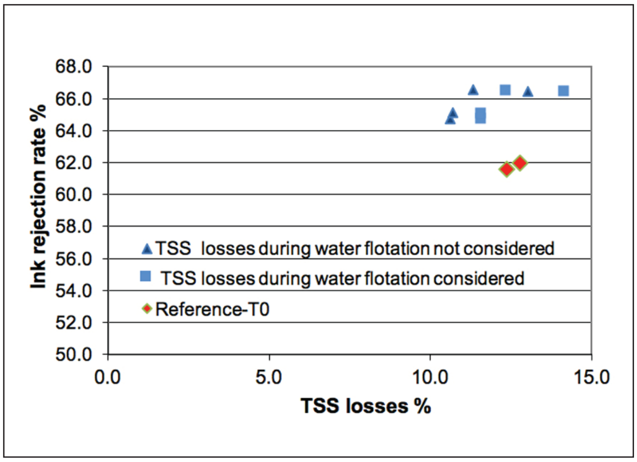
Table I shows the SAS rejection rates. Water flotation treats only 25% of the total available water in the first loop. Considering that, the configuration of the process (internal loop treatment with treated flow sent backwards), and pulp consistency at the end of the first loop, we can estimate, by a simple simulation that a reduction of 20% of the SAS amount entering the pre-flotation is achieved when compared with reference values.

Time	Inlet Surface Tension, mN/m	Reject Surface Tension, mN/m	Concentration Factor	SAS Reject Rate, %
3 h	60.9	54.9	1.9	23.2
16 h	61.9	55.1	2.2	25.9
20 h	61.5	55.0	2.1	24.8
24 h	62.6	54.8	2.3	27.9
			Average SAS Reject Rate, %	25.4

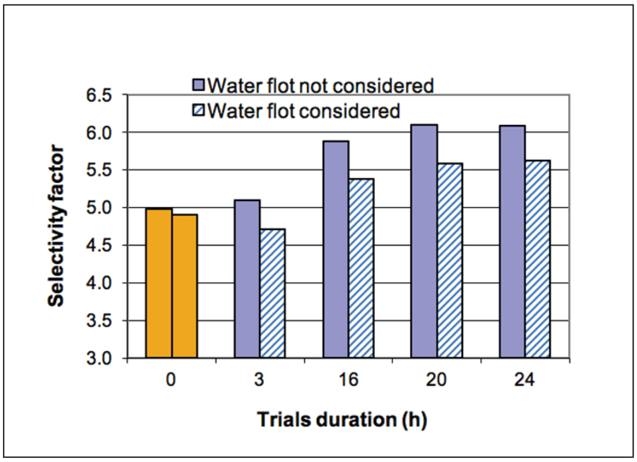
I. Surface active substances (SAS) rejection rates determined during flotation trials.



5. Evolution of surface tension at pre-floation and post-flotation during water flotation trials.



6. Evolution of ink removal rate versus total suspended solids (TSS) losses during water flotation trials.



7. Evolution of pre-flotation selectivity factor (ink rejection rate/ TSS rejection rate) during water flotation trials.

This estimated reduction is confirmed by the evolution of the surface tension measured at different positions during the trial period. **Figure 5** shows the results. It clearly appears from these measurements that surface tension increases in all process parts (first loop as well as second loop) as soon as water flotation is started, which indicates a reduction of the SAS concentration.

Influence on deinking efficiency

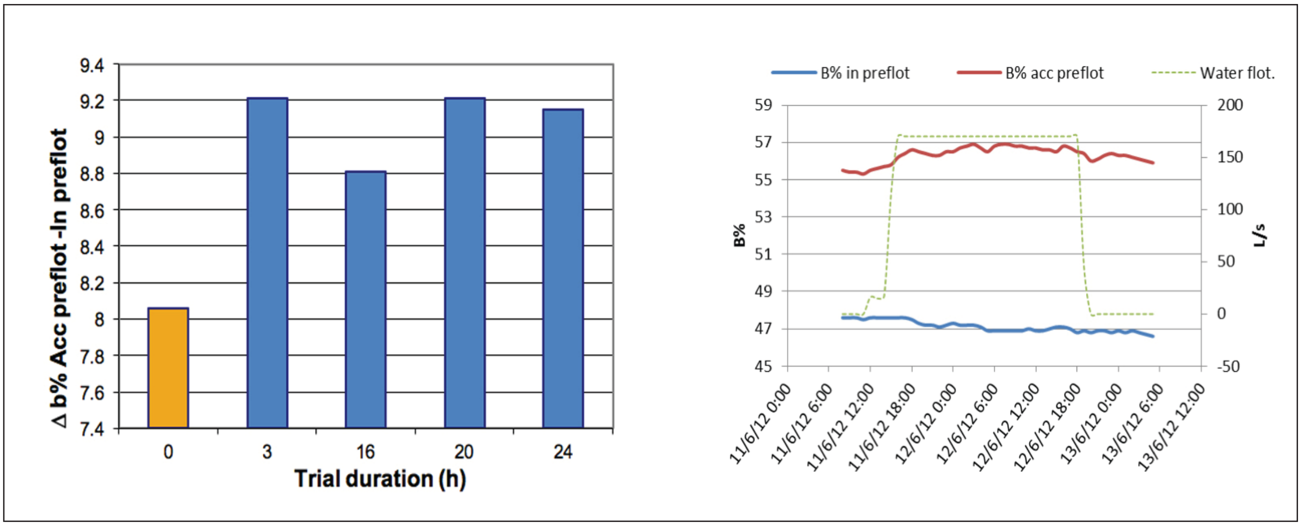
Reduction of SAS concentration should result in an improved flotation efficiency and selectivity. In order to check this aspect, ink and TSS balances were performed during the trials in both pre and post-flotation.

Influence on pre-flotation

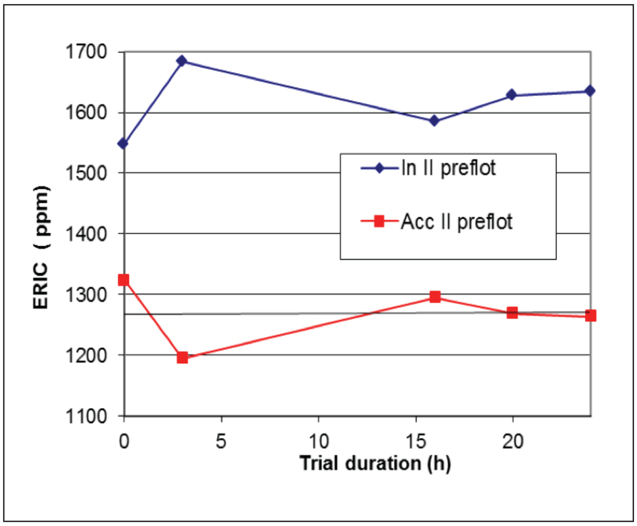
Ink removal rates and TSS losses were determined during the trial and compared with those of the reference period (T0). The results (**Fig. 6**) show two positive aspects achieved by the water treatment implemented:

1. For all trial durations, the ink removal rate was higher than what is commonly achieved in usual conditions (without water flotation): 65%–66% versus 62%.
2. This additional ink removal rate is obtained for similar or lower suspended solids losses (except at one point, corresponding to the shorter trial duration).

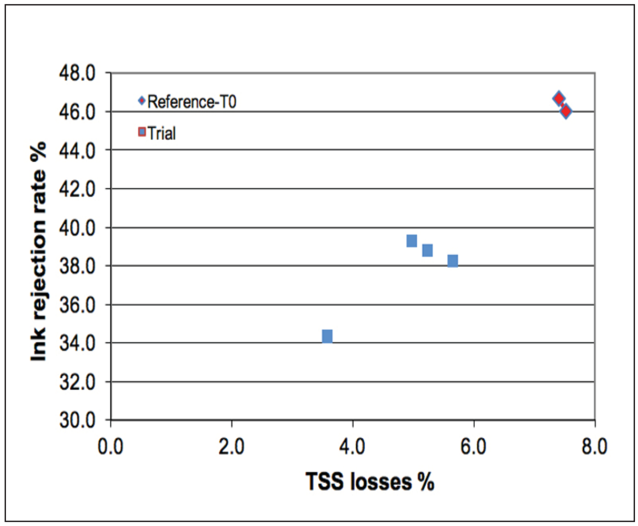
Furthermore, this is always true when suspended material losses generated by water flotation are counted with pre-flotation losses.



8. Evolution of brightness in pre-flotation. Left hand side: off-line measurements performed on pads. Brightness gain between inlet and accept reported. Right hand side: brightness value measured through mill sensor at the end of first loop.



9. Evolution of effective residual ink concentration (ERIC) at secondary stage inlet and accept.



10. Evolution of ink removal rate versus TSS losses during water flotation trial.

Thus, the ink rejection rate and the selectivity of the pre-flotation step were improved. **Figure 7** shows the evolution of the selectivity factor (representing ink quantity rejected achieved on average when 1% of TSS are lost). The selectivity factor increases from 5 to 6 in pre-flotation, or from 5 to 5.5 if we also consider the losses generated by the water flotation step.

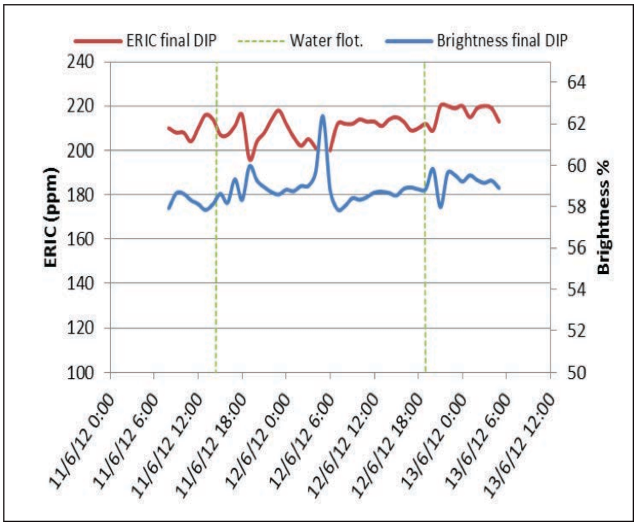
In the framework of this trial, the treated first loop process water contained high concentrations of suspended solids (1.2 g/L). Nevertheless, it is recommended, as the treatment is mostly intended to remove SAS, to treat a water flow having a lower suspended solids concentration (clear filtrate should be more suitable).

As a consequence, deinked pulp after pre-flotation has improved optical properties compared with the reference situation; residual ink content is reduced (from 290 ppm to 255 ppm, on average) and brightness is increased. **Figure 8** shows the evolution of brightness determined on pulp samples collected during the trial and by an on-line mill sensor at the end of first flotation. An increase of 1 to 2 points is reported.

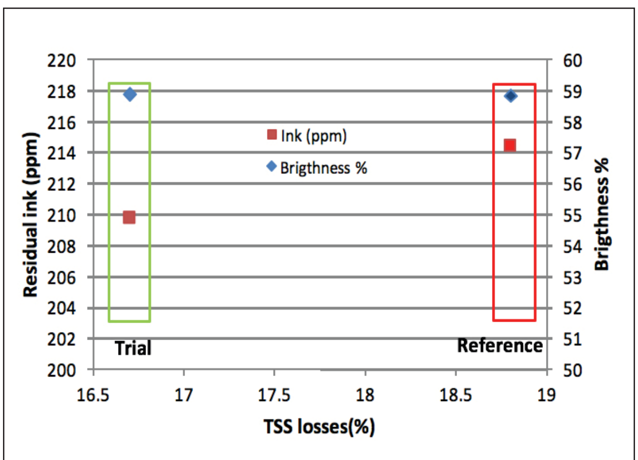
A better functioning of the secondary stage of the pre-flotation probably explained the improvement observed at pre-flotation. **Figure 9** shows the evolution of ink content at the secondary stage inlet and outlet during the trial. An increase of ink concentration at the inlet is in accordance with a better ink removal rate and selectivity at the primary stage. Moreover, in spite of this ink concentration increase at the inlet, accepts show a reduced ink concentration, which indicates better ink removal and selectivity at the secondary stage. As SAS are concentrated in the secondary stage, it makes this stage particularly sensitive to any variations of SAS concentration.

Influence on post-flotation

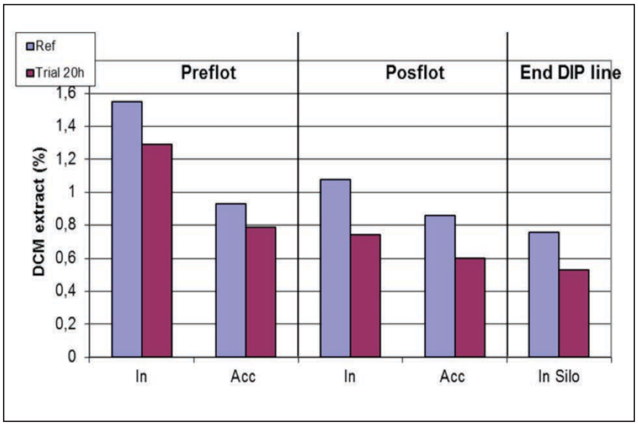
As in pre-flotation, the ink removal rate and TSS losses were determined during the trial and compared with the reference period. **Figure 10** shows the results obtained. Contrary to pre-flotation, the ink removal rate was slightly reduced when water flotation is applied. This might be the consequence of reduced foam stability, which favors the drainage of ink particles back to the suspension. However, in parallel, a decrease of TSS losses is observed. All in all, a slightly better selectivity of the post-flotation is observed (selectivity factor from 6.5 to 7 during trial versus 6.1 to 6.3 in reference). This reduced ink removal in post-flotation is compensated for by the lower residual ink level achieved at the end of the first loop, resulting in a final pulp having optical quality close to that measured in the reference period (**Fig. 11**). This same quality is achieved for improved yield, due to lower losses generated in pre- and post-flotations. As shown in **Fig. 12**, a roughly 2% yield improvement is observed for a pulp production of similar optical characteristics.



11. Evolution of final deinked pulp brightness and ERIC during the trial.



12. Final pulp quality (brightness and ink content) versus total deinked pulp process losses.



13. Influence of water flotation on dichloromethane extract content of the pulp at different locations in the deinking line.

Additional effects

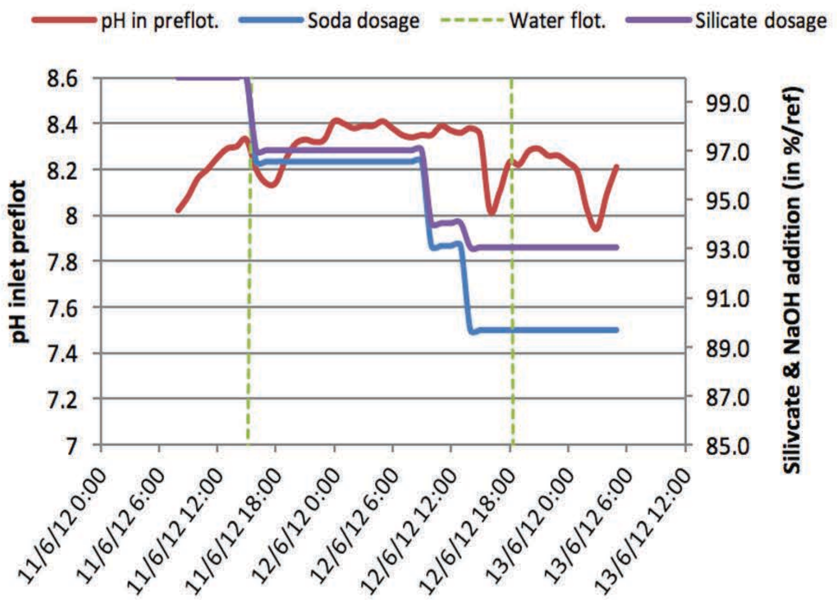
Apart from the improvement of deinking, water flotation has some additional beneficial effects, including a reduction of stickies-related material contained in the pulp. Dichloromethane extract of the pulp was measured during the reference period and during the trial. Results show a significant decrease of the extract rate contained in the pulp in all samples tested: pre-flotation (in and accept), post-flotation (in and accept), and inlet of DIP storage (**Fig. 13**). This improvement results from removal of microstickies contained in process water. As in this trial, treated filtrate was rather rich in fine suspended solids (1.2 g/L – mainly fillers). The good runnability of the paper machine during the trials and immediately after was probably partly explained by this pulp quality improvement.

An increase of pH in pulping was observed during the trial. As a consequence, the mill has had to reduce the alkaline sources added in the pulper (soda and silicate) to maintain pH in the same range (**Fig. 14**). This probably originates from the aeration of the water, which acts on different parameters, stripping CO_2 and volatile fatty acids and reducing anaerobic conditions that reduce the production of volatile fatty acids by the bacteria. Another consequence of these modified conditions is the presence of residual peroxide at the pulper outlet, which is rarely observed in usual conditions and which is also reported as a positive effect.

CONCLUSION

Innovative, patented, concept to control SAS in deinking lines

Floating process water with adapted technology is an efficient way to decrease SAS concentration in the deinking line. This concept was tested at industrial scale with success; first loop clear filtrates of a deinked pulp line (for newsprint production) were floated. At steady state, treating 25% of the water



14. Evolution of pH, soda, and silicate dosages during the flotation trial.

available in first loop allows for a 20% reduction of SAS concentration, with extremely limited solid losses.

Positive effect on the deinking line

Floating process water has two major effects on the deinking line:

1. It improves deinking selectivity, corresponding to a brightness improvement of 1 to 2 points, and yield remains constant; or the reverse, yield improves 1 to 2 points, and brightness remains at the same level.
2. It significantly reduces consumption of deinking and bleaching chemicals: soda = -30%, silicate = -35%, and bleaching agent = -80%.

Attractive economical balance

Economic calculations were made taking into account all aspects of DIP production: raw material costs, energy and chemical consumption, reject management, and yield. Depending on the strategy of the mill, two situations must be considered:

1. The mill wants to improve the brightness, while keeping yield at the same level. In this condition, the gain is estimated at 1.2 €/metric ton when water flotation is applied.
2. The mill wants to improve yield and keep brightness at the same level. In this case, the gain is estimated at 3 €/metric ton.

The second option seems to be more attractive, but the drastic reduction of bleaching in the first option might have a very positive effect on paper machine runnability, which is not counted in this estimation. **TJ**

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ABOUT THE AUTHORS

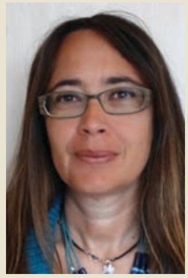
We conducted this study to help deinking mills solve deinking difficulties resulting from increased water contamination by surfactants. The mill trials reported in this paper are the complement of years of laboratory and pilot studies performed in our laboratory and which had led to a patented technology.

The most difficult part of this research was to move from laboratory and pilot research to industrial trials. In these studies, we discovered the influence of surfactants (positive and negative) in many aspects of the deinking.

The most interesting findings concerned the additional effects of the treatment that were not anticipated from the previous laboratory and pilot studies. These additional effects strongly enhance the interest of the technology.

Mills can benefit from this study by implementing the technology in their production lines.


Delagoutte


Zeno


Carré

Our next step will be implementation of the technology in deinking mills.

Delagoutte and Zeno are research engineers and Carré is team manager at the Centre Technique du Papier (CTP), Grenoble, France. Email Delagoutte at Thierry.Delagoutte@webctp.com.