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# Parameters of Deinking Efficiency in an Industrial Flotation Bank

P. Huber,<sup>\*,†</sup> X. Rousset,<sup>†</sup> E. Zeno,<sup>†</sup> and T. Vazhure<sup>‡</sup>

<sup>†</sup>Centre Technique du Papier, BP 251, 38044 Grenoble Cedex 9, France

<sup>‡</sup>Aylesford Newsprint, Kent ME20 7DL, United Kingdom

**ABSTRACT:** There is a need to monitor flotation deinking operations, in order to better control their efficiency, reduce losses, and save fiber resources. The objective of this work is to understand the variations of ink removal efficiency and flotation yield at industrial scale. A preflotation bank has been instrumented with air probes, and parameters relevant to flotation operations (chemicals dosages, brightness, effective residual ink content (ERIC), pH, temperature, water levels in cells, rejects flows, consistencies) have been exported from the mill data logging system. Then, relationships among flotation parameters are investigated. Air content in preflotation primary cells is found to vary considerably over time. On the other hand, air content in secondary cells is much higher, but rather stable. Ink removal is enhanced by a higher air content in primary cells. High air content is also found to impair the flotation yield. Most air content variations are explained by opposite variations of pulp concentration. It is proposed that pulp concentration lowers air content, by causing stronger pulp flocculation. These results suggest that deinking flotation yield may be maximized by running the flotation bank at the highest possible pulp concentration, while maintaining the target on the effective residual ink content.

## INTRODUCTION

The objective of the flotation process is to remove detached ink particles from the fiber suspension by injecting air bubbles: ideally, the hydrophobic ink particles are collected by air bubbles, then removed with the rejected froth, while hydrophilic cellulosic fibers and mineral filler are not separated. Usually, high ink removal efficiency is obtained at the expense of flotation yield.<sup>1</sup> Thus, ink removal by flotation still entails large losses. Handling of generated sludges raises various problems. Besides, flotation is not fully selective toward ink particles, so that a fraction of raw fibers and mineral filler are lost with the rejects,<sup>2</sup> mainly due to entrainment phenomena, rather than flotation of hydrophilic particles.<sup>3–6</sup>

Thus, there is an imperative need to improve the deinking flotation selectivity, for further enhancing the efficiency of recycling technology and the produced pulp quality. Therefore, a deep knowledge of the parameters affecting flotation efficiency is needed. Then, these findings, together with relevant monitoring sensors, will help control the flotation process. Improvements of the latter are likely to translate into increased fiber reuse (and recovery), with direct effect on conservation of energy and cellulosic resources.

The monitoring and control of industrial flotation cells has considerably evolved over the past decades. Once limited to concentration or level controls, online sensors in flotation cells now include effective residual ink and brightness sensors, in state-of-the-art mills. Technologies such as gas hold-up monitoring or in situ tracking of bubble size distribution open promising opportunities for flotation control.

Brightness of the accept floated pulp is the main quality criterion that is followed online, together with effective residual ink content (ERIC<sup>7</sup>). With an additional sensor on pulp feed flow, brightness gain and ink removal efficiency by flotation can be estimated in real time.<sup>8,9</sup> ERIC measurements are however only performed on whole pulp, so that it is not possible to discriminate

the free ink fraction (that can be floated) from the ink fraction still attached to the fibers and filler. Besides, the online analysis of the effective residual free ink in flotation cell accepts is regarded as a promising tool to control the flotation efficiency. Julien Saint Amand et al.<sup>10</sup> presented an online sensor to measure small ink particles (down to 3  $\mu\text{m}$ ). This device is able to measure the free ink, as it also makes a measurement on the washed pulp. Moreover, online specks control (for ink particles larger than 50  $\mu\text{m}$ ) is also a useful (and now classical) tool to improve pulp quality.<sup>11</sup>

Online control of ink removal is standard in state-of-the-art flotation deinking mills; however, online estimation of flotation yield is much more difficult. This crucial parameter of flotation efficiency is practically never measured online. In theory, it is feasible to calculate flotation yield from ash measurements only [ $\text{yield} = (\text{ash\_reject} - \text{ash\_inlet}) / (\text{ash\_reject} - \text{ash\_accept})$ ].<sup>1</sup> In practice, it is difficult to calibrate consistency and ash sensors for that task, especially for measurements in reject flows. Estimations of total losses (together with lost fiber fraction) have been proposed on the basis of conductivity measurements.<sup>12,13</sup> Those methods have yet to be applied online.

Gas hold-up, i.e., the average air content [ $\text{gas volume} / (\text{pulp} + \text{gas volume})$ ] in the flotation cell, is also a key parameter of flotation. It depends on the injected air ratio (air flow to pulp flow in the injector of the cell) and on the air to pulp residence time ratio in the cell. The air ratio does not tell how much of the injected air is actually present in the aerated pulp, while the air content is a true assessment of pulp aeration within the flotation cell. The general understanding is that, for a given air ratio, bubbles of various sizes will be formed in the pulp flow, depending on

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content data was logged at a 1 min frequency, and then averaged over a sliding period of 10 min. Four air probes were installed in the preflotation primary stage (third, fourth, fifth, and sixth cell), and another one in the preflotation secondary stage (first cell).

Besides, additional data relevant to the preflotation operations were exported from the mill data logging system [chemicals dosages, brightness, effective residual ink content (ERIC), pH, temperature, water levels in cells, rejects flows, consistencies; see Figure 1 for locations of sensors]. The frequency was 10 min for each variable. The data recording campaign lasted 4 weeks.

The preflotation loop actually consists of 2 symmetrical lines running in parallel. All measurements and recording were carried on line 1. Note that the consistency used here as an indicator of flotation consistency is measured after the preflotation loop, at the first stage cleaner loop (however variations and even absolute values are assumed to be very similar from mill experience).

The mill is equipped with online ERIC sensors (BT-53000, BTG instruments) at flotation inlet (ERIC<sub>inl</sub>) and flotation accepts (ERIC<sub>acc</sub>), so that ink removal efficiency at preflotation can be directly calculated:

$$\text{ink removal (\%)} = (1 - \text{ERIC}_{\text{inl}} / \text{ERIC}_{\text{acc}}) \times 100$$

The preflotation yield is not readily accessible with existing sensors. However, some information is available concerning reject flows and consistencies, so that an index which varies like preflotation yield could be calculated instead

$$\begin{aligned} \text{yield index (\%)} = & [1 - \text{total sludge flow (L/min)} \\ & \times \text{total sludge concentration (\%)} \\ & \times \text{adjusted coefficient}] / [\text{preflot thick stock flow (L/min)} \\ & \times \text{concentration (\%)} \text{ in 1st stage cleaner}] \times 100 \end{aligned}$$

where total sludge rejects are a combination of (preflotation line 1 + preflotation line 2 + first stage cleaner) rejects.

(Given the numerical values of the involved variables, the adjusted coefficient is arbitrarily set to 9 so that the average calculated yield index value is close to 90%, to match realistic values. Note that the value of this adjusted coefficient will not affect the detected trends).

Deinking selectivity has been defined as the ratio of ink removal to the relative fiber (oven-dry basis) rejection loss by Zhu et al.<sup>2</sup> Here, we defined it as the ratio of ink removal to the loss of solids present in the pulp suspension.

Prior to the analysis, data recordings were time-shifted, on the basis of a global cross-correlation analysis. Calculated time lags were consistent with knowledge from the process layout. Then, the data set has been purged from down-time periods or sensor malfunction.

Then, inter-relationships among process parameters have been investigated through PLS regression (partial least square or projection on latent structure). PLS attempts to find factors (latent structures) that maximize the amount of variation explained in X (predictor space) that is relevant for predicting Y [response(s)]. Thus, PLS combines advantages of both PCR (principal components regression) and MLR (multilinear regression). The PLS method has the advantage of being less sensitive to noise, numerous X variables, and collinearities among variables, than standard multilinear regression.<sup>24</sup> The significant effects are selected on the basis of the VIP criteria > 1 (variable importance in the projection, see ref 25).

**Table 1. Correlation Matrix (Pearson R) among Recorded Air Contents in Preflotation Primary Stage<sup>a</sup>**

	third cell	fourth cell	fifth cell	sixth cell
third cell	1	0.981	0.987	0.982
fourth cell	0.981	1	0.976	0.968
fifth cell	0.987	0.976	1	0.992
sixth cell	0.982	0.968	0.992	1

<sup>a</sup> Over a preliminary trial period lasting 1 week, prior to the data analysis period.

The cross-correlation analysis was performed with WinSTAT version 2009.1 (Robert K. Fitch software). All other statistical calculations were performed with XLSTAT Version 2010.3.02 (Addinsoft).

Surface tension values were measured on pulp filtrates (passing through a 100 mesh wire) by a maximum bubble pressure tension-meter (Sita t60, bubble lifetime = 15 s).

## RESULTS

First, the online air content recordings at the preflotation primary stage are analyzed. During a preliminary period (lasting 1 week), the air contents in the four instrumented cells were found to follow very similar variations (as the air signals are highly correlated together, see Table 1). So, it is equivalent to use any of the air content signals for the data analysis. Over this 1 week period, some other mill sensors were not operational, so that the data recording campaign effectively started after this preliminary period, and then lasted for 3 more weeks. For the data analysis, the air content in the third cell was selected, and the other preflotation air contents were discarded, in order to avoid collinearities problems.

The studied parameters are presented in Table 2, together with typical range of variations. First of all, it can be seen that the flotation efficiency of this particular line is rather stable, with typical ink removal and yield variability about 2.5%, so that it may not be easy to analyze these limited variations (it is thought that the presence of flexographic newspaper in the raw material dampens the observed variations of effective residual ink by the ERIC sensors). Besides, the flotation cell undergoes quite large variations of air content (during normal flotation operations): the average air content in the primary stage is 13.4%, with typical variations ranging from 11.2% to 15.6% ( $\pm 1$  SD interval, representing 70% of data points). Although air content is a key parameter of the flotation process, there is very little information about either average air content level or possible variations over time in commercial flotation cells. The average air content measured at the preflotation primary stage in this study is consistent with data reported by Dorris et al.<sup>14</sup> for similar flotation cell technology. On the other hand, the air content in the secondary stage was much higher, but showed very limited variations (31.1% on average, with typical variations from 29.3% to 32.9%). This may be explained by surfactant build-up in the secondary stage (surface tension = 52.0 mN/m, compared to 57.2 mN/m in primary stage feed) which tends to stabilize smaller bubbles, hence increase the air content. Also, lower fiber content and higher ash content (compared to primary stage) clearly thicken the flow, which may favor air trapping in the secondary stage cell, due to viscosity effects. The evolution of air contents is plotted in Figure 2, with large variations of air content in the primary stage clearly visible during certain periods. With

Table 2. Descriptive Statistics of Valid Data Set<sup>a</sup>

variable	unit	nb. valid data	min	max	av	SD	coeff var (%)
brightn dump tank2	%	1758	46.3	49.2	47.9	0.3	0.7
silicate to pulper	kg/T	1758	0.18	4.55	3.46	0.68	19.7
specific soap	kg/T	1758	1.50	11.35	3.75	0.68	18.2
feed pH thinstock		1758	7.13	7.58	7.42	0.05	0.7
temperature	°C	1758	42.5	54.1	51.5	2.2	4.2
1ry feed level (accept side)	%	1758	81.2	100.0	99.2	3.2	3.2
1ry feed level (reject side)	%	1758	31.6	40.0	37.7	0.8	2.1
2ry feed level (reject side)	%	1758	34.8	42.7	38.3	1.7	4.5
first stage cleaner feed cons.	%	1758	0.81	1.53	1.40	0.10	7.5
air 1ry, third cell	%	1758	9.9	21.9	13.4	2.2	16.7
air 2ry, first cell	%	1758	25.5	33.9	31.1	1.8	5.9
ink removal	%	1758	52.7	60.9	56.1	1.4	2.5
yield index	%	1758	78.5	97.2	90.1	2.3	2.5

<sup>a</sup> After pre-processing. Time period = 3 weeks; 1ry = primary preflotation stage, 2ry = secondary preflotation stage.

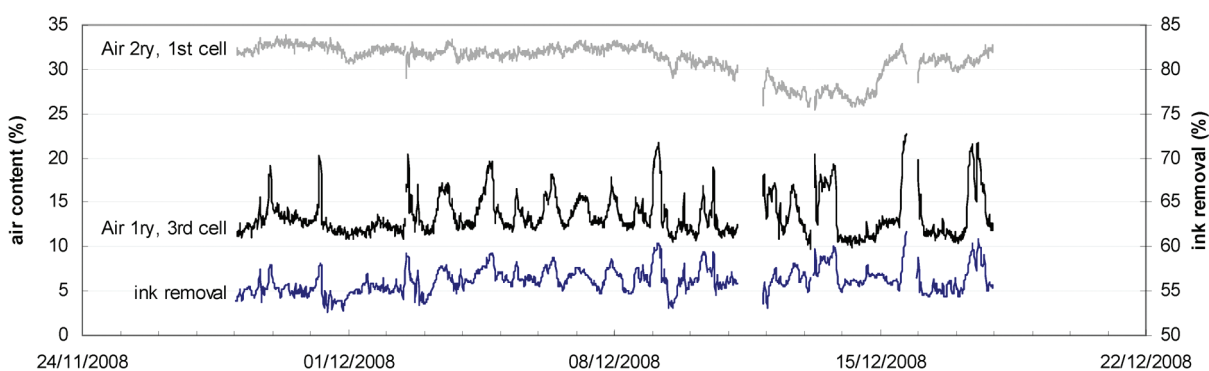


Figure 2. Evolution of air contents, together with ink removal (time span = 3 weeks; 1ry = primary preflotation stage, 2ry = secondary preflotation stage).

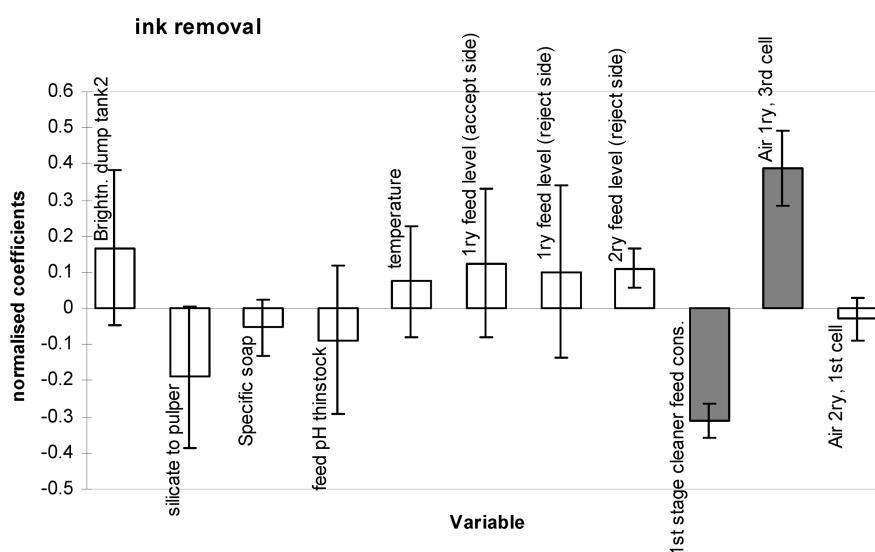


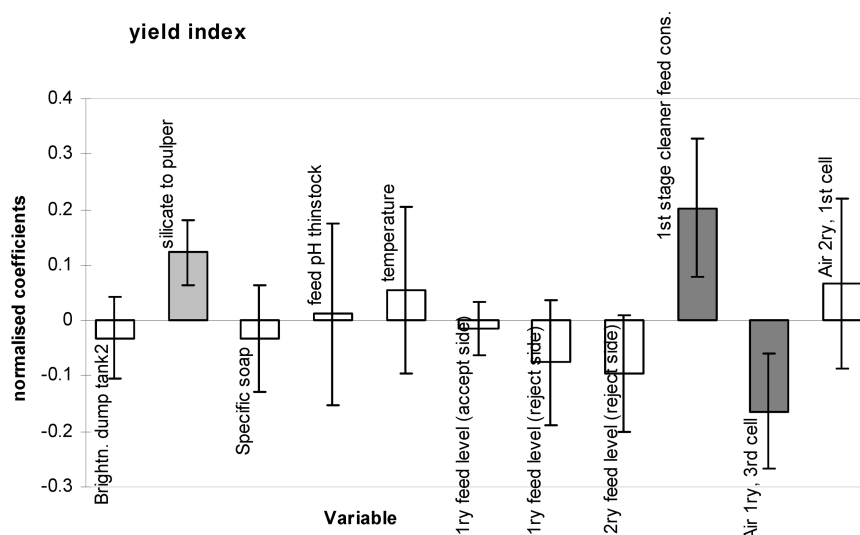
Figure 3. Coefficients of the PLS regression model for “ink removal” response (significant parameters highlighted in gray,  $R^2 = 0.767$ ; 1ry = primary preflotation stage, 2ry = secondary preflotation stage).

the Voith Ecocell flotation technology, aeration conditions are generally not changed. Indeed, the cell is designed to minimize fluctuations of applied air ratio. In order to maintain a constant air ratio in the venturi injector, a steady feed flow to the injector is

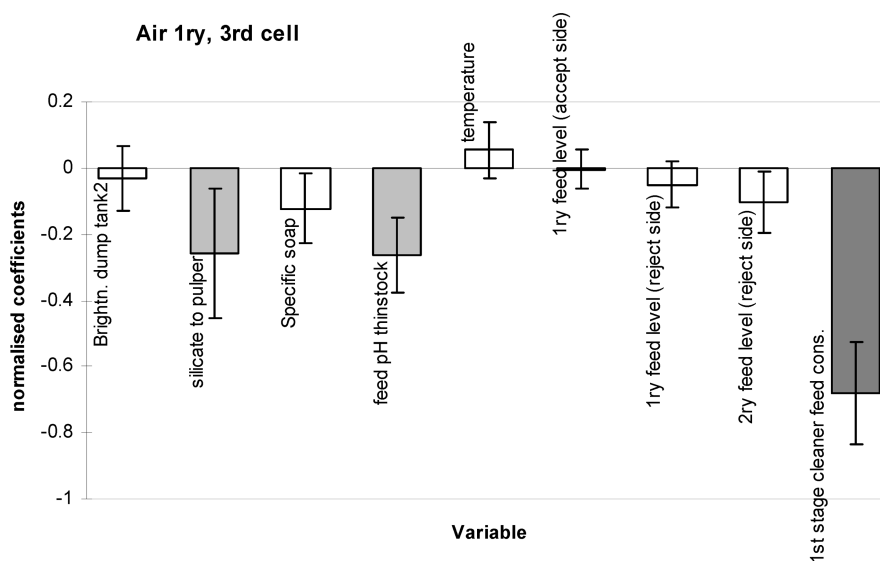
imposed by a constant speed pump. So, the observed variations of air content must be caused by another phenomenon.

Following Dorris et al.,<sup>14</sup> the brightness at the dump tank is included as a possible indicator of raw material quality variations





**Figure 4.** Coefficients of the PLS regression model for “yield index” response [significant parameters highlighted in gray (most significant in dark), warning: low  $R^2 = 0.282$ ; 1ry = primary preflotation stage, 2ry = secondary preflotation stage].



**Figure 5.** Coefficients of the PLS regression model for “Air 1ry, 3rd cell” response [significant parameters highlighted in gray (most significant in dark),  $R^2 = 0.745$ ; 1ry = primary preflotation stage, 2ry = secondary preflotation stage].

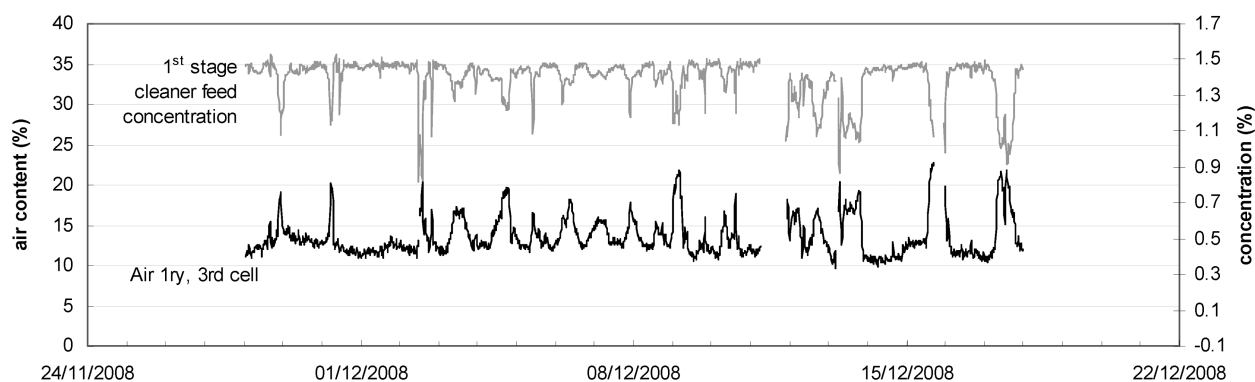
(as a high magazine/newspaper ratio in the recovered paper furnish is anticipated to be associated with a higher ash content, thus higher feed brightness to flotation).

Water levels in cells do not vary much. They are in fact regulated and follow only minor step changes (almost). Thus, their possible effect on flotation efficiency will be very difficult to detect.

Then, the flotation efficiency indicators (ink removal and yield) have been modeled as a function of the other remaining parameters in the data set. The PLS shows that ink removal is mostly influenced by air content in the primary stage and by pulp concentration. The higher the air content, the higher the ink removal, while pulp concentration has an opposite effect (Figure 3). It is also interesting to note that all other recorded parameters [raw material quality (as brightness at dump tank), chemical dosage, pH, temperature, water levels in cells or air content in secondary stage] have no significant effect on ink removal, over the observed range of variation (although specific

silicate and soap dosage varied significantly, see coefficients of variation in Table 2). The identified model explains the observed variations of ink removal quite well, over the whole 3 week studied period ( $R^2 = 0.767$ ). When plotting the evolution of ink removal together with air content, it is clear from visual inspection that the 2 parameters are following similar trends, most of the time (Figure 2).

The variations of yield index have been analyzed in the same way. The identified model has a low  $R^2 = 0.282$  (Figure 4). So the model cannot be used to predict the evolutions of yield. However, the detected influential parameters are still significant. The flotation yield appears to be mainly affected by pulp concentration and air content as well (however, the trends are opposite compared to ink removal). A high pulp concentration tends to boost the flotation yield, while a higher air content is detrimental to the yield. The silicate dosage also positively influences the yield, to a lesser extent.



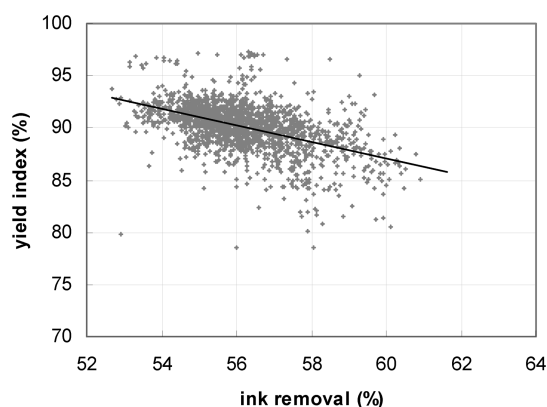
**Figure 6.** Evolution of pulp concentration, together with air content in primary stage (time span = 3 weeks; 1ry = primary preflotation stage, 2ry = secondary preflotation stage).

Finally, in order to better understand the causes of air content variations, the evolutions of air content in primary stage have been analyzed through PLS regression as well (Figure 5). The pulp concentration appears to be the main parameter that significantly affects the air content in the primary stage: the higher the pulp concentration, the lower the air content in primary stage. Silicate and pH are found to have a slight negative influence on air content (however, the significance of these effects is less clear: the VIP criteria was higher than 1, but with error bars overlapping that critical value). The evolutions of air content in the primary stage together with pulp concentration clearly show opposite variations, most of the time (Figure 6).

## DISCUSSION

Ink removal was found to be enhanced by a higher air content in the primary stage. This is likely due to the corresponding increase of bubble surface area available for ink particles collection (Hernandez et al.<sup>26</sup> showed that the flotation rate constant increased linearly with the bubble surface area flux, an exchange surface parameter which is typically proportional to air content in given flotation conditions<sup>27</sup>). This correlation between ink removal and air content is therefore anticipated from basic principles of flotation; however, to our knowledge, it is the first time it has been shown to be valid online. Dorris et al.<sup>14</sup> report a similar relationship between ink removal and air content in industrial flotation cells. They made samplings on three parallel deinking lines supplied with the same raw material, but working at different air content, and found a linear relationship between ink removal and increasing air content, on the basis of these three data points. Note that this general correlation between ink removal and air content may be broken by the effect of deinking soaps. Indeed some soaps are known to improve ink collection efficiency, while decreasing air content at the same time (through bubble agglomeration).<sup>14</sup> This was not observed here, despite large variations of soap dosage to the pulper (coefficient of variation = 18.2%, see Table 2). The reason why air content in the secondary stage is not found to influence ink removal is that it is almost constant over the period. In the following, only air content in the primary stage is discussed.

On the other hand, a higher air content is found to impair the yield. In these conditions, high air content likely increases losses through transport mechanism.<sup>3,4</sup> Flotation yield is also found to be improved with a higher silicate dosage. The general role of silicate in flotation is not fully understood. Some contradictory results have been reported, and very few studies have actually



**Figure 7.** Yield index vs ink removal over the whole 3 week recording period.

investigated the combined effect of silicate on both ink removal and losses. Recent work by Beneventi et al.<sup>28</sup> using a laboratory flotation cell designed to study both transport and froth drainage phenomena showed that higher silicate dosage does not change the flotation rate constant of ink particles, but can limit total losses. That supports our industrial observations, with no effect of silicate on ink removal, and improvement of flotation yield, presumably due to increased drainage from the froth back to the pulp. In our study, silicate also tends to slightly lower air content: this is consistent with the observed positive effect on yield. However, the mechanism by which silicate may actually decrease air content is unclear.

It is interesting to note that air content has opposite effects on ink removal and flotation yield. Plotting ink removal versus yield data shows that a higher ink removal was generally associated with a lower yield (Figure 7): this is well-known from laboratory studies<sup>2,28</sup> and validates our findings at an industrial scale on a long-term basis. Air content is thought to be the link that explains their interdependence. It is not clear from the data whether deinking selectivity is actually changed by varying air content. We tried to analyze variations of deinking selectivity (definition based on ref 2 using total losses instead of fiber losses) through PLS regression; unfortunately no significant parameters were found.

As air content has such a strong influence on flotation efficiency (through ink removal and yield), it is essential to understand the causes of air content variations, which are linked to pulp concentration variations.

Note that, in this study, pulp concentration varies a lot, and variations of concentration are directly correlated to deinked pulp production, as variations of production are mainly accommodated by adjustment of dilution ratio at flotation inlet: the higher the production, the higher the flotation concentration. This admittedly causes the ink concentration to vary at flotation inlet. However, it is unlikely that these variations of ink concentration have an impact on ink removal efficiency (for a given air content). Indeed, the removal of ink particles in flotation deinking is well described by first order kinetics, as shown by a number of studies (see, for instance, refs 5, 29–31), so that the number of removed particles by each bubble is proportional to the number of particles in the suspension. As long as the bubble surface is not saturated, the ink removal efficiency is not expected to be affected by the incoming ink particle concentration.

As air content is strongly influenced by pulp concentration itself, it is thought to be the link that explains the observed effect of pulp concentration on flotation efficiency. The following mechanism is proposed to explain the results: (1) higher pulp concentration enhances pulp flocculation (at fiber level); (2) more heterogeneous pulp has larger interstitial space between flocs; (3) this favors channeling, thus bubble collision and coalescence; (4) bubbles then rise faster to the froth, and the air content is reduced; and (5) finally, ink removal is impaired, as a result of reduced bubble surface area flux available for ink collection. (The reason why flotation yield is improved at the same time is not clear: it is possible that channeling effects reduce pulp transport to the froth.)

Following that reasoning, fiber concentration would actually be the parameter that affects air content, rather than total concentration (as fine elements are not expected to contribute much to pulp flocculation here, see ref 32). That remains to be verified experimentally.

While it is not possible here to bring the direct proof of this mechanism (i.e., corresponding evolution of pulp flocculation), many observations support this hypothesis.

During a two day industrial trial on Voith EcoCells preflotation banks, Dorris et al.<sup>14</sup> established that the major operating factor affecting air content is pulp consistency, presumably because the fiber flocs formation in more concentrated pulp suspensions promotes growth of air bubbles.

This is supported by results from Tang and Heindel.<sup>33</sup> They studied how various fiber types affect gas-hold-up in a laboratory column. Although virgin fibers were used only and no deinking was performed, they showed that fiber flocculation determines gas hold-up to a large extent. Indeed, by studying mixtures of short and long fibers, they showed that the higher the crowding factor, the lower the air content (where the crowding factor is a dimensionless concentration that takes fiber morphology into account,<sup>34</sup> and has been shown to determine fiber flocculation<sup>35</sup>).

Surprisingly, little information is available concerning the effect of pulp concentration on flotation efficiency. Yu<sup>36</sup> found that increasing total concentration from 1% to 1.6% caused a linear decrease of brightness gain in laboratory flotation. Britz and Peschl<sup>37</sup> conducted a pilot scale flotation study (Sulzer Papertec, Ravensburg), where they found that increasing total concentration (from 0.8% to 2.1%) reduced ink removal in each cell of the flotation bank. When working the flotation bank at constant brightness gain, increasing the pulp concentration reduced the combined losses. This is in direct agreement with our observations in an industrial flotation bank. Recently, Li et al.<sup>38</sup> made a laboratory investigation of the effect of

concentration on ink removal at flotation: concentration was found to have little effect in the range 1.0–1.2%, and then caused a marked decrease of ink removal efficiency over the range 1.2–1.5%.

Finally, the effect of pulp flocculation on flotation selectivity has been recently investigated in our group.<sup>39</sup> In this study, we have varied pulp flocculation in a pilot flotation cell by adding nonsurface active hydrophilic dispersants, that effectively deflocculated fibers (namely, guar gum and CMC). We found that air content increased as a result of pulp deflocculation (measured with an appropriate sensor, see refs 40, 41), with direct improvement of ink removal (the losses decreased at the same time, therefore enhancing the flotation selectivity, but that is likely due to an other mechanism).

All in all, pulp concentration appears to be the most influential parameter of flotation operations: it affects ink removal, and improves the deinking yield. In order to increase the yield of industrial flotation cells, an increase in consistency can be recommended. A compromise is nevertheless to be found, as such a solution contributes to reduce the ink removal.

Before concluding, it should be emphasized that trends observed here on a Voith Ecocell may not be valid with other flotation cell technologies. Different hydraulic regimes and turbulence conditions are likely to modify ink collection efficiency and pulp flocculation, thereby changing the effects of consistency on ink removal and flotation yield. Also, the proposed mechanism concerning the effect of pulp concentration on air content (through modification of fiber flocculation) may not be extrapolated beyond the main concentration range observed here (roughly 0.9–1.5%).

## CONCLUSIONS

Successful implementation of air content sensors for flotation monitoring was achieved, and brought information relevant to flotation efficiency control. Air content in preflotation primary stage cells is found to undergo considerable variations over time, during normal production operations. On the other hand, air content in secondary stage is higher, but much more stable. The higher the air content in primary stage, the higher the ink removal. High air content is also found to impair the flotation yield. Most air content variations are explained by opposite variations of pulp concentration. It is proposed that pulp concentration affects air content, through effects on pulp flocculation. In general, a higher ink removal was associated with a lower yield, emphasizing that it is difficult to break their interdependence, and to increase flotation selectivity. These results suggest that deinking flotation yield may be maximized by running the flotation bank at the highest possible pulp concentration, while maintaining the target on the effective residual ink content.

## AUTHOR INFORMATION

### Corresponding Author

\*E-mail: Patrick.Huber@webctp.com. Tel: +33.(0)4.76.15.40.51.

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